Analytical Chemistry

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Department of Medicinal Chemistry, School of Pharmacy, Shiraz University of Medical Sciences(SUMS); Feb 2025

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9th Edition

Douglas A. Skoog, Donald M. West, F. James Holler, Stanley R. Crouch

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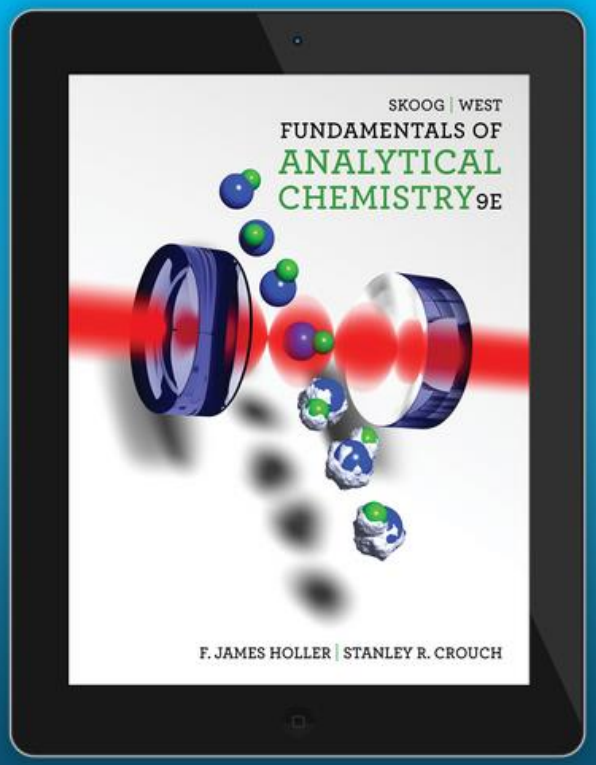

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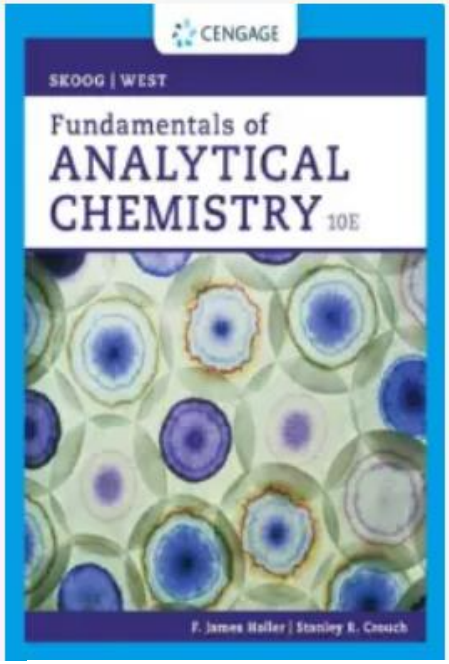

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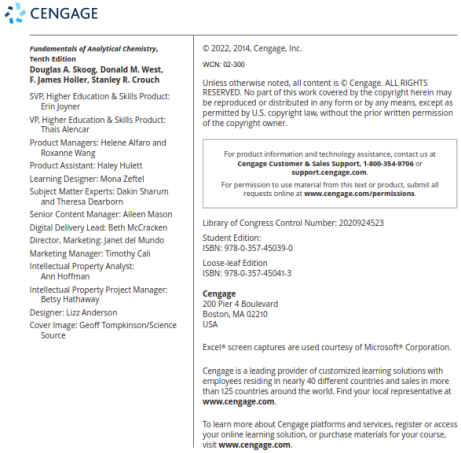
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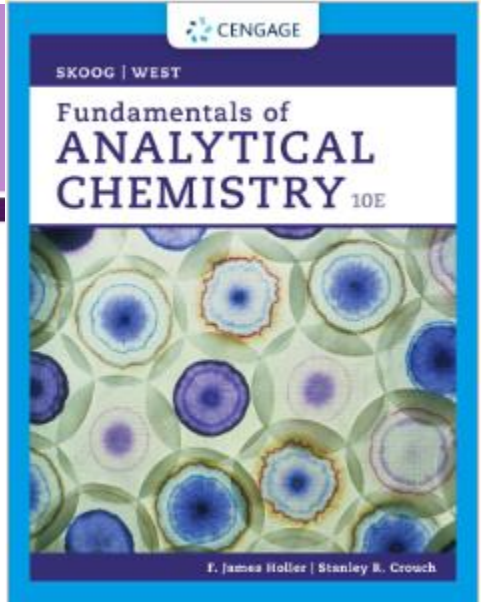
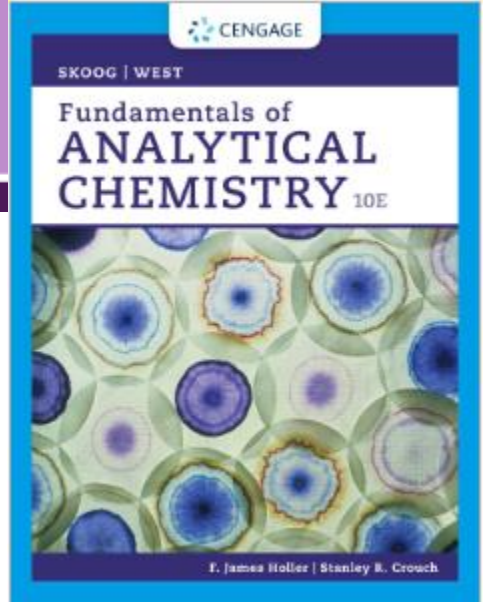
Contents in Brief



