

Validation of the Method

Purpose: The purpose of this assignment is to address questions related to the validation of **EPA method 525.2**. You will need a copy of this method to complete many of the questions in this assignment.

Learning Outcomes:

At the end of this assignment students will be able to:

1. Explain the importance of method validation.
2. Identify the standard solutions generally used in a validation procedure, and explain why each is necessary.
3. Calculate the detection limit of a method for a specific analyte.

To demonstrate that the numbers obtained in our measurements are correct and can be trusted, the entire procedure used in their determination must be **validated**. This process assures that the concentrations of analyte that are determined are not the result of errors in the sample workup, an interferent or matrix effect that gives a false “positive” reading, a matrix effect that diminishes the signal, or an improper instrument response. Validation procedures for **EPA method 525.2** include *Quality Control* (Section 9.0), allowing the demonstration of method accuracy and precision, and *Method Performance* (Section 10.0) that tests the reproducible day-to-day operation of the instrument.

Q1. Define the terms accuracy and precision. What types of error are associated with each?

For a method as complex as **EPA 525.2**, error can be introduced during many different steps in the procedure, from sampling to sample preparation to instrumental analysis. The process of validation is time-consuming and tedious, but many possible sources of errors must be addressed and either eliminated or compensated for before the values you obtain have any “value”.

Section 3.0 of **EPA 525.2** supplies a list of definitions of terms important in the validation process. Before continuing, you should read through that section carefully so that you can separate your FRBs from your IRCs. Additionally, Section 4.0 describes the major sources of interferences for the method.

An important part of method validation is having appropriate blanks.

Q2. What is the general definition of a blank?

Q3. Explain the difference between a field reagent blank and a lab reagent blank. What is the purpose of each?

Q4. What are fortified solutions? What is the purpose of a fortified solution?

Q5. The purpose of internal standards and surrogate analytes was covered in the section on sample preparation. Review these materials and describe how internal standards and surrogate analytes differ? What is the purpose of an internal standard and a surrogate?

Q6. Why is decafluorotriphenylphosphine (DFTPP) used in the method?

Q7. Table 3.1 gives the results of DFTPP performance tests. What are the three parts to the performance test?