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.

0-2: General Steps in a Chemical Analysis

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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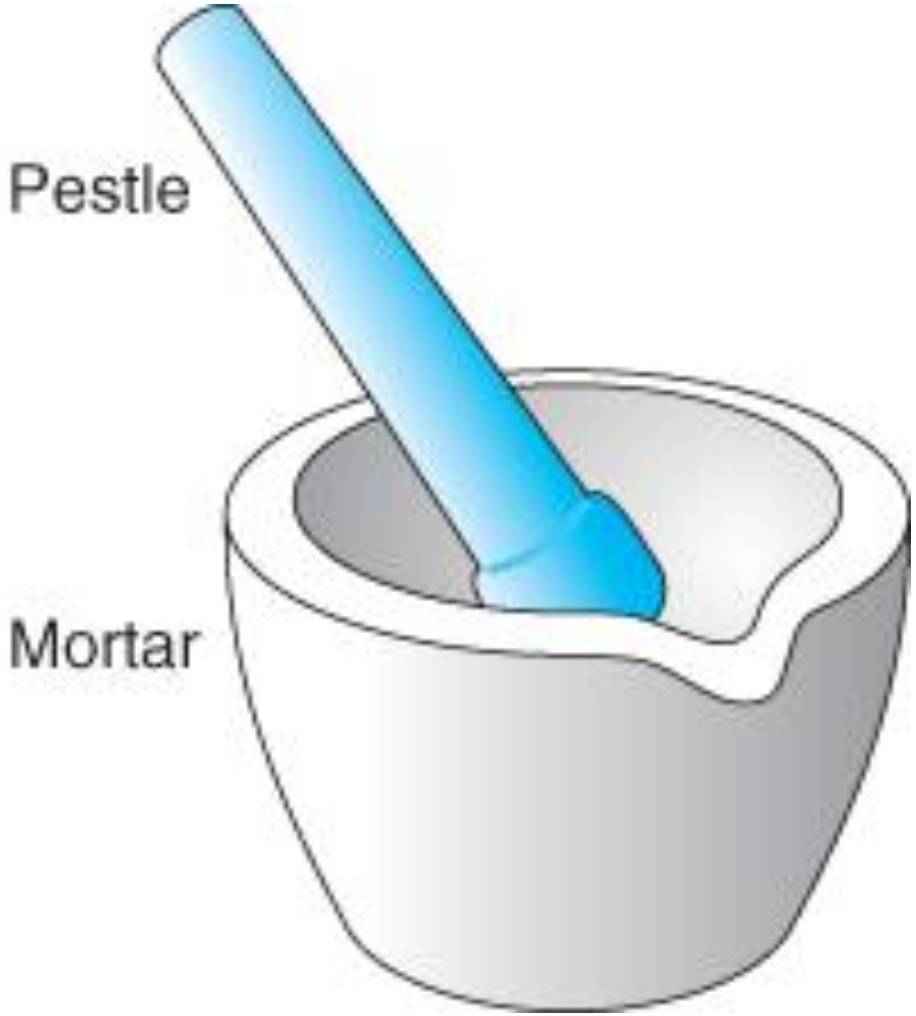