Contents in Brief



Analytical Chemistry

- What is the nature of analytical chemistry?
- What is the target field of analytical chemistry?
- What is the general types of analytical chemistry?

✓ qualitative: establishes the chemical identity of species

✓ quantitative: determines the relative amount of species

- What is the method types of analytical chemistry?

classic:

✓ volumetric

✓ gravimetric

✓ electrical charge

modern:

spectroscopy

Fields which Can be Applied in Analytical Chemistry

FIGURE 1-1 The relationship between analytical chemistry, other branches of chemistry, and the other sciences. The central location of analytical chemistry in the diagram signifies its importance and the breadth of its interactions with many

Keyword for the Goals of Analysis

- Separation; isolation: sometimes includes extraction step
- Purification
- Identification
- Determination: qualitative & quantitative

Keywords in Analysis

- To analyze: to do analysis
- Analyte: sample to be analyzed
- Assay: action of analysis mostly quantitative analysis
- Titrimetry

- Sampling: small mass/volume which is homogenous:

✓ represents the bulk sample

✓ in the same composition of the bulk sample

- Samples: Laboratory or Gross

Types of Analyses by Sample Size (Scale)
&
Types of Constituents in a Sample by Analyte Level

Sample Size	Type of Analysis
> 0.1 g	Macro
0.01 to 0.1 g	Semimicro
0.0001 to 0.01 g	Micro
$< 10^{-4}$ g	Ultramicro

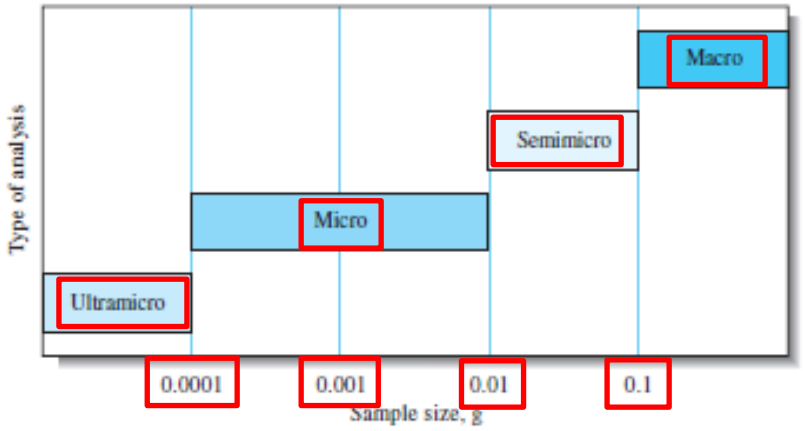


Figure 8-1 Classification of analyses by sample size.

