

Chapter 0

The Analytical Process

Objectives

0-1 The Analytical Chemist's Job

0-2 General Steps in a Chemical Analysis

0-1: The Analytical Chemist's Job

- **Quantitative chemical analysis** is the measurement of *how much* of a chemical substance is present.
- **Qualitative analysis** tells us **what** is present.
- In quantitative analysis, the chemical measurement is only part of a process that includes asking a meaningful question, collecting a relevant sample, treating the sample so that the chemical of interest can be measured, making the measurement, interpreting the results, and providing a report.

0-2: General Steps in a Chemical Analysis

1. Formulating the question
2. Selecting analytical procedures
3. Sampling
4. Sample preparation
5. Analysis
6. Reporting and interpretation
7. Drawing conclusions

0-2: General Steps in a Chemical Analysis

Consider the task of **determining** the caffeine content of a chocolate bar.

2. Selecting Analytical Procedures

- First begin with a search of ***Chemical Abstracts*** for analytical methods.
- Looking for the key words “caffeine” and “chocolate,” one can find numerous articles in chemistry journals.
- Look for a procedure suitable for the equipment in your laboratory.
- Or, if necessary, devise new procedures to make the required measurements.

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3. Sampling

- Sampling is the process of selecting representative material to analyze.
- If you begin with a poorly chosen sample or if the sample changes between the time it is collected and the time it is analyzed, results are meaningless.
- “Garbage in—garbage out!”
- Is all chocolate the same? If you wanted to make broad statements about “caffeine in chocolate,” you would need to analyze a variety of chocolates.

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Sampling

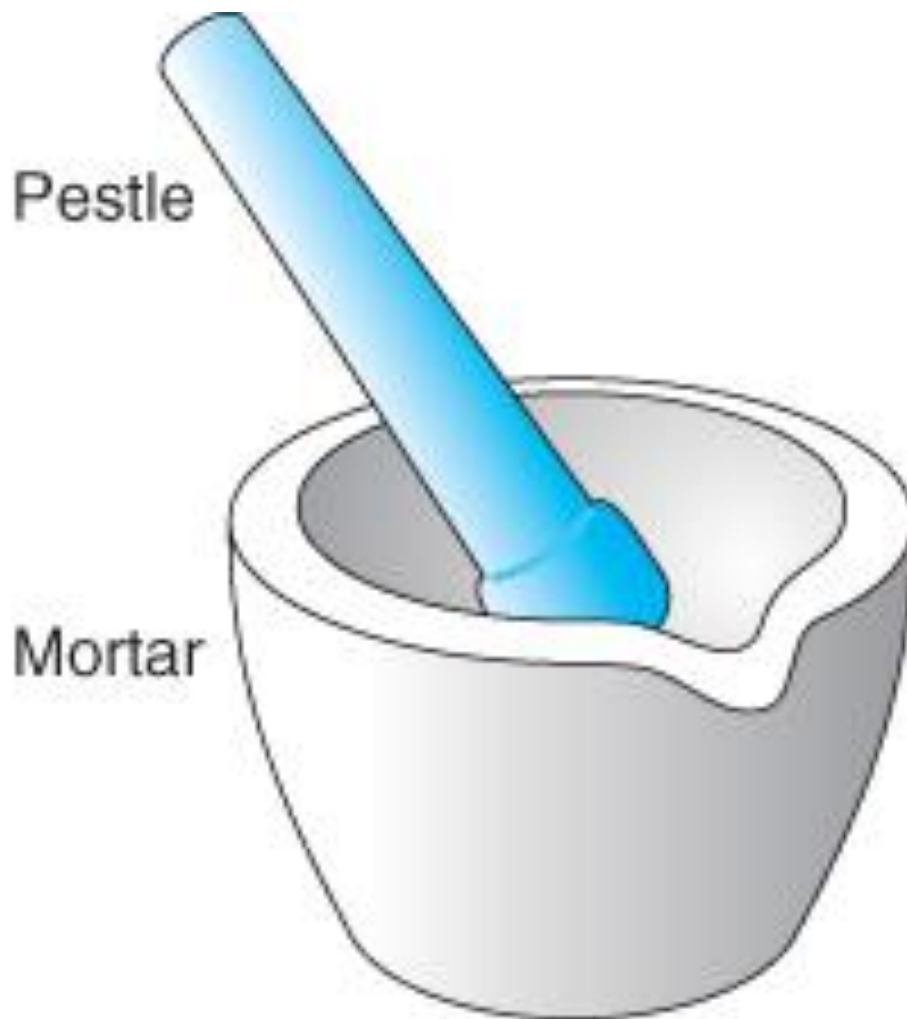
- A pure chocolate bar is fairly **homogeneous**, which means that its composition is the same everywhere. It might be safe to assume that a piece from one end has the same caffeine content as a piece from the other end.
- Chocolate with a macadamia nut in the middle is an example of a **heterogeneous** material—one whose composition differs from place to place.

