Analytical Chemistry

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Introduction to Analytical Chemistry & Analysis

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Pharmacy Curriculum

- دروس علوم پایه و تخصصی

۲۹	شیمی تجزیه نظری	۲	–	۳۴	–	۲	شیمی عمومی نظری کد۲۴
۳۰	شیمی تجزیه عملی	–	۱	–	۳۴	۱	شیمی عمومی نظری و عملی کدهای ۲۴و۲۵- همزمان با شیمی تجزیه نظری کد۲۹

Major Textbook as Reference

PRINT

Fundamentals of Analytical Chemistry

9th Edition

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

Copyright 2014 | Published | Previous Editions 2004, 1996, 1992 | 1072 Pages

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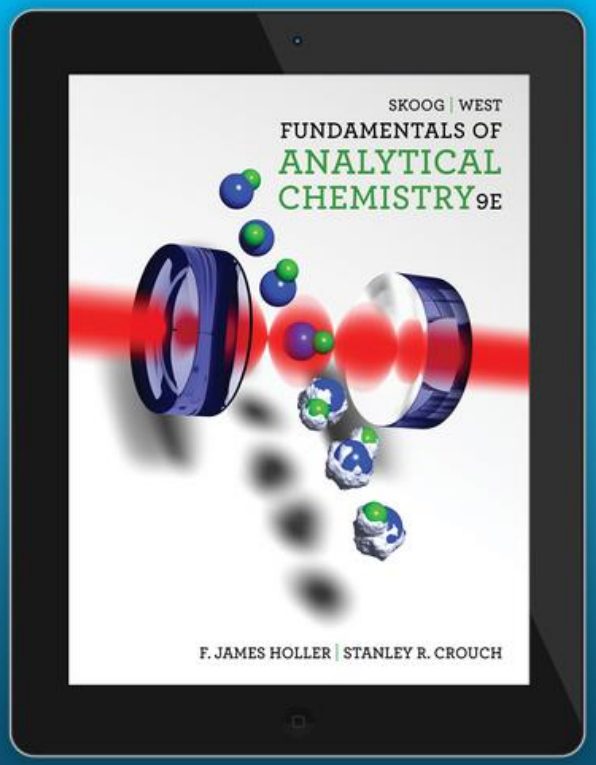
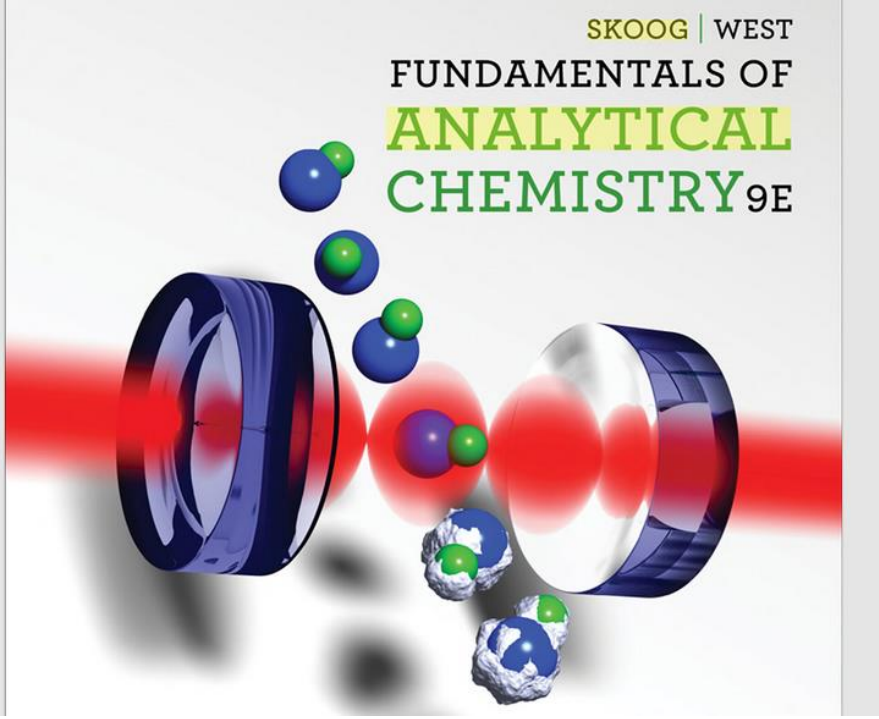


Contents in Brief



Analytical Chemistry

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

✓ qualitative

✓ quantitative

- What is the method types of analytical chemistry?