Analyte Level	Type of Constituent
1 to 100%	Major
0.01 (100 ppm) to 1%	Minor
1 ppb to 100 ppm	Trace
< 1 ppb	Ultratrace

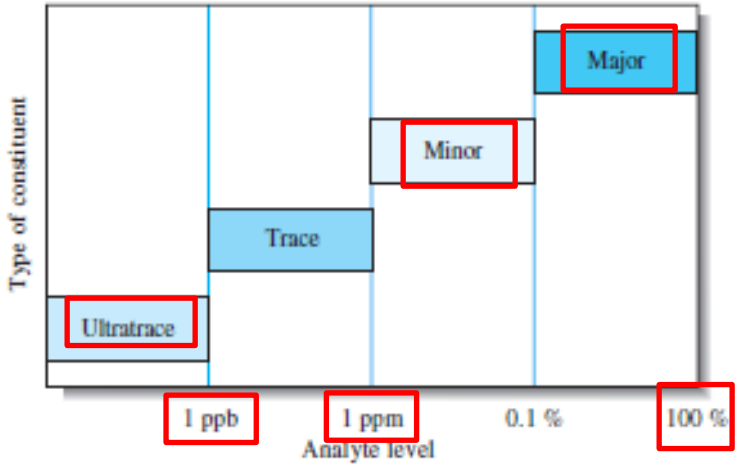
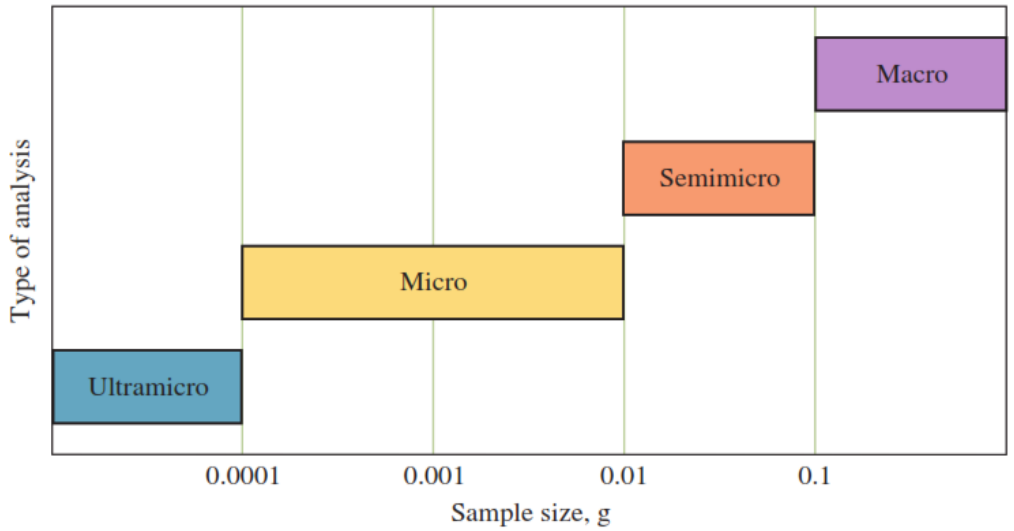


Figure 8-2 Classification of constituent types by analyte level.

Types of Analyses by Sample Size (Scale)
&
Types of Constituents in a Sample by Analyte Level

FIGURE 6-1

Classification of analyses by sample size.