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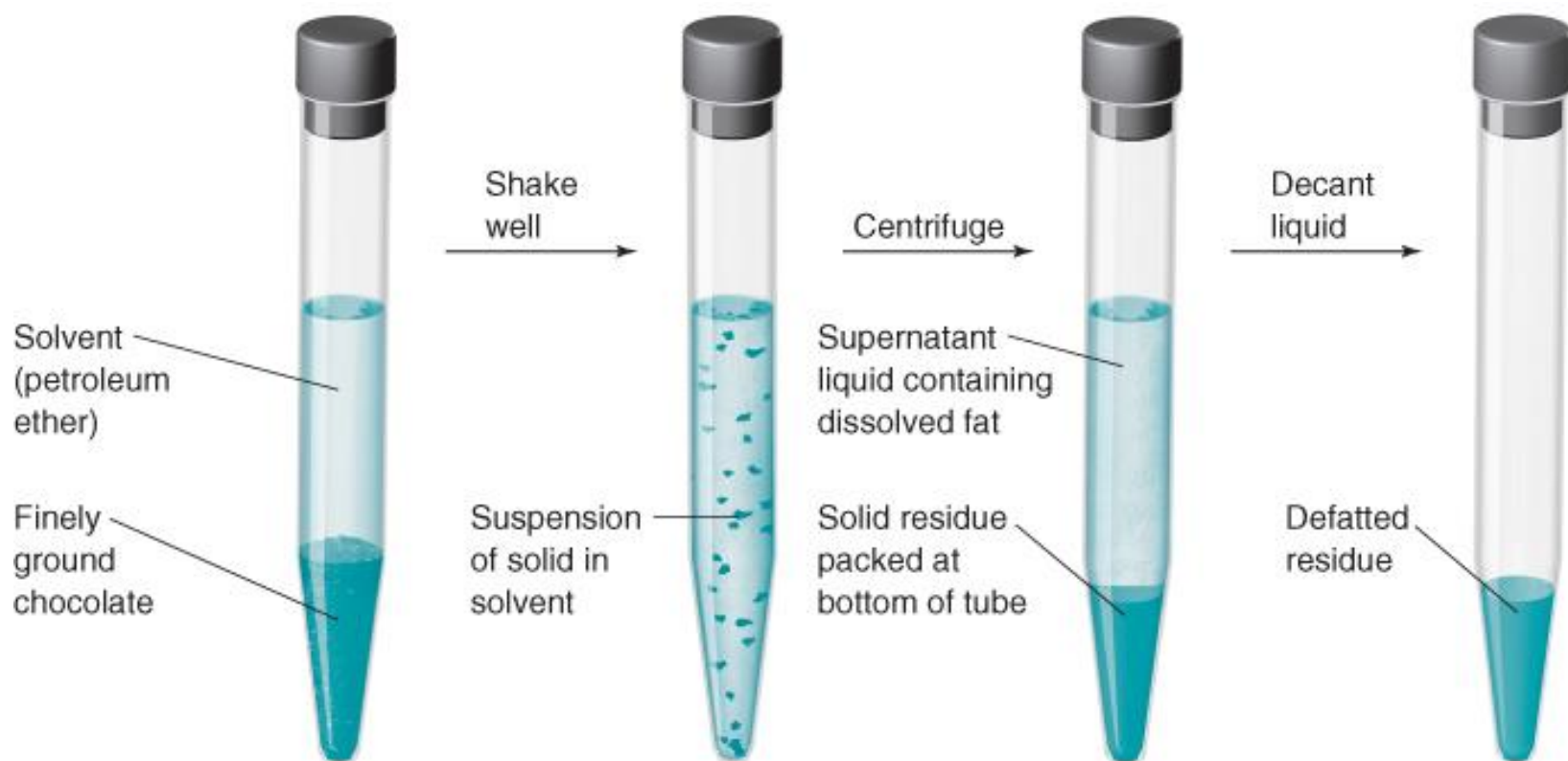
4. Sample preparation