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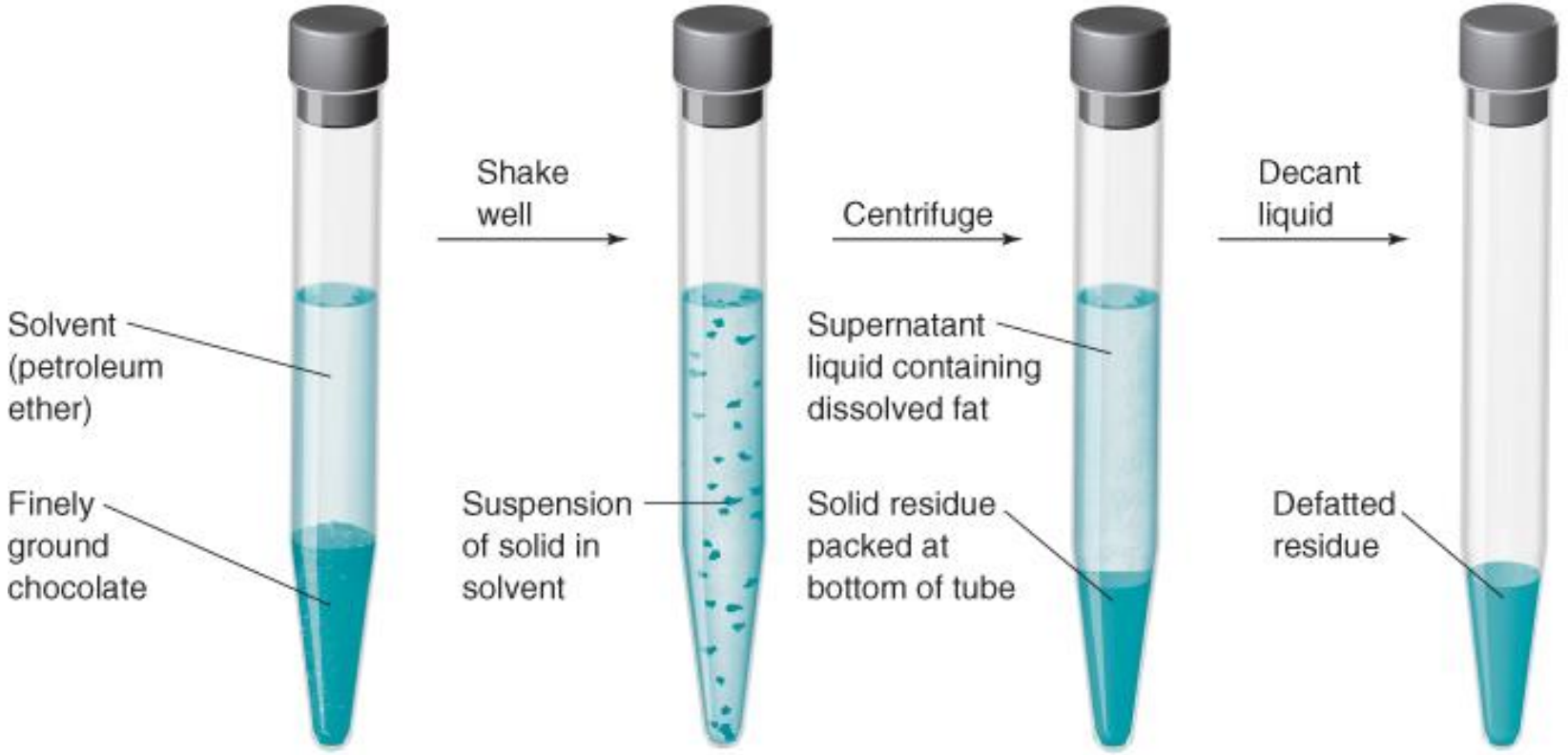