Flow Diagram for Quantitative Analysis

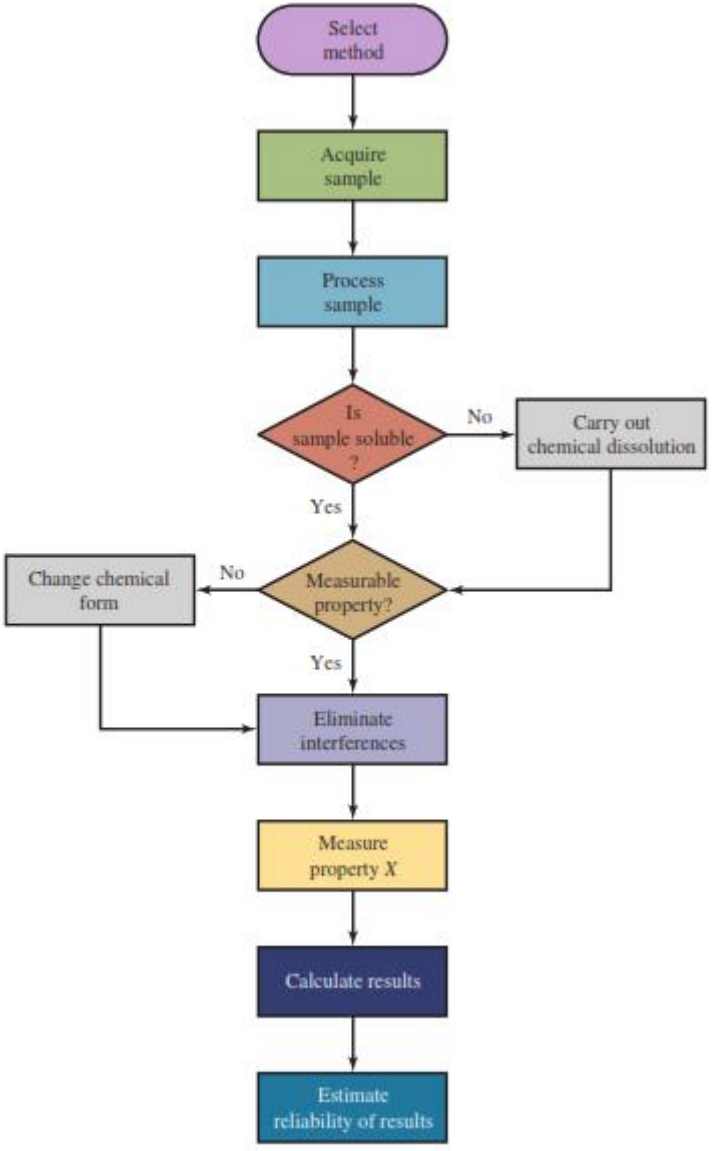


FIGURE 1-2
Flow diagram showing the steps in a quantitative analysis. There are a number of possible paths through these steps. In the simplest example represented by the central vertical pathway, we select a method, acquire and process the sample, dissolve the sample in a suitable solvent, measure a property of the analyte, calculate the results, and estimate the reliability of the results. Depending on the complexity of the sample and the chosen method, various other pathways may be necessary.

Flow Diagram for Analysis

- Method: accuracy, time & money
- ✓ specific
- ✓ selective
- Acquiring the sample:
- ✓ providing accurate representative sampling
- Processing the sample:
- ✓ preparing lab samples: manner of analyte/ substance
- ✓ replicate samples
- Eliminating interferences
- Analysis step: determine substance: qualitative &/or quantitative
- Calibrating (standardization) & measurement of physical/chemical property of the analyte
- Calculating the results: significant digits & rounding data
- Evaluating the estimation of reliability of results

Keywords to Introduce Reagents & Reactants Applied in Analysis

- Reagent grade

✓ specific-purpose reagent grade

- Standard grade:

✓ Primary Standard

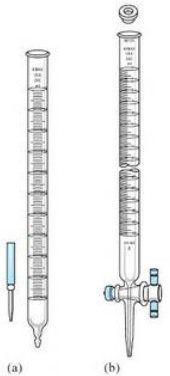
✓ Reference Standard (RS)

✓ Secondary Standard

Major Labwares for Analysis

- Volumetric flasks

- Buret



(a) (b)

Figure 2-19 Burets: (a) glass-bead valve, (b) Teflon valve.

Figure 2-20 Typical volumetric flasks.

- Volumetric pipette

(a)

Keywords for Chemical Techniques in Analysis

- Washing
- Transferring
- Decanting: decantation
- Filtering: through filter paper or sintered glass
- Ignition

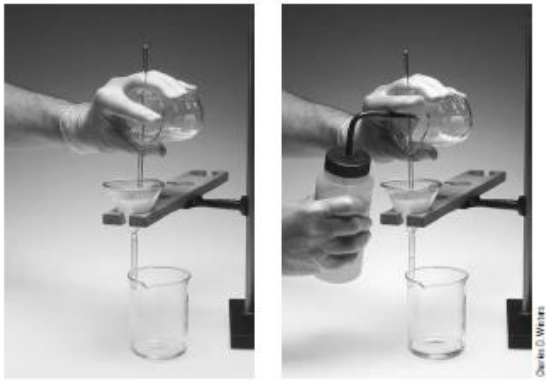


Figure 2-12 (a) Washing by decantation. (b) Transferring the precipitate.