- Converting a representative sample into a form suitable for analysis is called **sample preparation**, which usually means dissolving the sample.
- Samples with a low concentration of analyte may need to be concentrated prior to analysis.
- It may be necessary to remove or **mask** species that interfere with the chemical analysis.
- For a chocolate bar, sample preparation consisted of removing fat and dissolving the desired analytes.
- Fat was removed because it would interfere with chromatography.

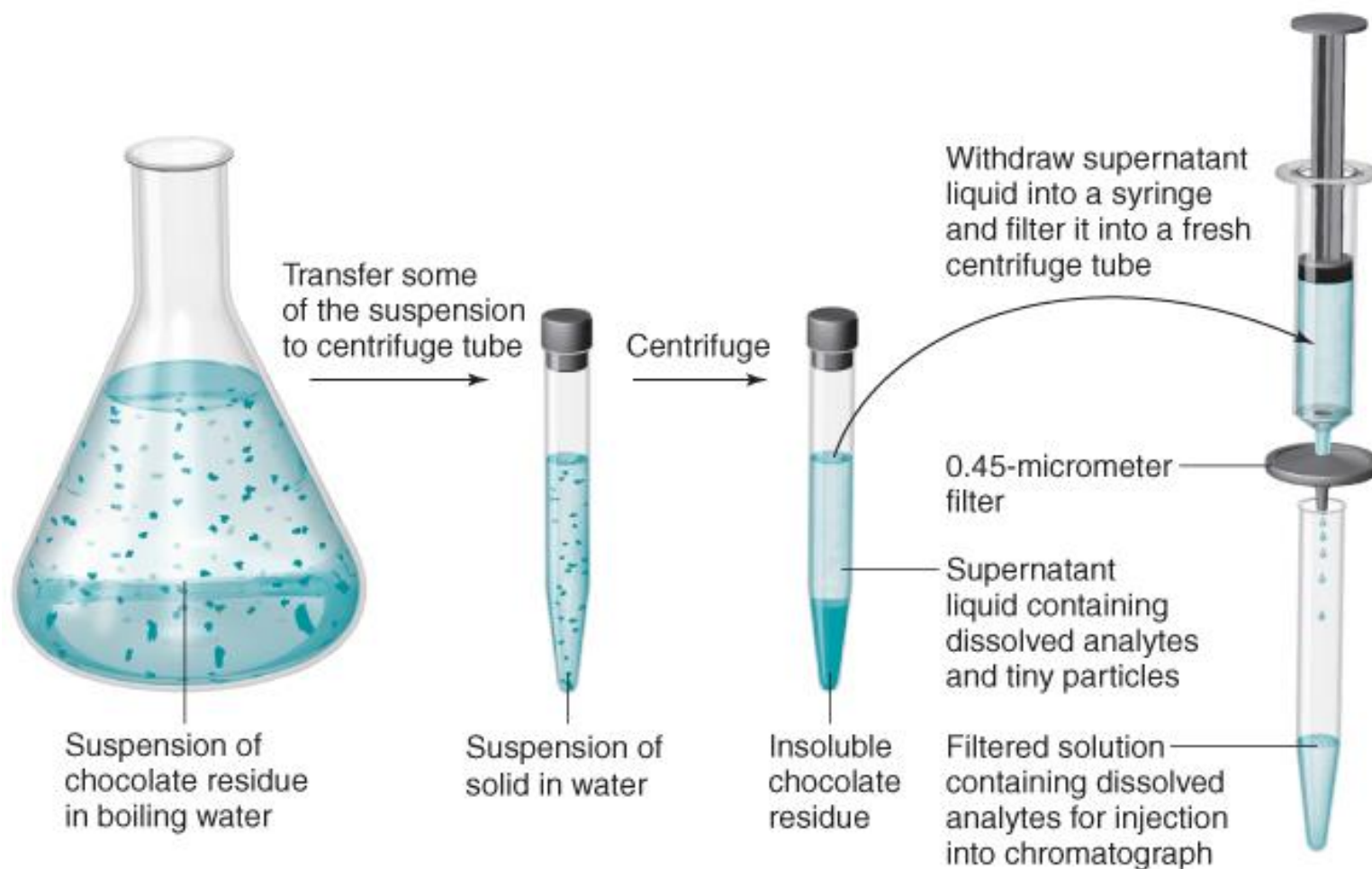
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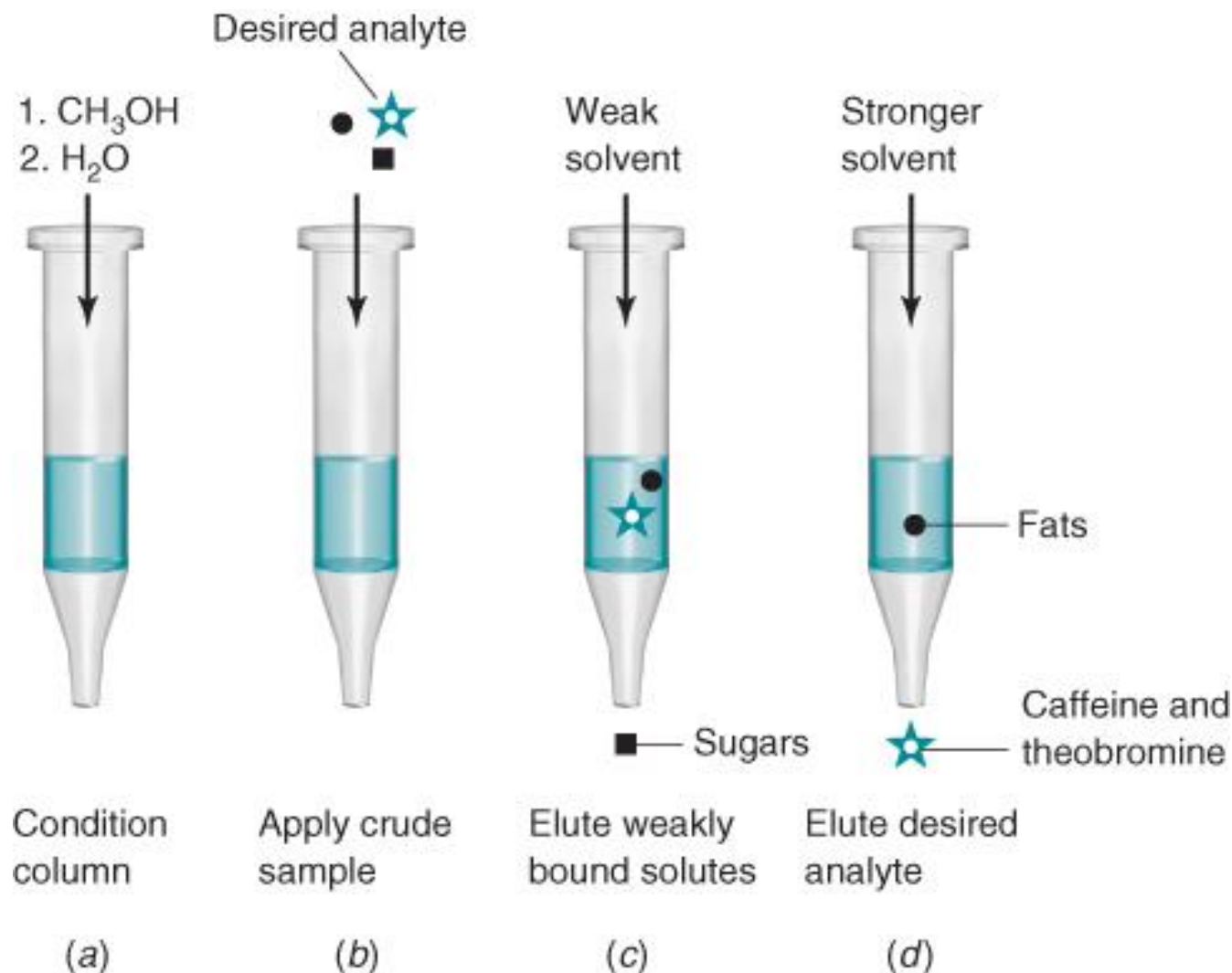