✓ volumetric

✓ gravimetric

✓ electrical charge

✓ spectroscopy

Fields Could be Applied in Analytical Chemistry

Keyword of Goals in Analysis

- Separation; isolation
- Purification
- Identification
- Determination: qualitative & quantitative

Keywords in Analysis

- To analyze
- Assay: action of analysis
- Analyte: sample to be analyzed.

- Sampling: small mass/volume which is homogenous:

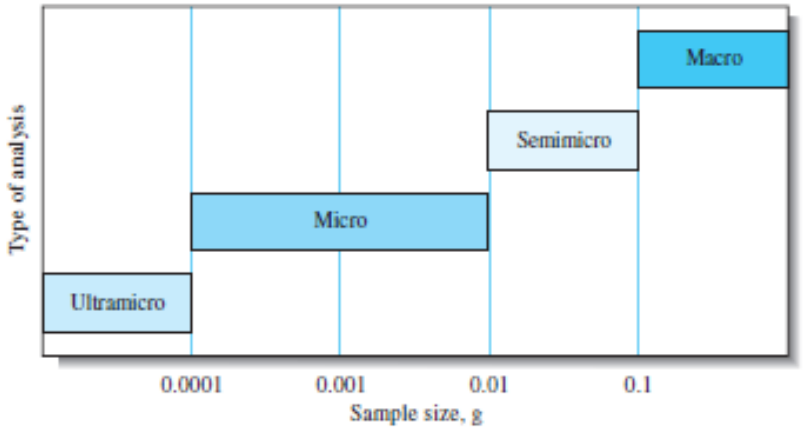
✓ represents the bulk sample

✓ in the same composition of the bulk sample

Classification of Analyses by Sample Size (Scale)
&
Classification of Constituent Types by Analyte Level

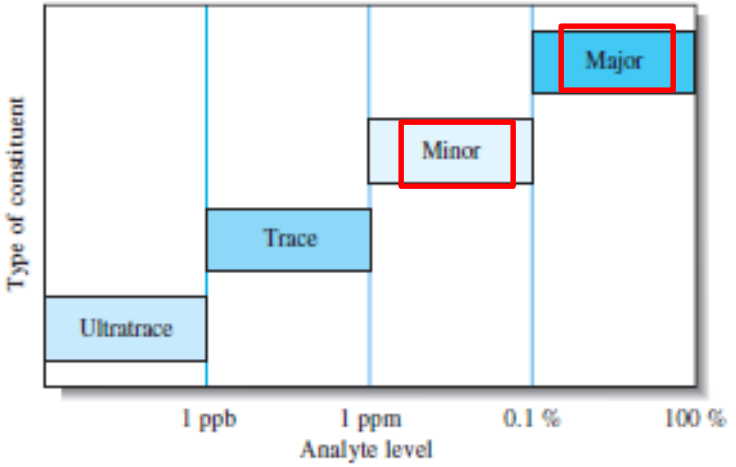
Sample Size	Type of Analysis
> 0.1 g	Macro
0.01 to 0.1 g	Semimicro
0.0001 to 0.01 g	Micro
$< 10^{-4}$ g	Ultramicro

Figure 8-1 Classification of analyses by sample size.

