Chapter 1

1. (a) A qualitative and a quantitative analysis is the best choice because we need to determine the identify of the possible contaminants and determine if their concentrations are greater than the expected background levels.

   (b) A forged work of art often contains compounds that are not present in authentic materials or contains a distribution of compounds that do not match the authentic materials. Either a qualitative analysis (to identify a compound that should not be present in authentic materials) or a quantitative analysis (to determine if the concentrations of compounds present do not match the distribution expected in authentic materials) is appropriate.

   (c) Because we are interested in detecting the presence of specific compounds known to be present in explosive materials, a qualitative analysis is the best choice.

   (d) A compound's structure is one of its characteristic properties; a characterization analysis, therefore, is the best approach.

   (e) In searching for a new acid–base indicator we are seeking to improve the performance of an existing analytical method, which requires a fundamental analysis of the method's properties.

   (f) A quantitative analysis is used to determine if an automobile emits too much carbon monoxide.

2. Answers to this problem will vary, but here is a list of important points that you might address:

   The goal of this research is to develop a fast, automated, and real-time instrumental method for determining a coffee's sensory profile that yields results similar to those from trained human sensory panels.

   One challenge the authors have to address is that a human sensory panelist reports results on a relative scale, typically 0–10, for characteristics that are somewhat arbitrary: What does it mean, for example, to say that a coffee is bitter? An instrumental method, on the other hand, reports results on an absolute scale and for a clearly defined signal; in this case, the signal is a raw count of the number of ions with a particular mass–to-charge ratio. Much of the mathematical processing described by the authors is used to transform the instrumental data into a relative form and to normalize the two sets of data to the same relative scale.

   The instrumental technique relies on gas chromatography equipped with a mass spectrometer as a detector. The specific details of the instrument are not important, but the characteristics the authors describe—low fragmentation, high time resolution, broad linear dy-

See Chapter 12 for a discussion of gas chromatography and for detection using a mass spectrometer.

namic range—are important. When a species enters a mass spectrometer it is ionized (the PTR—proton transfer reaction—in PTR-MS simply describes the method of ionization) and the individual ions, being unstable, may decompose into smaller ions. As a roasted coffee has more than 1000 volatile components, many of which do not contribute to the sensory profile, the authors wish to limit the number of ions produced in the mass spectrometer. In addition, they want to ensure that the origin of each ion traces back to just a small number of volatile compounds so that the signal for each ion carries information about a small number of compounds. Table 1, for example, shows that the 16 ions monitored in this study trace back to just 32 unique volatile compounds, and that, on average, each ion traces back to 3–4 unique volatile compounds with a range of one to eight.

The authors need high time resolution so that they can monitor the release of volatile species as a function of time, as seen in Figure 1, and so that they can report the maximum signal for each ion during the three-minute monitoring period. A rapid analysis also means they can monitor the production of coffee in real time on the production line instead of relying on a lengthy off-line analysis completed by a sensory panel. This is advantageous when it comes to quality control where time is important.

For a discussion of quality control and quality assurance, see Chapter 15.

A broad linear dynamic range simply means there is a linear relationship between the measured signal and the concentration of the compounds contributing to that signal over a wide range of concentrations. The assumption of a linear relationship between signal and concentration is important because a relative change in concentration has the same affect on the signal regardless of the original concentration. A broad range is important because it means the signal is sensitive to a very small concentration of a volatile compound and that the signal does not become saturated, or constant, at higher concentrations of the volatile compound; thus, the signal carries information about a much wider range of concentrations.

For a discussion of the relationship between signal and concentration, see Chapter 5.

To test their method, the authors divide their samples into two sets: a training set and a validation set. The authors use the training set to build a mathematical model that relates the normalized intensities of the 16 ions measured by the instrument to the eight normalized relative attributes evaluated by members of the sensory panel. The specific details of how they created the mathematical model are not important here, but the agreement between the panel's sensory profile and that predicted using the instrumental method generally is very good (see Figure 3; note that the results for Espresso No. 5 and No. 11 show the least agreement).

Any attempt to create a model that relates one measurement (results from the sensory panel) to a second measurement (results from the instrumental analysis) is subject to a number of limitations, the most important of which is that the model works well for the data set used to build the model, but that it fails to work for other samples. To test the more general applicability of their model—what they refer to as a robust model—the authors use the model to evaluate the data in their validation set; the results, shown in Figure 4, suggest that the can apply their model both to coffees of the same type, but harvested in a different year, and to coffees of a different type.

See Chapter 14 for a discussion of robustness and other ways to characterize an analytical method.