

Qualitative tests may be performed by selective chemical reactions or with the use of instrumentation. The formation of a white precipitate when adding a solution of silver nitrate in dilute nitric acid to a dissolved sample indicates the presence of a halide. Certain chemical reactions will produce colors to indicate the presence of classes of organic compounds, for example, ketones. Infrared spectra will give “fingerprints” of organic compounds or their functional groups.

A clear distinction should be made between the terms *selective* and *specific*:

- A *selective* reaction or test is one that can occur with other substances but exhibits a degree of preference for the substance of interest.
- A *specific* reaction or test is one that occurs *only* with the substance of interest.

Unfortunately, very few reactions are truly specific but many exhibit selectivity. Selectivity may be also achieved by a number of strategies. Some examples are:

Few analyses are specific.
Selectivity may be achieved
through proper preparation and
measurement.

1.2 QUALITATIVE AND QUANTITATIVE ANALYSIS: WHAT DOES EACH TELL US?

- Sample preparation (e.g., extractions, precipitation)
- Instrumentation (selective detectors)
- Target analyte derivatization (e.g., derivatize specific functional groups)
- Chromatography, which separates the sample constituents

For quantitative analysis, the typical sample composition will often be known (we know that blood contains glucose), or else the analyst will need to perform a qualitative test prior to performing the more difficult quantitative analysis. Modern chemical measurement systems often exhibit sufficient selectivity that a quantitative measurement can also serve as a qualitative measurement. However, simple qualitative tests are usually more rapid and less expensive than quantitative procedures. Qualitative analysis has historically been composed of two fields: inorganic and organic. The former is usually covered in introductory chemistry courses, whereas the latter is best left until after the student has had a course in organic chemistry.

In comparing qualitative versus quantitative analysis, consider, for example, the sequence of analytical procedures followed in testing for banned substances at the Olympic Games. The list of prohibited substances includes about 500 different active constituents: stimulants, steroids, beta-blockers, diuretics, narcotics, analgesics, local anesthetics, and sedatives. Some are detectable only as their metabolites. Many athletes must be tested rapidly, and it is not practical to perform a detailed quantitative analysis on each. There are three phases in the analysis: the fast-screening phase, the identification phase, and possible quantification. In the fast-screening phase, urine samples are rapidly tested for the presence of classes of compounds that will differentiate them from “normal” samples. Techniques used include immunoassays, gas chromatography–mass spectrometry, and liquid chromatography–mass spectrometry. About 5% of the samples may indicate the presence of unknown compounds that may or may not be prohibited but need to be identified. Samples showing a suspicious profile during the screening undergo a new preparation cycle (possible hydrolysis, extraction, derivatization), depending on the nature of the compounds that have been detected. The compounds are then identified using the highly selective combination of



Quantitative analysis

Qualitative analysis

gas chromatography/mass spectrometry (GC/MS). In this technique, complex mixtures are separated by gas chromatography, and they are then detected by mass spectrometry, which provides molecular structural data on the compounds. The MS data, combined with the time of elution from the gas chromatograph, provide a high probability of the presence of a given detected compound. GC/MS is expensive and time consuming, and so it is used only when necessary. Following the identification phase, some compounds must be precisely quantified since they may normally be present at low levels, for example, from food, pharmaceutical preparations, or endogenous steroids, and elevated levels must be confirmed. This is done using quantitative techniques such as spectrophotometry or gas chromatography.

This text deals principally with quantitative analysis. In the consideration of applications of different techniques, examples are drawn from the life sciences, clinical chemistry, environmental chemistry, occupational health and safety applications, and industrial analysis.

We describe briefly in this chapter the analytical process. More details are provided in subsequent chapters.

1.3 Getting Started: The Analytical Process

The general analytical process is shown in Figure 1.1. The analytical chemist should be involved in every step. The analyst is really a problem solver, a critical part of the team deciding what, why, and how. The unit operations of analytical chemistry that are common to most types of analyses are considered in more detail below.

DEFINING THE PROBLEM—WHAT DO WE REALLY NEED TO KNOW? (NOT NECESSARILY EVERYTHING)

Before the analyst can design an analysis procedure, he or she must know what information is needed, by whom, for what purpose, and what type of sample is to be analyzed. As the analyst, you must have good communication with the client. This stage of an analysis is perhaps the most critical. The client may be the Environmental Protection Agency (EPA), an industrial chemist, an engineer, or your grandmother—each of which will have different criteria or needs, and each having their own understanding of what a chemical analysis involves or means. It is important to communicate in language that is understandable by both sides. If someone puts a bottle on your desk and asks, “What is in here?” or “Is this safe?”, you may have to explain that there are 10 million known compounds and substances. A client who says, “I want to know what elements are in here” needs to understand that at perhaps \$20 per analysis for 85 elements it will cost \$1700 to test for them all, when perhaps only a few elements are of interest.

