Data Analysis Expectations and Requirements

Reproducible Data

All analytical measurements and online measurements must have the validation or calibration statistics to clearly state capable range of measurement, statistical confidence intervals, and, where applicable, drift.

Every validated test method must have a written test method and operators or analysts who have qualified on the specific test method utilized. All methods should be validated and analysts qualified by the same process.

Validation Process

The process described below uses validating an ICP as an example. For many other instruments there is no dilution step so step 2 is ignored.

0) Take 1 sample from validation set, dilute, and run on ICP to ensure that sample falls within the desired validation range. Ideally it is within the center of the range.
   a) If it does not fall within the range do not continue with the validation process. Repeat step 0 with new validation set until the set falls within range.
1) 1 sample diluted once and run seven times.
2) 1 sample spilt into 7 different samples, each one diluted separately, and run.
3) 7 separate samples sampled by an operator within a steady state run period, each one diluted separately and run.

Complete steps 1-3 with both the master and the second with the same validation sample set.

Compare data in the attached validation spreadsheet to ensure f-test is passed and determine the confidence interval. Confidence intervals are to be reported in absolute values (not percentages) for a published range.

Qualification Process

Complete step 0 of validation plan. If sample falls within range, complete step 3 of validation plan.

Compare data in the attached validation spreadsheet to ensure f-test is passed and confidence interval falls within published range.

OAG

There are different OAG products for the analytical team to deliver.

1. Analytical Validation, Confidence Intervals, and OAG Spec Level Reporting
    a. A full data set of information for a comprehensive understanding.
    b. Format detailed in immediate section below
2. Analytical Validation Roll Up
    a. Meant as an executive level presentation of data.
        i. Clean, concise, to the point.
    b. For each test method should include the range, CI, MDL, and Op Spec
    c. Analyst Validation and Qualification (Illustrated in the chart below)
    d. This should simply be a chart of the test method (specific to type and range), the master, notebook number and page with master validation set, and qualified analysts with qualification notebook number and page. Sample layout:

Test Method	Master	Validation Notebook Number:Page	Qualified Analyst	Qualification Notebook Number:Page

Analytical Validation, Confidence Intervals, and OAG Spec Level Reporting

1. Submit a one page document for all analytical validation data. Should follow the following format
    a. Columns are each of the tests or elements
    b. Rows are as follows:
        i. Range
        ii. Instrument Confidence Interval for range
        iii. Dilution Confidence Interval for range (where applicable)
        iv. Sample Confidence Interval for range
        v. Process Operation Specs for range
            1. OAG formatting compared to Sample Confidence Interval
        vi. Customer Specs for above range
            1. OAG formatting compared to Sample Confidence Interval
    c. Areas that are not applicable are to be greyed out.
    d. Areas that are not finished are to be left blank.
2. Label all units.
    a. Avoid ppm
        i. Specifically indicate mg/kg per reporting line.

3. Significant figures.
    a. One sig fig is the rule for confidence intervals.
        i. Must round up.
    b. Remove zeros and multiple digit values in confidence intervals.
        i. For example change the value from 0.002343283728 to 0.002 mg/kg.
4. Other numbers to report with corresponding confidence intervals
    a. Add a line for range of values tested that resulted in each confidence interval
    b. Add a line to indicate instrument MDL values
5. Identify specs

    NOTE: All OAG math must be conducted with confidence intervals that correspond to the range they are measuring.
    a. Customer specs
        i. Identify existing contractual specs if any
        ii. Identify customer specs that are currently being discussed
        iii. Understand that customer specs should be defined as USL and LSL (upper and lower spec level)
        iv. OAG highlighting
            1. Green: Target run level +/- 4 times the confidence interval is less than the USL and greater than the LSL
            2. Yellow: Target run level +/- 6 times the confidence interval is less than the USL or greater than the LSL
            3. Red: Target run level +/- 6 times the confidence interval is greater than the USL or less than the LSL
    b. Process Operation specs
        i. Identify existing specs UCL and LCL (upper and lower control limit)
        ii. Identify current average run levels and compare to target run levels
        iii. OAG highlighting
            1. Green: Target run level +/- 3 times the confidence interval is less than the UCL and greater than the LCL
            2. Yellow: Target run level +/- 5 times the confidence interval is less than the UCL or greater than the LCL
            3. Red: Target run level +/- 5 times the confidence interval is greater than the UCL or less than the LCL

