

Chem 231 Quiz Number 3 - Solutions
Mar. 11, 2013

A. Short Answer/Multiple Choice Section (2 points each)

1. List one modification from the GCs that we are using in the lab if we wish to use these GCs for analysis of permanent gases. Modification = 1) cryogenic cooling, 2) use of trap injector, 3) gas injection valve

2. In sample analysis by GC, why is it often necessary to have sample clean up steps even if the solutes of interest elute without co-eluting interferences?

reason = other compounds in the sample (e.g. non-volatile compounds) can damage the injector, column or detector

3. The key advantage in using SPME with GC in the analysis of flower volatiles is:

a) faster GC analyses

b) no extraction of flowers is needed

c) better precision

d) can use isothermal GC method

4. Which of the following errors can result in permanent damage to the SPME fibers:

a) use with certain chlorinated solvents

b) forgetting to sheath the fiber when piercing a septum

c) setting the injector to too high of a temperature

d) all of the above

B. Calculations/Longer Answer. (6 pts)

1. A chemist wants to analyze roasted coffee beans for phenols containing carboxylic acids such as vanillic acid (these have pK_a values for the carboxylic acids of around 4). She is using HPLC with a C18 column and an isocratic eluent of 45% 0.01 M formic acid in water and 55% acetonitrile. To develop a method, she dissolves the carboxylic acid containing phenols in 100% acetonitrile and initially uses a 5 μ L injection volume. The peaks are well separated, have a reasonable shape (a little tailing), and are fairly narrow. To improve sensitivity, she decides to increase the injection volume to 50 μ L. Suddenly, the peak shapes look very bad (some splitting of peaks, more tailing, and much broader peaks).

a) Describe what caused the change in going to a larger injection volume

Too large of a volume can cause broadening and non-Gaussian peak shapes due to either a sample overload or a volume overload. In the case of a volume overload, peak broadening can occur because the time needed to inject the sample is approaching or greater than the peak widths (in time units). In this case, a large volume is suspect because the sample solvent (100% acetonitrile) is not retentive at all.

b) Describe how the method could be modified to allow large volume injection of carboxylic acid containing phenols without distorted peak shapes.

To avoid poor peak shapes and peak broadening, the compound should be injected in a solvent that leads to strong retention. This could be done by using 0.01 M formic acid as a solvent (with only enough methanol to get compounds to dissolve).