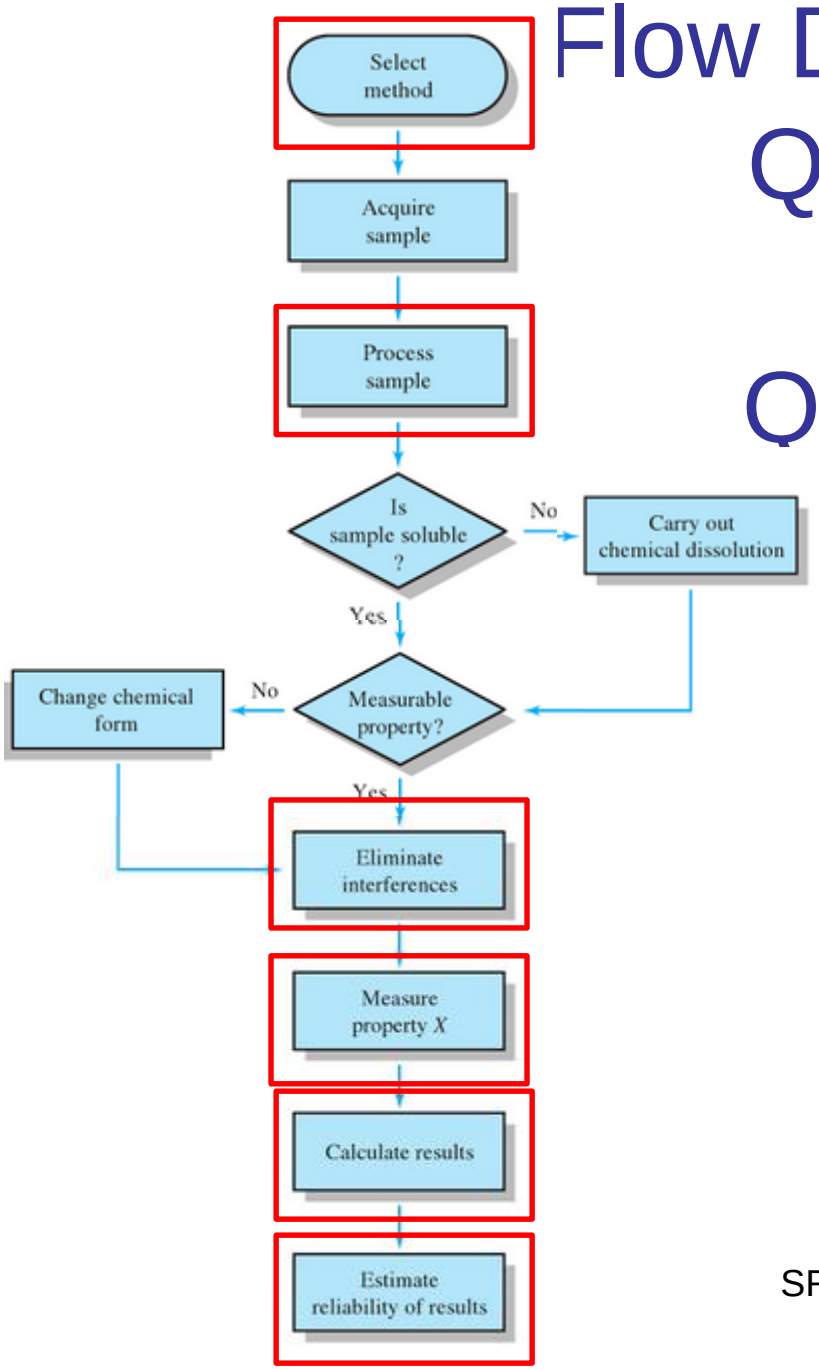


Analyte Level	Type of Constituent
1 to 100%	Major
0.01 (100 ppm) to 1%	Minor
1 ppb to 100 ppm	Trace
< 1 ppb	Ultratrace