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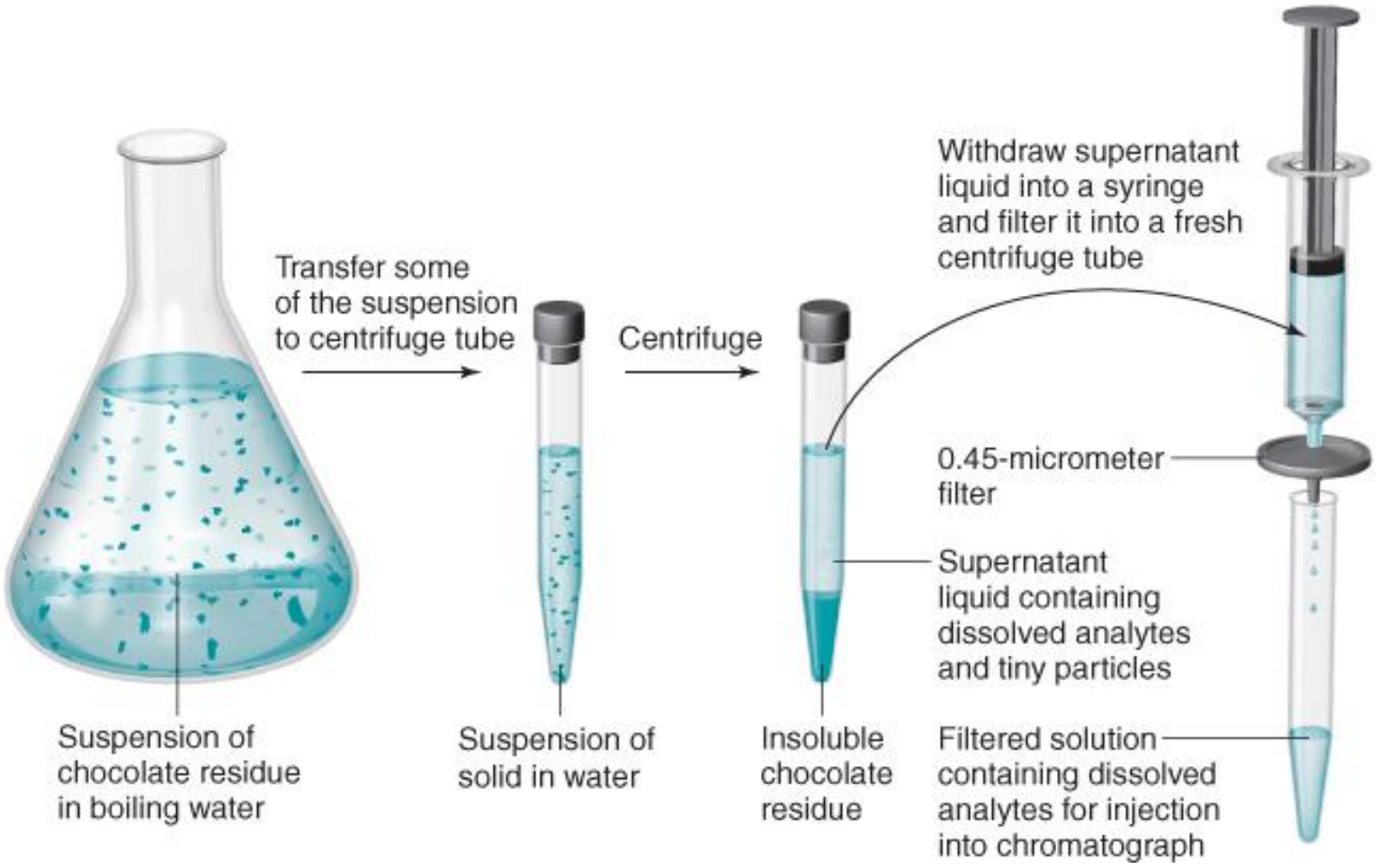