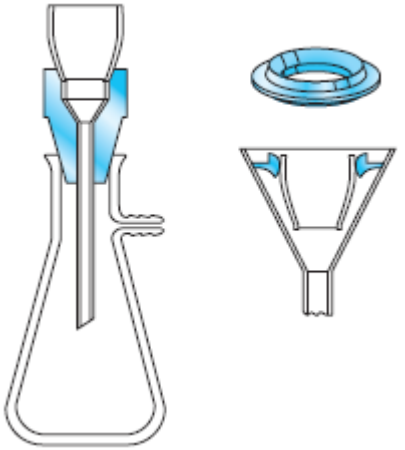


Figure 2-15 Ignition of a precipitate. Proper crucible position for preliminary charring is shown.

Figure 2-11 Adaptors for filtering crucibles.

Method of Folding a Filter Paper

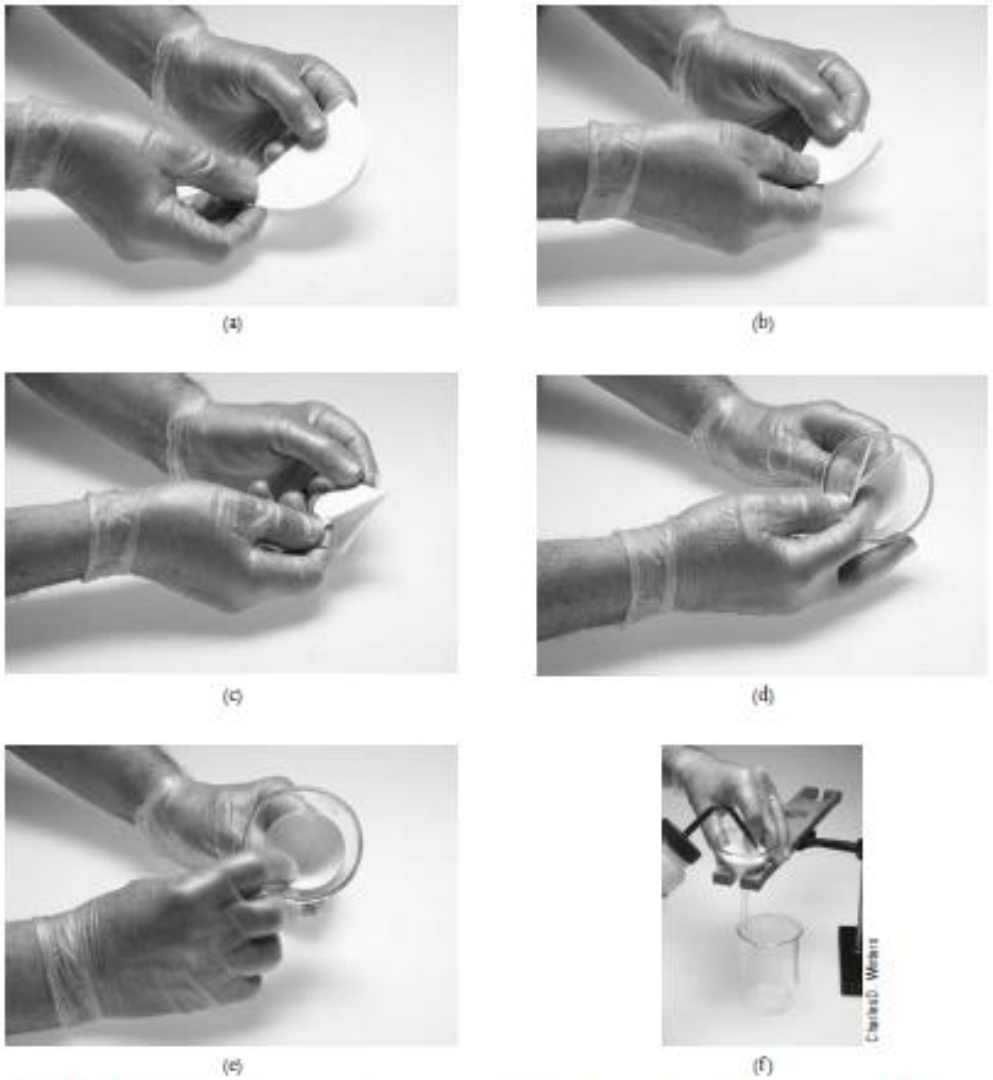
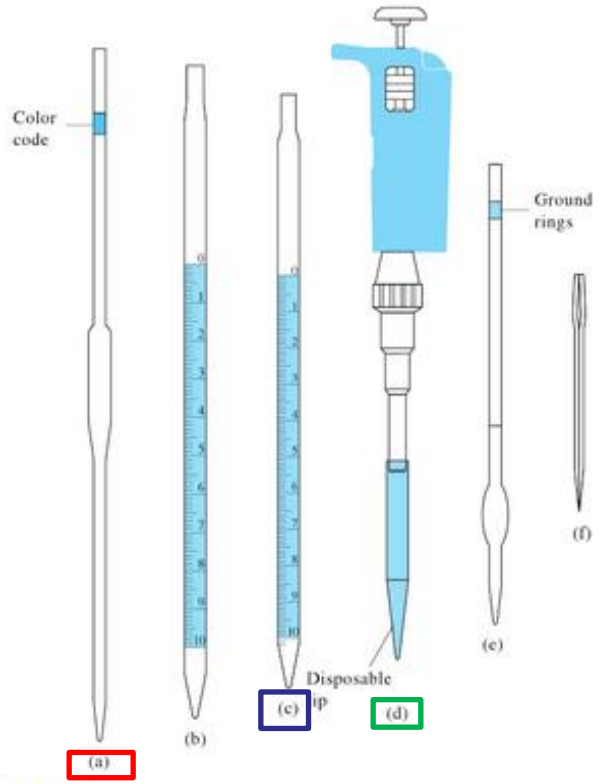


