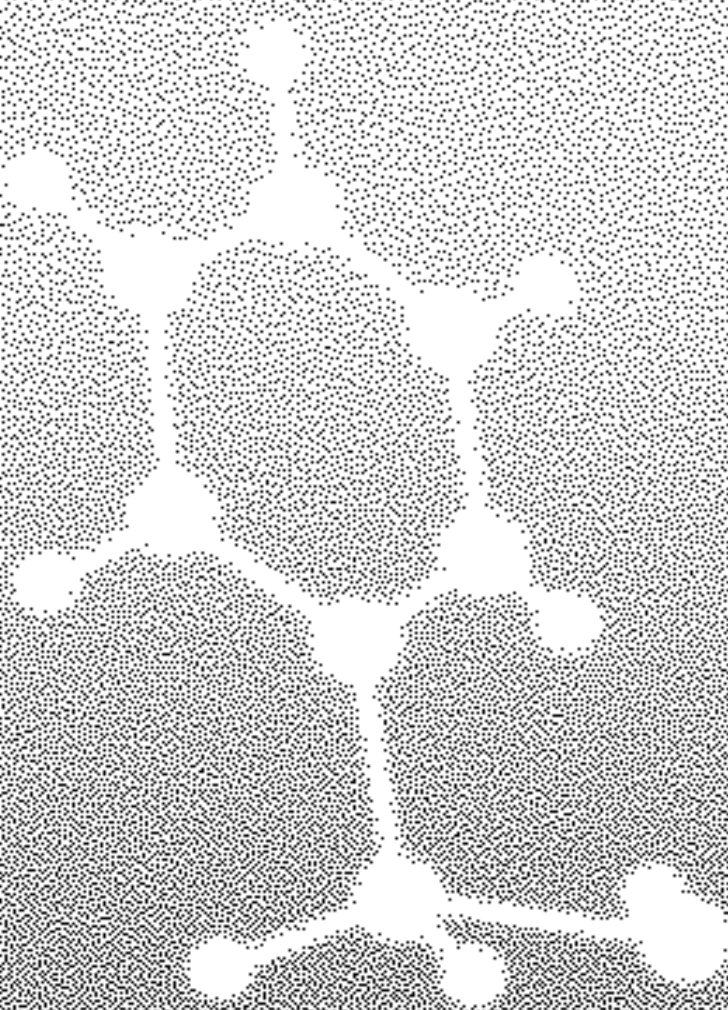
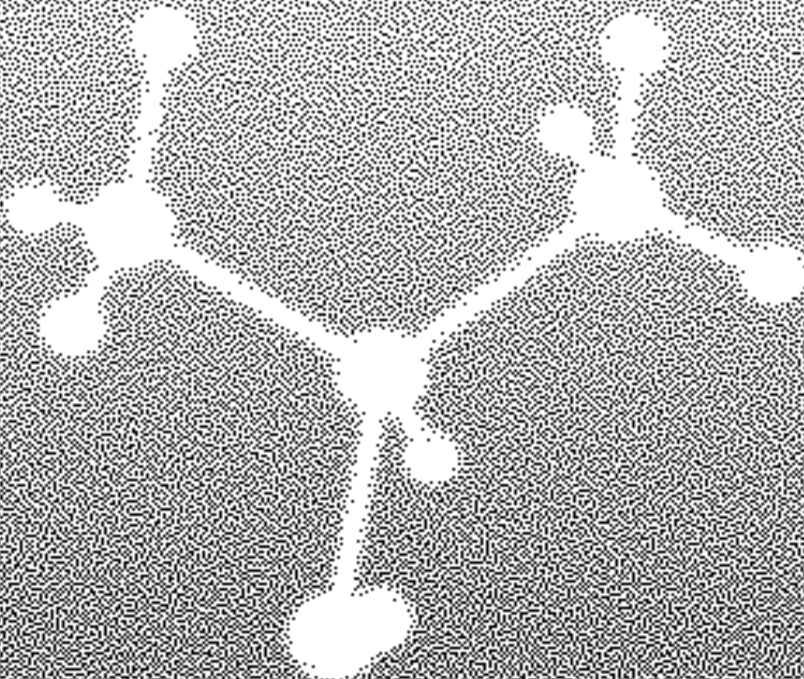


SECTION I

Introduction





Chapter 1

How to Use the Handbook

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Purpose and Scope

The goal of the *Handbook of Instrumental Techniques for Analytical Chemistry* is to provide scientists and engineers from many disciplines with an easily understood reference to current, established techniques of chemical analysis. The *Handbook* describes each technique in uncomplicated terms, gives common applications, points out its strengths and limitations, and provides references to more detailed discussions. The intent is to assist the reader in identifying the techniques that can best provide information to solve the problem at hand. For some problems, there may be a choice of techniques or the option of using complementary techniques. The *Handbook* is intended to provide the reader with sufficient knowledge to interact with analytical specialists or to pursue more detailed references.