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Sample preparation

- **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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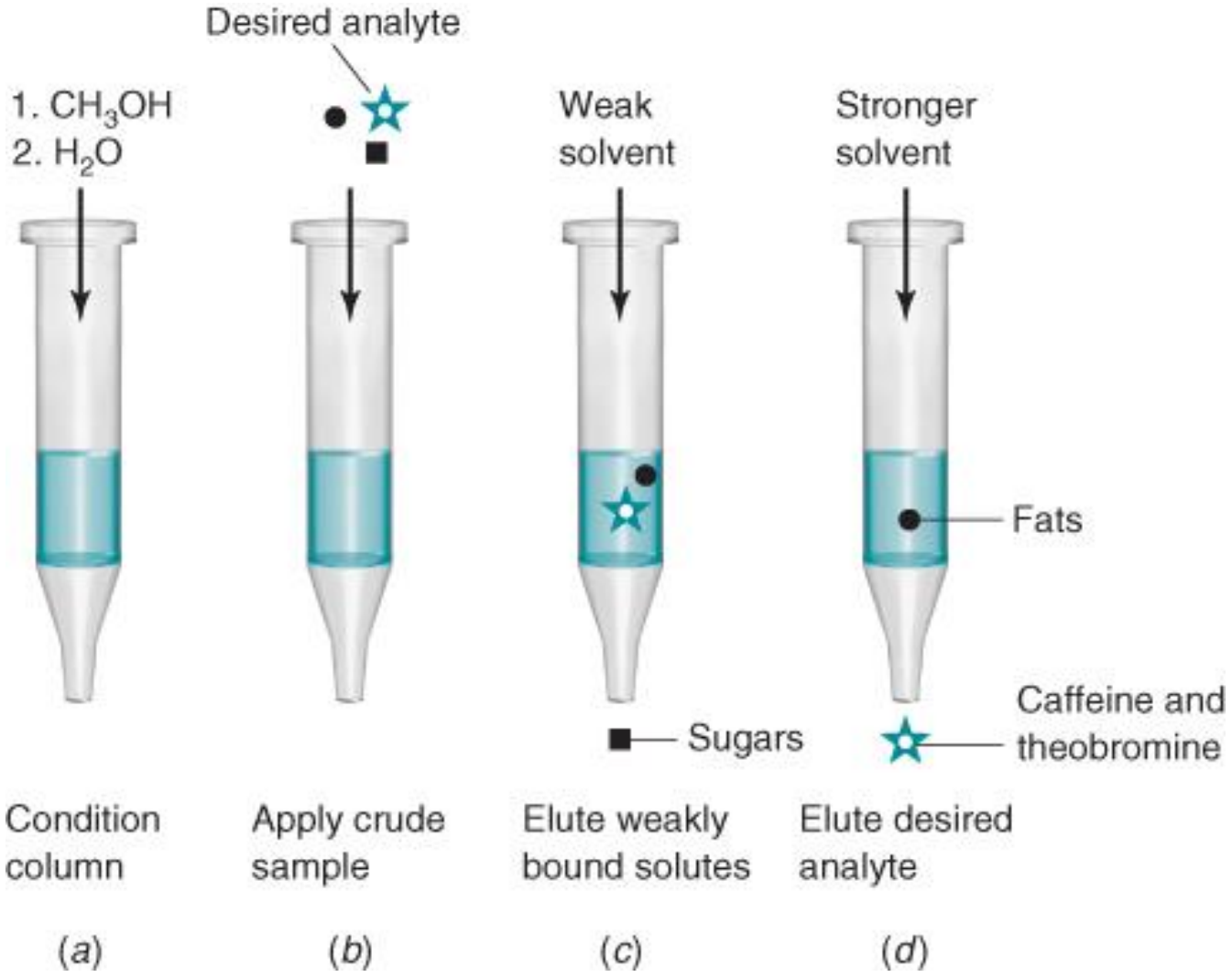

0-2: General Steps in a Chemical Analysis

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 that the choice of analytical method is not biasing the result.