Figure 2-13 Folding and seating a filter paper. (a) Fold the paper exactly in half and crease it firmly. (b) Fold the paper a second time. (c) Tear off one of the corners on a line parallel to the second fold. (d) Open the untorn half of the folded paper to form a cone. (e) Seat the cone firmly into the funnel. (f) Moisten the paper slightly and gently pat the paper into place.

Typical Pipets, Samplers and Pro-pipets



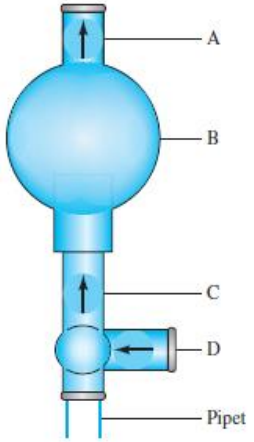


Figure 2-17 Typical pipets: (a) volumetric pipet, (b) Mohr pipet, (c) serological pipet, (d) Eppendorf micropipet, (e) Ostwald–Folin pipet, (f) lambda pipet.

Tolerances, Class A Transfer Pipets

Capacity, mL	Tolerances, mL
0.5	±0.006
1	±0.006
2	±0.006
5	±0.01
10	±0.02
20	±0.03
25	±0.03
50	±0.05
100	±0.08

Range and Precision of Typical Eppendorf Micropipets

Volume Range, µL	Standard Deviation, µL
1–20	<0.04 @ 2 µL <0.06 @ 20 µL
10–100	<0.10 @ 15 µL <0.15 @ 100 µL
20–200	<0.15 @ 25 µL <0.30 @ 200 µL
100–1000	<0.6 @ 250 µL <1.3 @ 1000 µL
500–5000	<3 @ 1.0 mL <8 @ 5.0 mL