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Sample preparation

- **Solid-phase extraction (SPE)** simplifies sample preparation by separating some major interfering components of the mixture from the desired analytes.
- An SPE cartridge consists of a short, disposable column containing a chromatography solid phase that can clean the sample enough prior to performing chromatography on an expensive analytical column.
- Solid-phase extraction separates caffeine and theobromine from sugars and fats found in chocolate.

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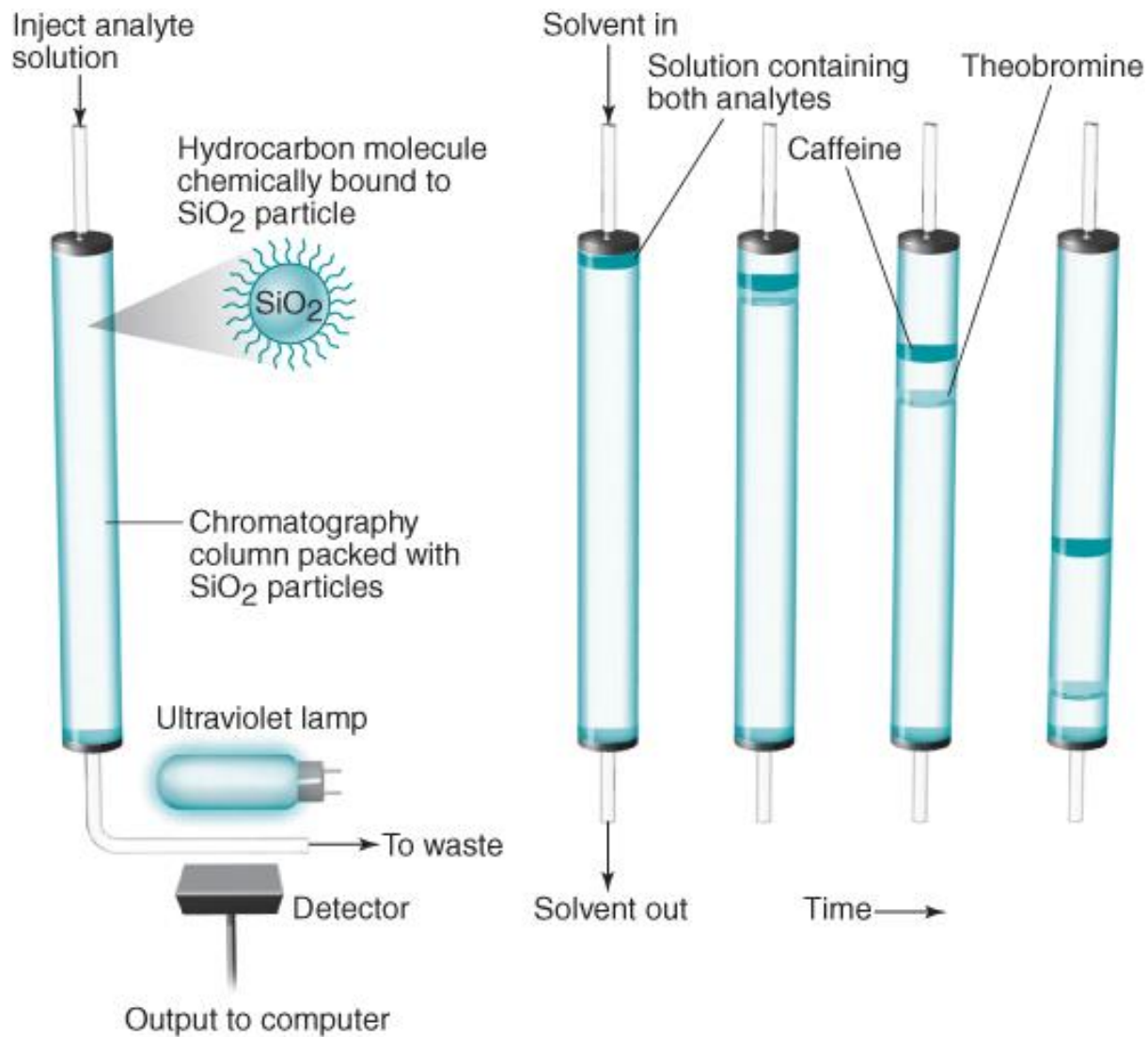
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5. Analysis