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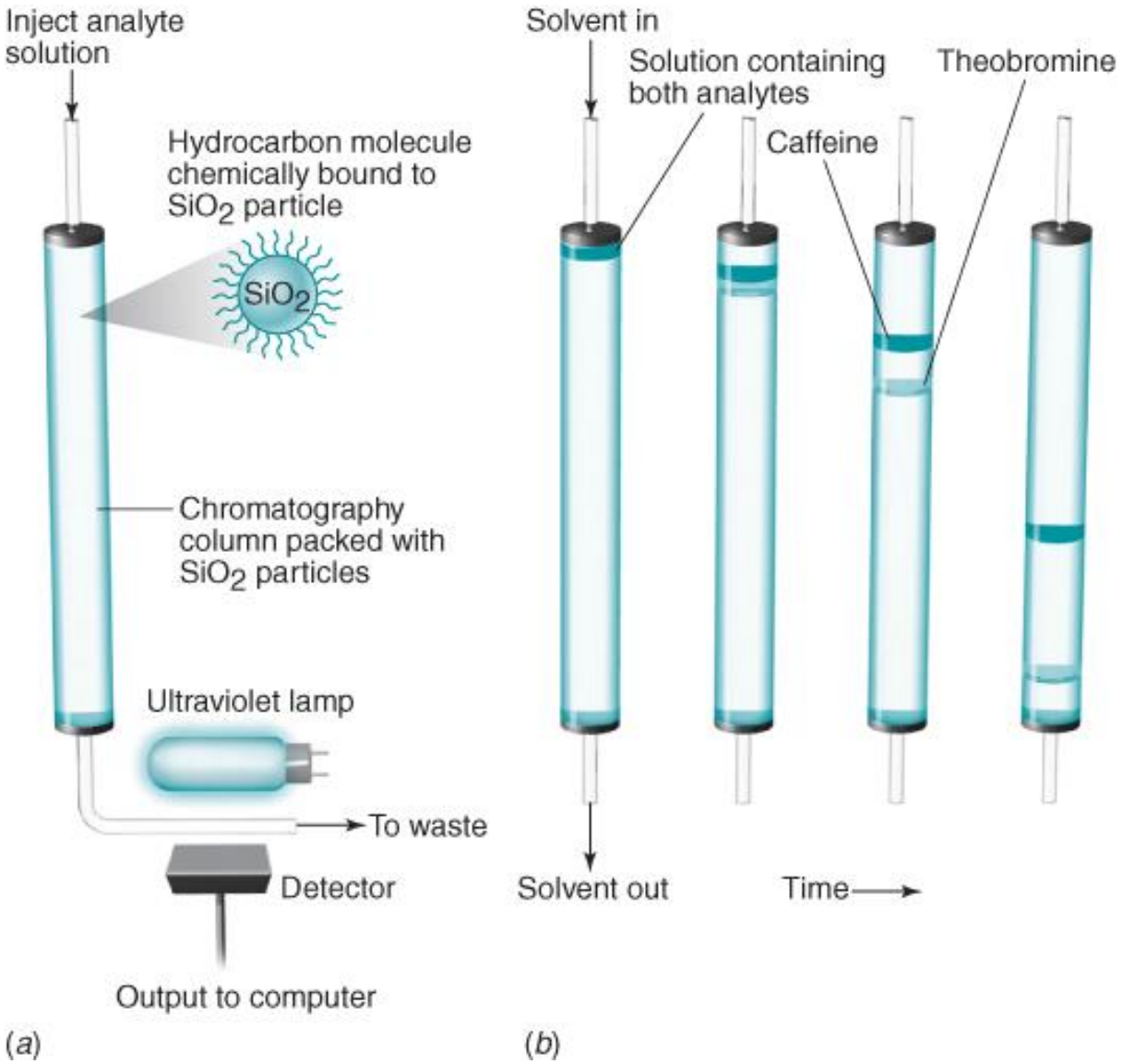

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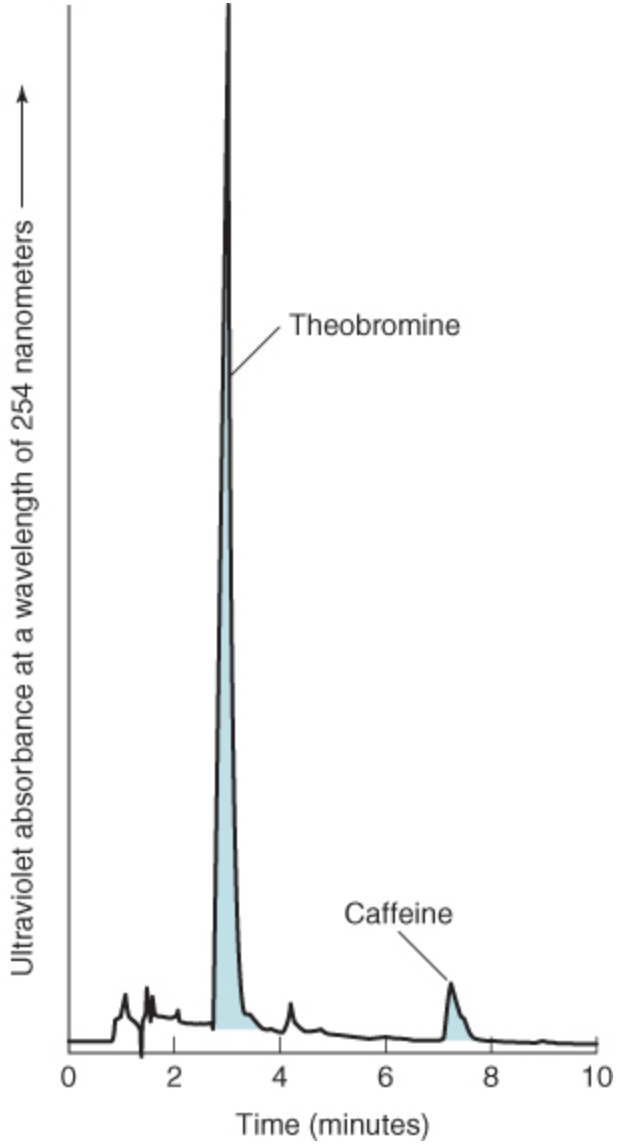