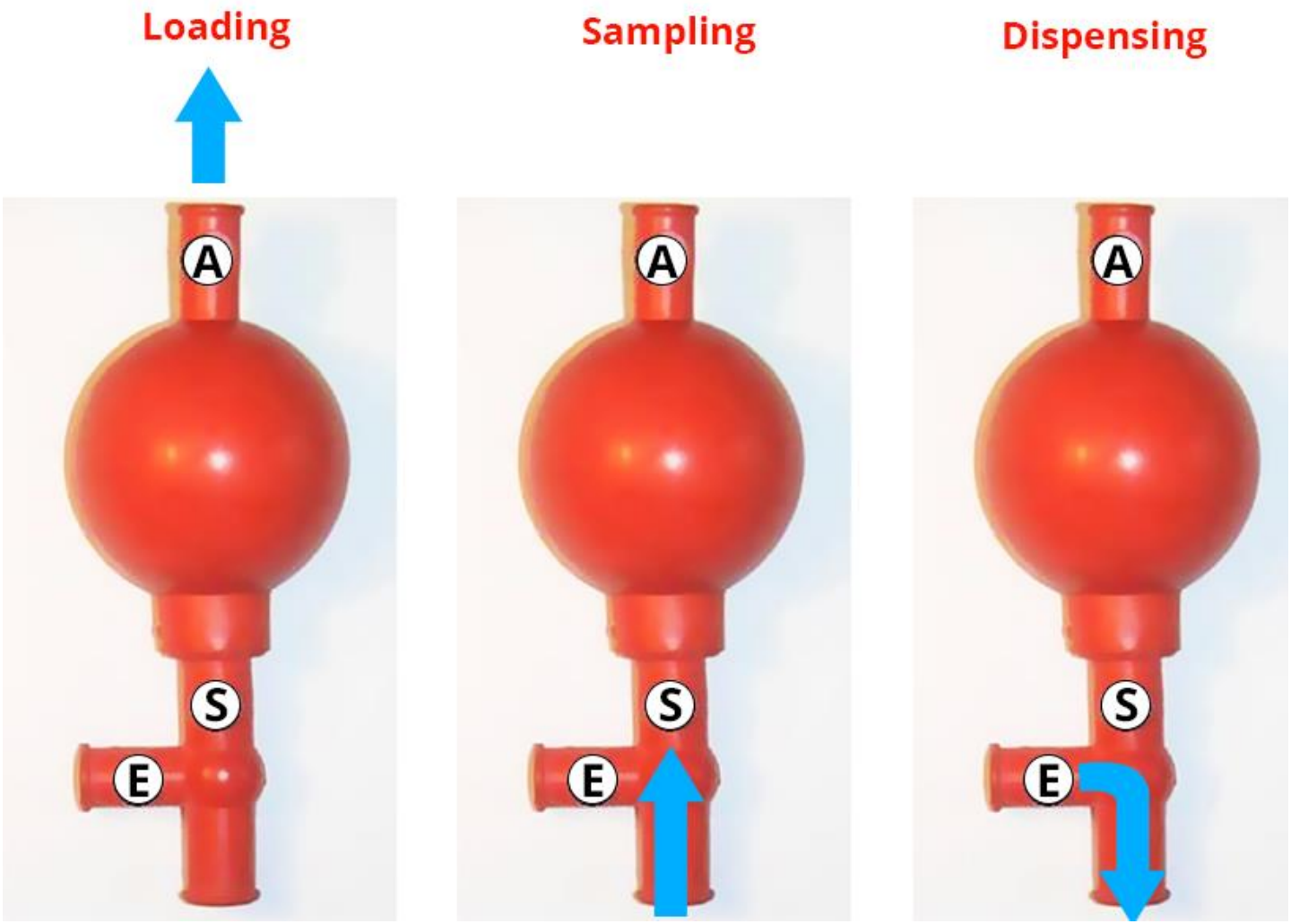
TABLE 2-2

Characteristics of Pipets

Name	Type of Calibration*	Function	Available Capacity, mL	Type of Drainage
Volumetric	TD	Delivery of fixed volume	1–200	Free
Mohr	TD	Delivery of variable volume	1–25	To lower calibration line
Serological	TD	Delivery of variable volume	0.1–10	Blow out last drop**
Serological	TD	Delivery of variable volume	0.1–10	To lower calibration line
Ostwald-Folin	TD	Delivery of fixed volume	0.5–10	Blow out last drop**
Lambda	TC	Containment of fixed volume	0.001–2	Wash out with suitable solvent
Lambda	TD	Delivery of fixed volume	0.001–2	Blow out last drop**
Eppendorf	TD	Delivery of variable or fixed volume	0.001–1	Tip emptied by air displacement

*TD, to deliver; TC, to contain.
**A frosted ring near the top of pipets indicates that the last drop is to be blown out.

Pro-pipets



Automatic pipet: Sampler

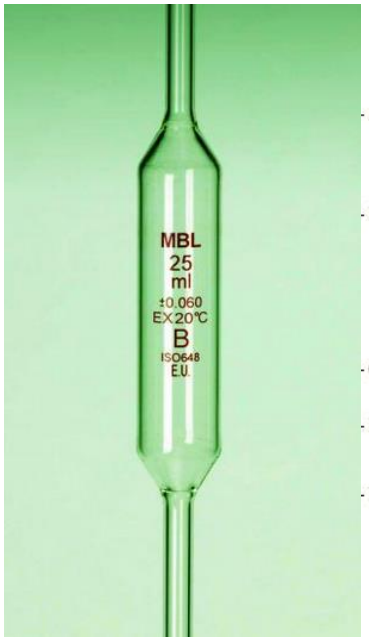
(a)

(b)

Figure 2-18 (a) Variable-volume automatic pipet, 100–1000 µL. At 100 µL, accuracy is 3.0%, and precision is 0.6%. At 1000 µL, accuracy is 0.6%, and precision is 0.2%. Volume is adjusted using the thumbwheel as shown. Volume shown is 525 µL.