Many of the techniques are used in growing areas such as biotechnology and materials science. However, techniques used predominantly for medical analysis are omitted as well as highly specialized techniques used in fundamental research. Discussions of hyphenated techniques, such as gas chromatography–mass spectrometry, are presented in the chapters on the individual techniques, with appropriate emphasis. For example, the gas chromatography chapter identifies mass spectrometers as detectors and refers the reader to the more detailed presentation in the mass spectrometry chapter. Conversely, the mass spectrometry chapter cites gas chromatography as a useful method for the preparation and introduction of samples and refers to the gas chromatography chapter.

Organization

The *Handbook* is designed for ease of use. This chapter contains general information and tables to assist the reader in selecting techniques for a given problem. The remaining chapters in the first section cover the fundamental principles of sample preparation for both inorganic and organic analyses, quantitative analysis, laboratory automation, and management of laboratory information.

The main part of the *Handbook* consists of seven sections, each addressing a group of related techniques. Techniques in Sections II through VI are organized according to fundamental phenomenon, chromatography, electromagnetic spectroscopies, mass spectrometry, and electrochemistry. The remaining two sections, on surface analysis and macromolecular analysis, include techniques specific to these major areas. Each section opens with an introduction by the section editor, followed by chapters on specific techniques. The identical format of each chapter facilitates comparison and selection of techniques. An introductory summary page assists the reader in deciding whether the technique is applicable to the problem at hand. The organization for each technique is shown in the following outline.

- Summary
 - General Use
 - Common Applications
 - Samples
 - State, Amount, Preparation
 - Analysis Time
 - Limitations
 - Complementary or Related Techniques
- Introduction
 - Brief History
 - Current Use
- How It Works
 - Physical and Chemical Principles
 - Instrumentation (Modular Approach)
 - Description of Each Major Component and Its Operation
- What It Does
- Analytical Information
 - Qualitative
 - Quantitative
 - Accuracy and Precision
 - Detection Limits
- Applications
 - General Discussion
 - Specific Examples
- Nuts and Bolts
 - Relative Costs
 - Capital Outlay
 - Maintenance and Operation
 - List of Instrument Manufacturers (see also Buyers' Guides, page 16)
- Nuts and Bolts (*continued*)

Required Training
Operation
Interpretation of Data
Service and Maintenance Requirements
Suggested Readings (Appropriate Level for Readers)

Analytical Methodology

Experienced analytical chemists use a systematic approach to obtain the required information from samples. This approach consists of a series of steps and accompanying thought processes. Professor Sidney Siggia, a general analytical chemist with extensive experience in industry and academics, captured this process in his book *Survey of Analytical Chemistry* (1). Much of his philosophy is reflected in the following sections.

Understanding and Defining the Problem

The goal of every chemical analysis is to obtain the required information within a period of time acceptable to the customer. This means that the analyst must know what information is needed to solve the current problem. For example, in qualitative analysis, complete identification of analytes is not necessary in many cases. Many problems require only a general classification or partial identification. Siggia cites the example of $C_{13}H_{22}(OCH_2CH_2)_4OSO_3H$, a surface-active agent for detergents, which can be analyzed at various levels depending on the information required. A cursory analysis would show the analyte to be an anionic detergent. Partial identification would indicate the analyte to be a sulfate ester of a fatty alcohol adduct of ethylene oxide. Finally, a complete analysis would identify the compound completely as written.

It is important for the analyst to determine the specific information required by a client. In the case of the detergent, the formulation people may need to know only the general classification, anionic detergent, because they mix components to produce a product with desirable characteristics. The detergent manufacturers may be most interested in the identification of functional groups and type of compound (sulfate ester of fatty alcohol adduct of ethylene oxide) for comparison with their products. When the compound is used in a commercial product, government regulations may require complete characterization of the compound.

Quantitative determinations are approached in the same way. Some problems require high precision and accuracy whereas much lower values are acceptable for others. Because there is considerable difference in the effort (and expense) required to provide different levels of qualitative and quantitative information, analysts must work closely with the clients to define the goals of analyses and determine proper limits for investigations.