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



Flow Diagram for Quantitative Analysis or Qualitative Analysis



Flow Diagram for Analysis

- Method: accuracy, time & money
- ✓ specific
- ✓ selective
- Acquiring the sample:
- ✓ provide accurate representative sampling
- Processing the sample:
- ✓ preparing lab samples: manner of analyte/ substance
- ✓ replicate samples
- Eliminating interferences
- Analysis step: determine substance
- Calibrating & measurement
- Calculating the results: significant digits & rounding data
- Evaluating the estimation of reliability of results

Keywords to Introduce Reagents & Reactants for Analysis

- Reagent grade

✓specific-purpose reagent grade

- Standard grade:

✓primary standard grade

✓Reference Standard (RS)

✓secondary standard grade

Labwares for Analysis

- Volumetric flasks

- Buret

(b)

Burets:

- Volumetric pipette

(a)

Chemical Techniques in Analysis

- Washing
- Transferring

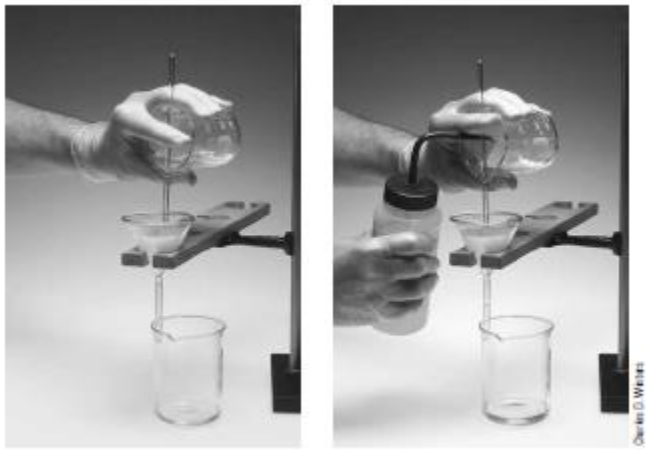


Figure 2-12 (a) Washing by decantation. (b) Transferring the precipitate.

- Decanting: decantation
- Filtering: filter paper; sintered glass

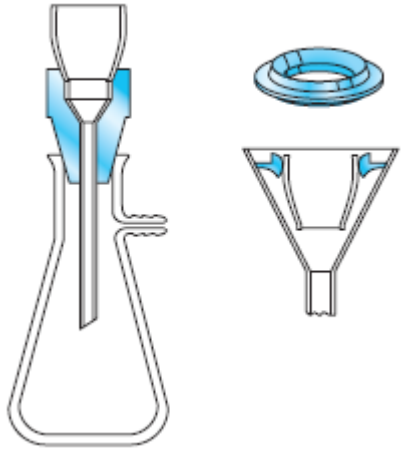


Figure 2-11 Adaptors for filtering crucibles.