Laypersons might come to analytical chemists with cosmetics they wish to “reverse engineer” so they can market them and make a fortune. When they realize it may cost a small fortune to determine the ingredients, requiring a number of sophisticated analyses, they always rethink their goals. On the other hand, a mother may come to you with a white pill that her teenage son insists is vitamin C and she fears is an illicit drug. While it is not trivial to determine what it is, it is rather straightforward to determine if it undergoes the same reactions that ascorbic acid (vitamin C) does. You may be able to greatly alleviate the concerns of an anxious mother.

The concept of “safe” or “zero/nothing” is one that many find hard to define or understand. Telling someone their water is safe is not for the analyst to say. All you

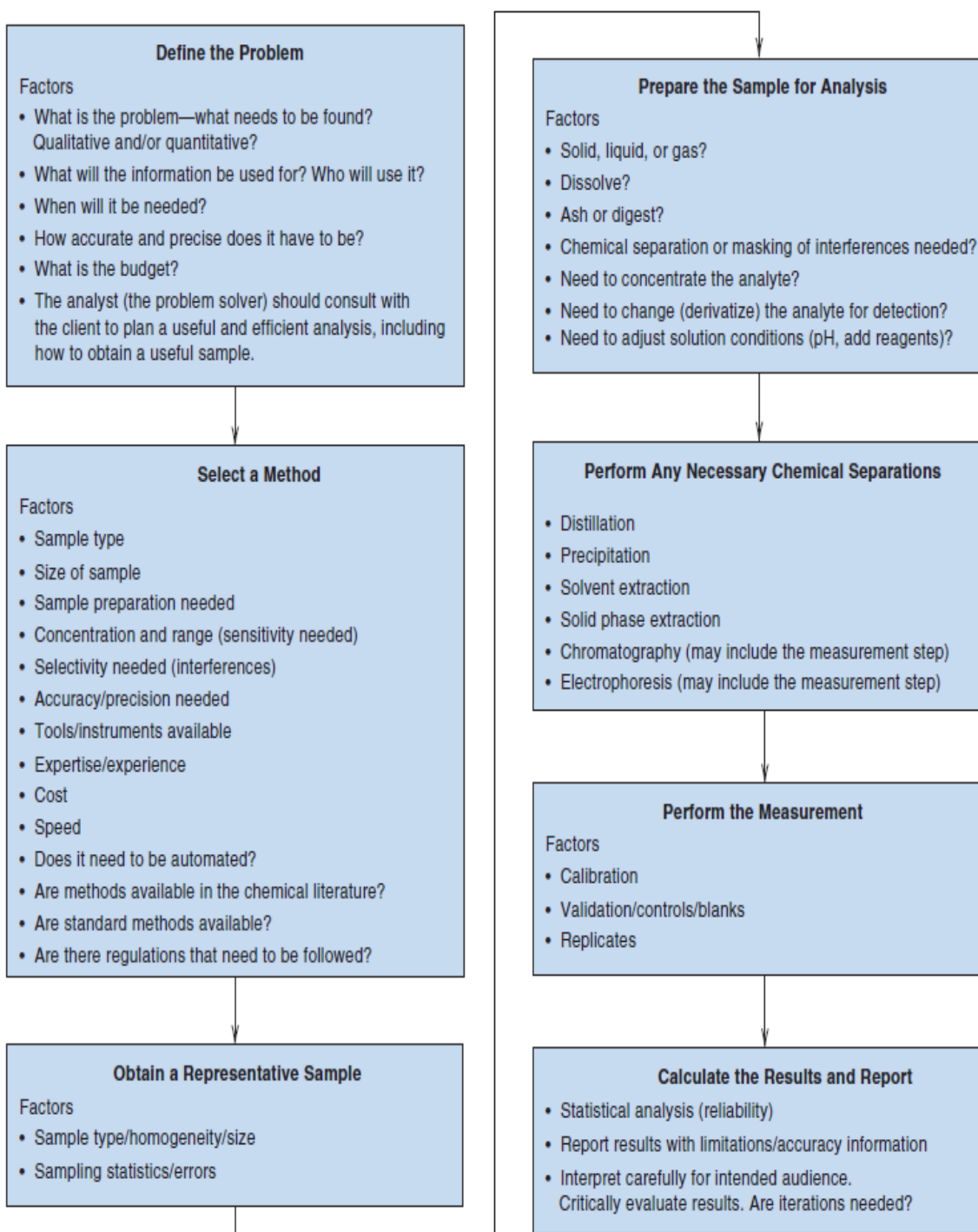


Fig. 1.1. Steps in an analysis.