Typical Pipette/ Sampler/ Pro-Pipette

(a)



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20	±0.03
25	±0.03
50	±0.05
100	±0.08

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	<0.06 @ 20 µL
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	<0.15 @ 100 µL
20–200	<0.15 @ 25 µL
	<0.30 @ 200 µL
100–1000	<0.6 @ 250 µL
	<1.3 @ 1000 µL
500–5000	<3 @ 1.0 mL
	<8 @ 5.0 mL

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Method of Manipulating a Buret Stopcock in Volumetric Analysis

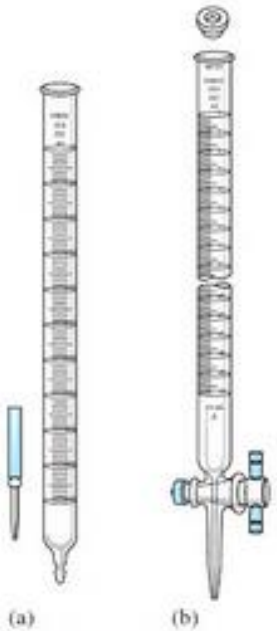

Figure 2-19 Burets: (a) glass-bead valve, (b) Teflon valve.

Figure 2-20 Typical volumetric flasks.

Figure 2-23 Recommended method for manipulating a buret stopcock.

Method of Reading a Buret

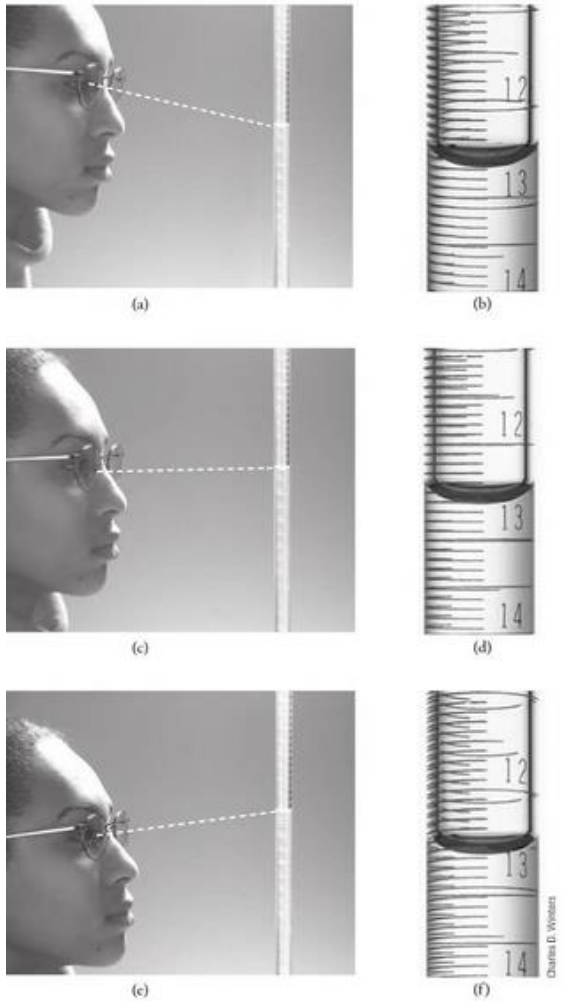

Figure 2-21 Reading a buret. (a) The student reads the buret from a position *above* a line perpendicular to the buret and makes a reading (b) of 12.58 mL. (c) The student reads the buret from a position *along* a line perpendicular to the buret and makes a reading (d) of 12.62 mL. (e) The student reads the buret from a position *below* a line perpendicular to the buret and makes a reading (f) of 12.67 mL. To avoid the problem of parallax, buret readings should be made consistently along a line perpendicular to the buret, as shown in (c) and (d).