- Ignition

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

Folding a Filter Paper

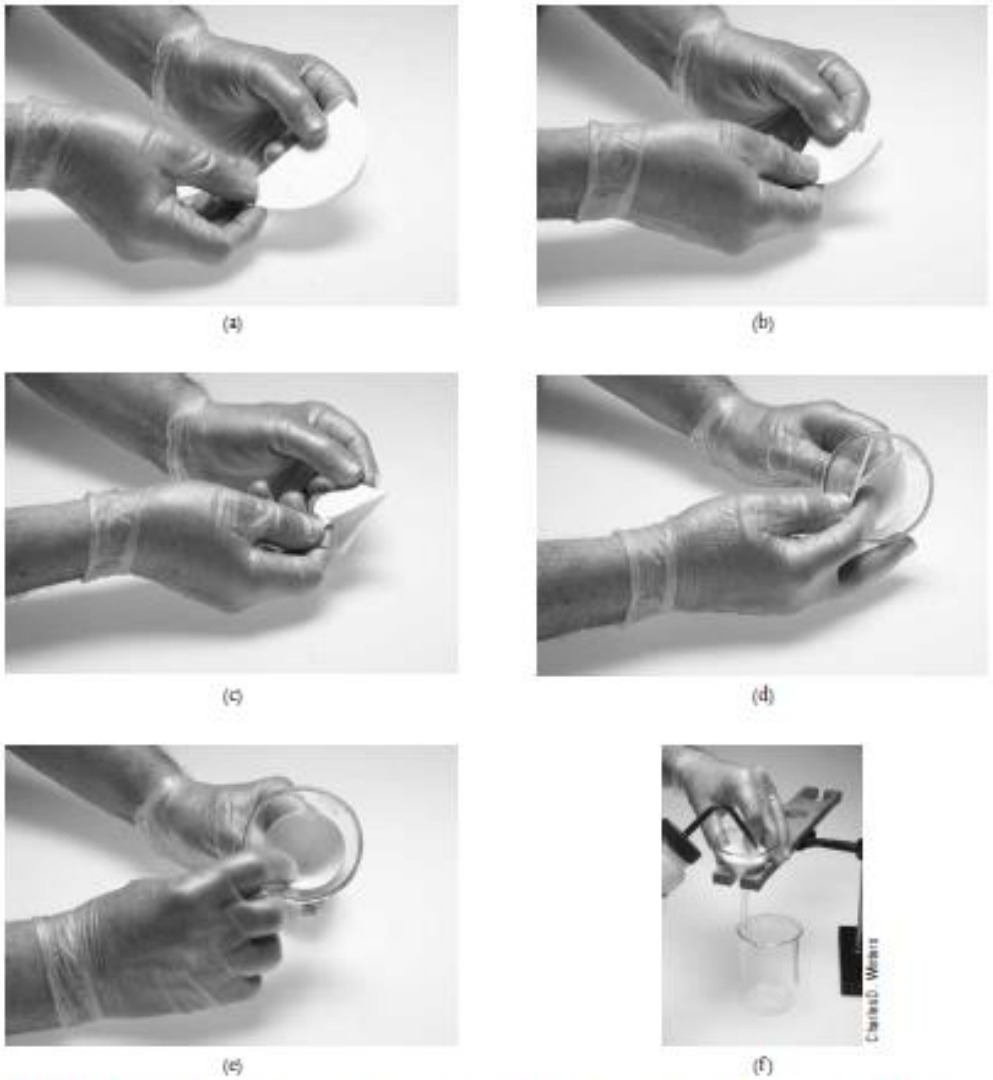
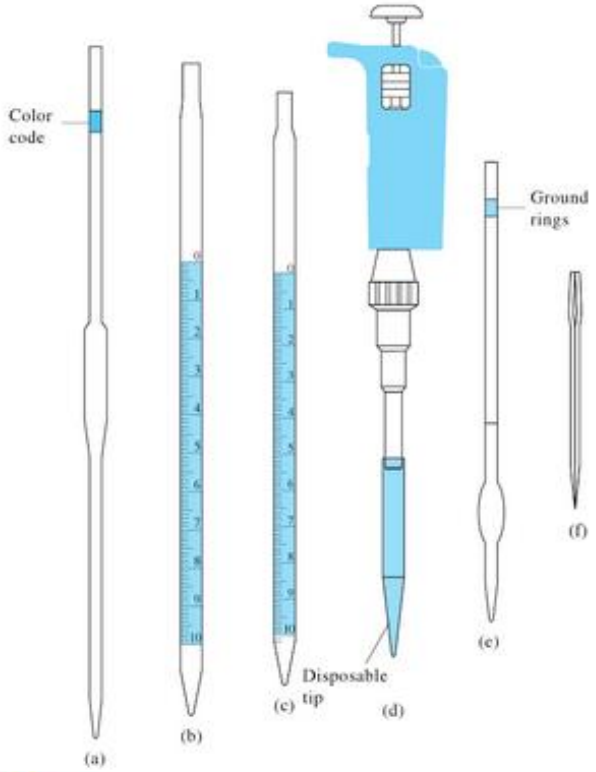