History of the Sample and Background of the Problem

Before attempting any laboratory analysis, the analyst should become familiar with the background of the problem and the history of the sample. Background information can originate from many sources.

The client (such as a chemist, engineer, salesperson, or lawyer) who brought the sample for analysis should know the reason for the analysis and the origin of the sample. The more knowledgeable the client, the better the goals of the analysis can be defined. For example, when a client mixes reactants under certain conditions and does not get the expected products, the identities and amounts of the unexpected products can provide useful information. Knowledge of the reactant identities and the reaction conditions narrows the number of possible products for the analyst.

Analyses of competitors' products are commonly requested by sales and legal departments. In these cases, knowledge of materials used for specific applications and the products manufactured by the competitors provide the analyst with useful background information. Sample histories also contain information on how, where, and when the sample was collected, transported, and stored. Every sample has a history. A good analyst obtains and uses the sample history to maximum advantage in solving problems.

Literature Search

Efficient use of the scientific, technical, government, and commercial literature is essential to efficient analysis. References from chemical journals can indicate by-products of the reaction under consideration and thus ease the process of identification. Patents or commercial literature usually contain the composition of industrial materials. Registry numbers from *Chemical Abstracts* provide an easy way to locate information on specific compounds. Analytical methods are available from many sources: texts, review articles, monographs devoted to specific sample types, and standards organizations, such as the American Society for Testing and Materials (ASTM) and the Association of Official Analytical Chemists (AOAC). Many analyses must be performed using protocols approved by the Environmental Protection Agency (EPA), the Food and Drug Administration (FDA), or other government agencies. Even if the search does not locate a method for the problem at hand, it may provide ideas for developing a new analytical method.

Use of electronic media can make literature searches more efficient and effective. Sites available on the Internet include libraries, *Chemical Abstracts*, government documents, instrument vendors, electronic journals including *Analytical Chemistry*, and other special World Wide Web sites such as the Virginia Tech Encyclopedia of Chemical Instrumentation (<http://www.chem.vt.edu/chem-ed/analytical/ac-methods.html>). In today's electronic environment, the problem is one of filtering the massive amount of information to obtain what is needed for the problem at hand. The more one knows about the problem and the techniques available, the more effective is the search to provide useful information.

Plan of Action and Execution

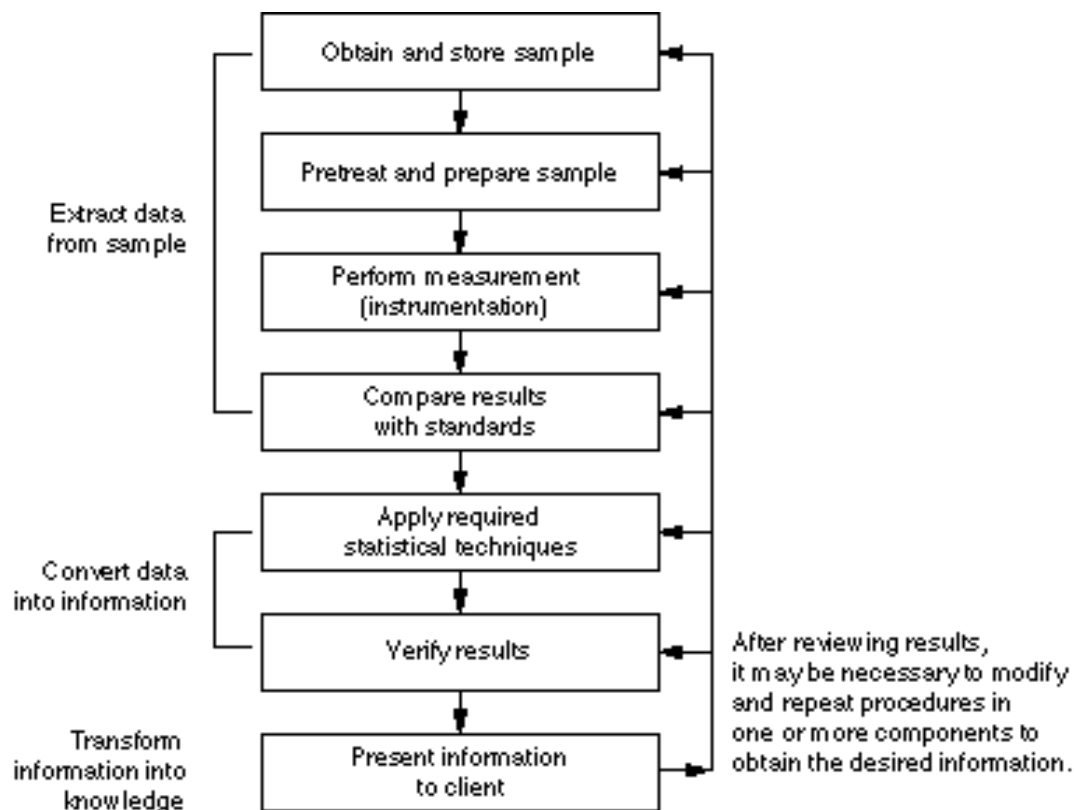
Once the goals and limits of the analysis are defined and the literature search is completed, a more detailed plan of action must be developed. The analyst selects the method or combination of methods most likely to provide the desired information. A good analyst is always alert to the chemistry involved in the analysis as well as instrumental techniques. Thus, a combination of chemical and instrumental techniques may be used.