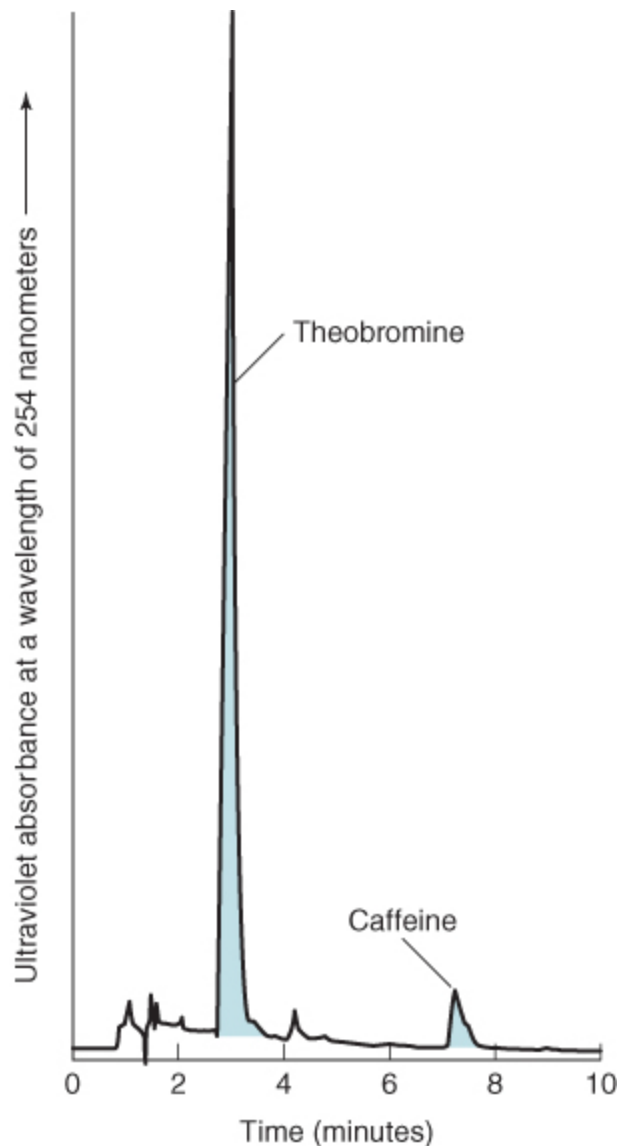
- Inject the solution into a **chromatography column**, which separates the analytes and measures the quantity of each.
- Measure the concentration of analyte in several identical **aliquots** (portions). The purpose of **replicate measurements** is to assess the variability (uncertainty) in the analysis and to guard against a gross error in the analysis of a single aliquot.
- The **uncertainty of a measurement** is as important as the measurement itself because it tells us how reliable the measurement is.