Figure 2-13 Folding and seating a filter paper. (a) Fold the paper exactly in half and crease it firmly. (b) Fold the paper a second time. (c) Tear off one of the corners on a line parallel to the second fold. (d) Open the untorn half of the folded paper to form a cone. (e) Seat the cone firmly into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

Typical Pipet (Sampler or Pro-pipet)



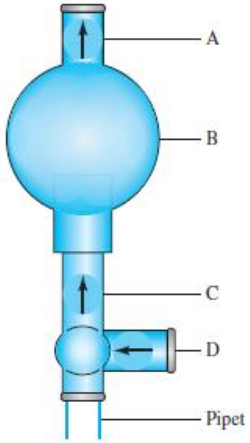


Figure 2-17 Typical pipets: (a) volumetric pipet, (b) Mohr pipet, (c) serological pipet, (d) Eppendorf micropipet, (e) Ostwald–Folin pipet, (f) lambda pipet.

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.

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

Automatic pipet: Sampler

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.

Manipulating a Buret Stopcock

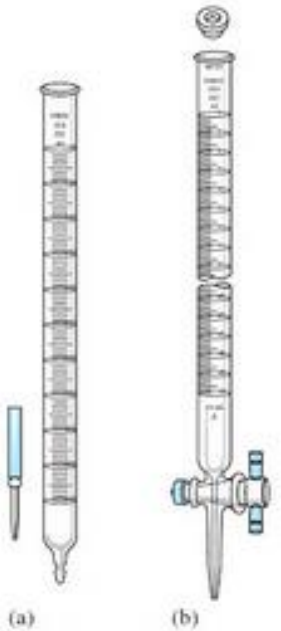


Figure 2-19 Burets: (a) glass-bead valve, (b) Teflon valve.

Figure 2-20 Typical volumetric flasks.

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

Reading a Buret Stopcock

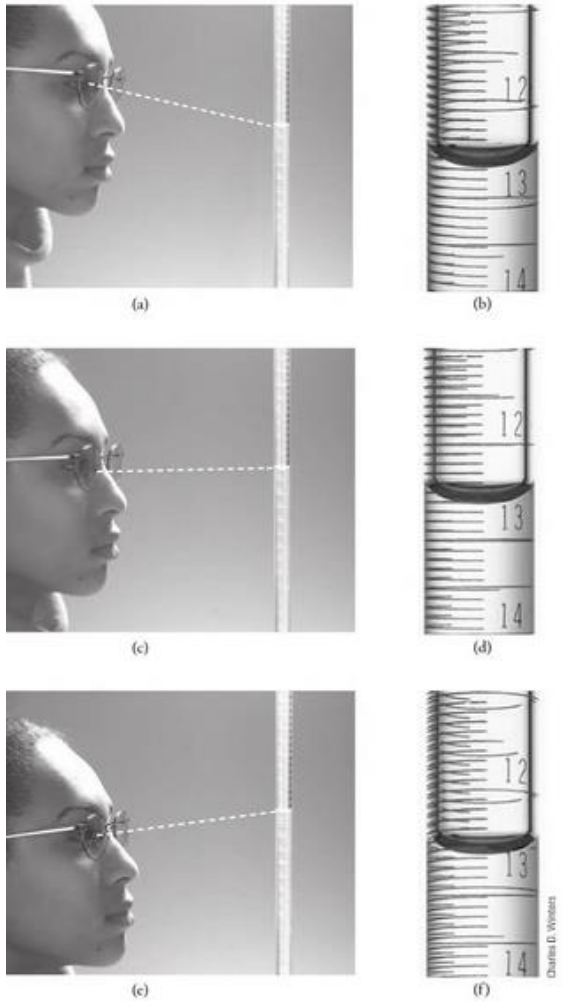


Figure 2-21 Reading a buret. (a) The student reads the buret from a position *above* a line perpendicular to the buret and makes a reading (b) of 12.58 mL. (c) The student reads the buret from a position *along* a line perpendicular to the buret and makes a reading (d) of 12.62 mL. (e) The student reads 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).