Major components to be considered in planning an analysis are shown in Fig. 1.1. Each component is important in obtaining reliable information from the analysis. Field sampling and laboratory subsampling procedures must be designed to ensure integrity of results. Proper procedures must be used to store both samples and standards. All samples must be properly labeled and recorded. Laboratory operations are often performed on samples before measurement. These physical or chemical procedures may reduce or remove interferences, adjust analyte concentrations to a range suitable for measurement, or produce species from the analyte that have quantitatively measurable properties. These procedures include dissolution, separation, dilution, concentration, and chemical derivatization.

Control of the chemical environment is often necessary to ensure that the analyte is measured in the desired form and to minimize the effect of interferences. Environmental parameters include temperature, pH, and atmosphere surrounding the sample. Instrumental parameters such as the amplitude and frequency of input signal, detector sensitivity, and sampling rate must be coordinated to obtain optimum conditions for measurements.

Once the measurement has been obtained, steps must be in place to ensure the desired precision

Figure 1.1 Components of an analytical method.



and accuracy for the analysis. The methods best suited for standardizing the analysis must be selected. These methods include calibration curves, internal standards, standard additions, blind samples, and control charts. Statistical methods must be incorporated to assess the accuracy and precision of results.

When planning a method, the analyst must be aware that instrumentation is only one component. The analyst should be able to trace the flow of information through the entire process, not just through the instrumentation. Inadequate attention to any component of an analytical method can render the results meaningless, regardless of the power of the instrumental technique.

As the initial data provide the analyst with insight into the situation, it may become necessary to modify the original plan to obtain the desired information; in other cases no changes are required. The analyst should examine the data critically and be ready to modify the original approach.

Critical evaluation of the data requires a knowledge of the limitations of the techniques so as not to reach unjustified conclusions. However, the analyst must gather all information possible from the data and be aware that this information is often known with varying degrees of certainty.

The analyst determines when enough data have been obtained to reach tentative conclusions. The validity of these conclusions depends on the reliability of the data and the judgment of the analyst. The conclusions should be confirmed whenever possible. In qualitative analysis the identity of the unknown can be confirmed by comparing an actual sample of the material in question to the unknown using fingerprinting techniques such as infrared spectroscopy, mass spectrometry, or nuclear magnetic resonance spectroscopy. Matching spectra confirm the identification. Once a method has been developed to determine the composition of a certain sample or mixture, samples of known concentrations are made and analyzed to confirm the validity of the method. The composition of the known samples must match the matrix of the unknown samples and must cover the range of concentrations expected in

the unknown samples of the material under study.

Approaches to Unknowns

“The Analytical Approach,” a continuing series of articles found in *Analytical Chemistry*, provides insight into the processes described in the previous sections. According to Siggia, the thought processes of understanding, selection, evaluation, and correlation are necessary for the solution of any problem (1).

An example of the solution to an actual problem found in the construction industry illustrates the steps in the analytical approach (2). The problem was to determine why concrete in a large New York City construction project sometimes failed to set. The problem became so bad that the entire project was halted until the cause of the failure could be determined. Samples of the unset concrete from the project hardened before they reached the laboratory.

The plan of analysis was to select a series of standard methods to determine the presence of materials that retard setting of the concrete. Microscopic analysis revealed an unusual thin film on the cement particles. This suggested the presence of excessive amounts of retardant. Because sabotage was a possibility, the concrete was next analyzed for sugar, a known set-retarder. After samples were extracted with appropriate solvents, colorimetric methods indicated the absence of sugar. Other extracts were analyzed by infrared and ultraviolet techniques for the presence of known organic set-retarders such as polysaccharides and lignosulfates. These results also proved negative.