If necessary, use different analytical methods on similar samples to show

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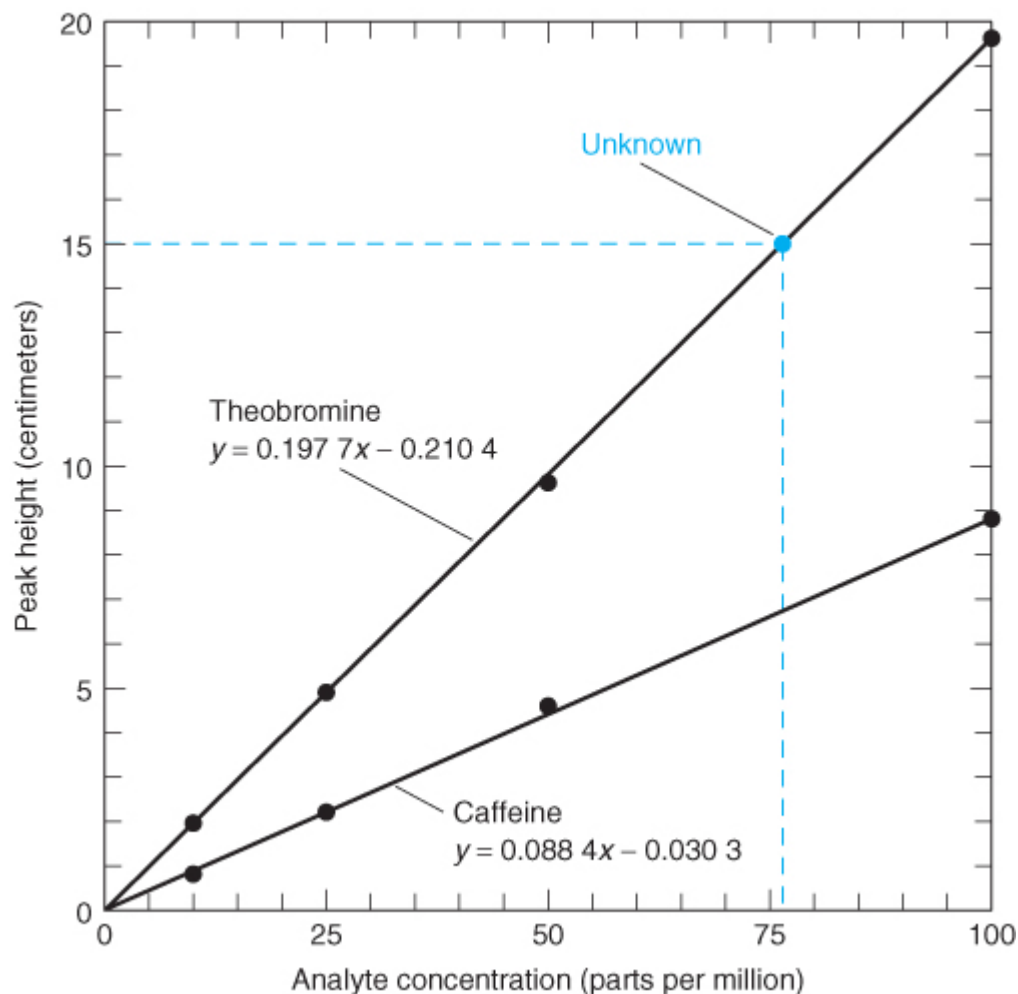


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Analysis: Calibration curves

- In general, analytes with equal concentrations give different detector responses.
- Therefore, the response must be measured for known concentrations of each analyte.
- A graph of detector response as a function of analyte concentration is called a **calibration curve**.
- To construct such a curve, **standard solutions** containing known concentrations of pure theobromine or caffeine are injected into the column, and the resulting peak heights measured.
- Straight lines drawn through the calibration points are then used to find the concentrations.

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6. Reporting and interpretation

- Deliver a clearly written, complete report of your results, highlighting any limitations that you attach to them.
- Be sure the report is appropriate for its intended audience.

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7. Drawing conclusions

- Once a report is written, the analyst might not be involved in what is done with the information, such as modifying the raw material supply for a factory or creating new laws to regulate food additives.
- The more clearly a report is written, the less likely it is to be misinterpreted by those who use it.

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TABLE 0-2 Caffeine content of beverages and foods

Source	Caffeine (milligrams per serving)	Serving size ^a (ounces)
Regular coffee	106–164	5
Decaffeinated coffee	2–5	5
Tea	21–50	5
Cocoa beverage	2–8	6
Baking chocolate	35	1
Sweet chocolate	20	1
Milk chocolate	6	1
Caffeinated soft drinks	36–57	12
Red Bull	80	8.2

a. 1 ounce = 28.35 grams

DATA SOURCES: http://www.holymtn.com/tea/caffeine_content.htm. Red Bull from <http://wilstar.com/caffeine.htm>.