

## Worksheet 16: Final Exam Review IV

1. What is quality assurance?

**Solution:** Quality assurance is what we do to get the right answer for our purpose.

2. What is an external standard? How does it relate to a calibration curve?

**Solution:** An external standard is a known solution of analyte that does not involve the unknown solution. Calibration curves are generated using external standards.

3. What are the different types of data and what are they used for?

**Solution:**

- Raw data are measurements obtained from experiment and are the basis for the other types of data.
- Treated data are created by performing some sort of procedure on the raw data. An example is obtaining raw absorbance values from a spectrophotometric experiment and using them to calculate concentrations.
- Results are the reported values that result from applying statistics to the treated data.

4. What do the use objectives for an analysis tell us?

**Solution:** Use objectives state the purpose for which results will be used. The text offers the example of manufacturing a drug that has a lethal dose just higher than the therapeutic dose compared to making spaghetti sauce. In one case the stakes are much higher and the use objectives reflect that.

5. What type of specifications might you want to make for an analytical procedure?

**Solution:** Examples include sampling requirements, accuracy and precision, rate of false results, selectivity, sensitivity, acceptable blank values, recovery of fortification, calibration checks, and quality control samples.

6. What is the difference between a false positive and a false negative?

**Solution:** A false positive reports that something is true when it is not, and a false negative reports that something is false when it is not. For example, a false positive in terms of a blood alcohol test would say that the concentration was above the legal limit when it actually is below, while a false negative would say the concentration was below the limit when it was actually above.

7. What is the difference between selectivity and sensitivity?

**Solution:** Selectivity means the ability to distinguish analyte from other species in the sample, while sensitivity is the capability of responding reliably and measurably to changes in analyte concentration.

8. How can we determine the sensitivity for an analytical procedure that generates a calibration curve?

**Solution:** The sensitivity will be the slope of the calibration curve.

9. What are the differences between method blanks, reagent blanks, and field blanks?

**Solution:** A method blank contains all the sample components except analyte, and it is taken through all the steps (the method) of the analytical procedure. A reagent blank is similar, but it has not been subjected to all sample preparation techniques, it is mainly testing the reagents themselves, not the preparation or method. A field blank is like a method blank, but it has also been exposed to the site of sampling.

10. In the context of a sample, what does the matrix refer to?

**Solution:** The matrix is everything in the sample other than the analyte.

11. What does adding a spike to your solution entail? How do you calculate spike recovery?

**Solution:** A spike or fortification is the addition of a known quantity of an analyte to a sample to test whether the response to the spike is the same as expected from a calibration curve. You calculate spike recovery using the equation

$$\% \text{recovery} = \frac{C_{\text{spiked sample}} - C_{\text{unspiked sample}}}{C_{\text{added}}} \times 100$$

12. What is the use of a performance test sample?

**Solution:** A performance test sample is provided to an analyst as an unknown and the analyst's results can be compared to the known value to test the performance of the analyst and the method.

13. What is method validation?

**Solution:** Method validation is the process of proving that an analytical method is acceptable for its intended purpose.

14. What are some ways to demonstrate the accuracy of a method?

**Solution:**

- Analyze a certified reference material in a matrix similar to that of your unknown.
- Compare results from two or more different analytical methods.
- Analyze a blank sample spiked with a known addition of analyte.
- Use the method of standard additions if you cannot prepare a blank with the same matrix as the unknown.

15. What are the implications of the Horwitz Trumpet?

**Solution:** The Horwitz Trumpet shows the typical relationship between concentration and the coefficient of variation (the standard deviation divided by the mean). As concentration decreases, the curve flares widely, demonstrating the greater uncertainty as concentration decreases. Basically, the less there is of something the harder it is to measure reliably.

16. How do you calculate the lower limit of quantitation for an analytical method? What about the detection limit?

**Solution:** Lower limit of quantitative  $\equiv \frac{10s}{m}$ , where  $s$  is the standard deviation of low-concentration samples, and  $m$  is the slope of the calibration curve. Detection limit  $\equiv \frac{3s}{m}$ .

17. Under what conditions is it necessary to use the method of standard additions?

**Solution:** Standard additions is especially appropriate when the sample composition is unknown or complex and affects the analytical signal. It is called a matrix effect when the sample matrix changes the analytical signal.

18. What is an internal standard and when should it be used?

**Solution:** An internal standard is a known amount of a compound different from the analyte that is added to the unknown. These standards are particularly useful when the quantity of sample analyzed or instrument response varies slightly from run to run, so the response factor  $F$  can be used to determine analyte concentration by comparing it to the known internal standard concentration. The equation for doing this is

$$\frac{\text{Signal from analyte}}{\text{Concentration of analyte}} = F \left( \frac{\text{signal from standard}}{\text{concentration of standard}} \right)$$