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Analysis: Calibration curves

- 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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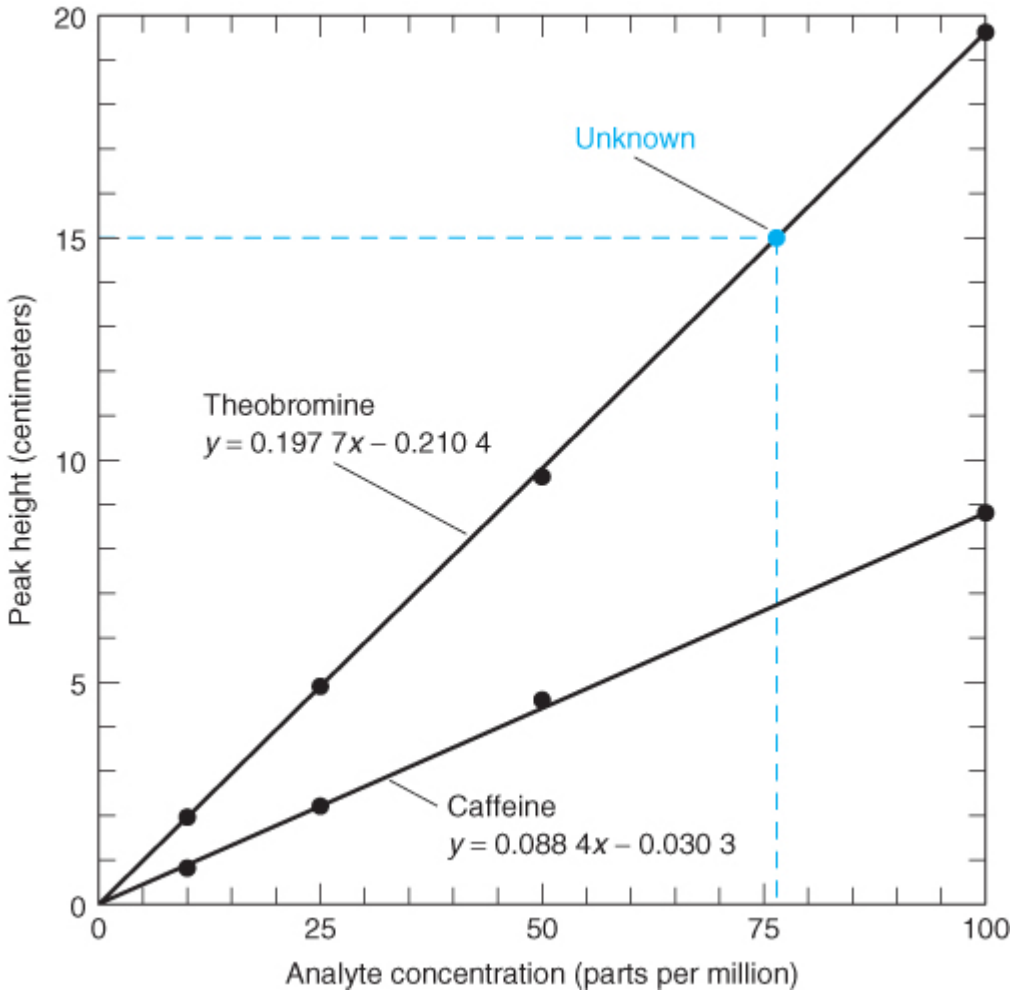

0-2: General Steps in a Chemical Analysis

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.

0-2: General Steps in a Chemical Analysis

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.

0-2: General Steps in a Chemical Analysis

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.