X-ray fluorescence spectrometry was then used to check for the presence of metals; trace amounts of both lead and zinc were found. A more accurate quantitative technique, atomic absorption spectrometry, revealed that both metals were present in the concrete at concentrations high enough to retard cement hydration. Further investigation determined the origin of the metals: dredged river gravel used as an aggregate in the concrete. This gravel was coated with lead and zinc materials found in the river.

Knowledge of the literature, experience with concrete analysis, and interactions with the clients (construction personnel) contributed to the solution. It was first necessary to understand the problem, then select appropriate methods for analysis, collect samples with historical information, perform the analysis, evaluate the results, and finally correlate the results with the situation to resolve the problem.

Thus, chemical analysis is more than just detecting or determining a specific component or the general composition of a sample. It is the resolution or interpretation of a given problem. What component in the fuel caused the engine to fail? Has a competitor infringed on the patent for the composition of our tinted glass? What substance in the feed line poisoned the catalyst? What is the effective concentration of a new drug? The analytical chemist must work closely with clients to provide the information required for the situation.

Technique Selection

The goal of any analysis is to measure a signal related to the concentration of the analyte. This signal usually contains a background or noise component whose magnitude limits the lowest analyte concentration that can be determined (the detection limit). The precision of the measured signal depends on several factors, some associated with fundamental phenomena and others with instrumentation. These may include noise from a variety of sources, instrument drift, and detector sensitivities to specific analytes. Interferences from components in the sample matrix or solvents can adversely affect many techniques. It is usually necessary to validate the accuracy of results of a technique by comparative analysis of standard reference materials representative of the sample matrix. Chapter 4 discusses quantitative analysis in detail.

Using the philosophy outlined in the previous section, the analyst, working closely with the client,

examines the sample and determines the information required to solve the problem. The analyst then chooses the techniques available to provide this information. Table 1.2 follows this same approach to assist the reader in locating techniques for the problem at hand. Table 1.1 contains a key to symbols used in Table 1.2. The page numbers in the Reference column of Table 1.2 direct the reader to the summary pages for the techniques. Once an appropriate technique is located the reader should further verify the suitability of the method by checking any endnotes associated with the technique and the information on the technique summary pages. Some techniques provide more definitive information than others; the amount of information varies with the technique. In many cases, Table 1.2 suggests several techniques for a given situation. Consultation with the information contained in the endnotes and technique summary page will often eliminate techniques from further consideration.

Many of the techniques listed for solid and liquid samples can also be used for gases if the gas is adsorbed onto a solid or absorbed into a liquid. In the case of liquid samples, some techniques listed for solids can be used for inorganic residues after the solution is evaporated to dryness or on organic residues after the solution has been ashed. Conversely, many of the techniques recommended for liquids can be used for solids if the solid can be dissolved in an appropriate solvent. Furthermore, if the solid can be vaporized without decomposition, techniques recommended for gas samples can be used.

Single-component samples and simple mixtures can often be analyzed directly by a given technique without prior separation. Complex mixtures often require separation before analysis. There are a variety of combined, or hyphenated, methods available in which the initial technique separates the analytes and the final technique provides the measurement component. Many gases and low-boiling-point organic liquids (boiling points below 300 °C) are relatively easy to determine.

It is often possible to extend the applications of techniques by chemical reactions. Analytes that are not sensitive to a specific technique may be converted to compounds or complexes that have the required sensitivity. Chelation of metal ions has extended the utility of ultraviolet/visible spectroscopy and quantitative conversion of high-boiling compounds to lower-boiling derivatives has increased the usefulness of gas chromatography.

Table 1.1 provides a key to the symbols used with the flow charts and assists in organizing the information desired from the samples. In some cases it is necessary to preserve the original sample. If elemental information is required, is it desirable to know the oxidation (valence) or complexed state of the element? For example, in the case of iron, does the technique determine iron as iron(II), iron(III), iron metal, iron(III) chelated with heme, or total iron in all forms? The ability to detect and measure an element in a specific chemical state, such as iron(III), is called speciation, abbreviated Sp. The ability to determine specific isotopes of elements as well as masses of both elements, compound fragments, and compounds present in samples is abbreviated as Is/Ms for the charts.

The ability to determine compounds is shown as Cp. Other useful information associated with compounds is obtained from functional group and structural analyses, including stereochemical information. Functional groups are specific groupings of atoms appearing in many compounds, such as the carboxyl grouping, COOH, that can give acidic properties to many organic compounds. Stereochemical analysis provides detailed information on the spatial arrangement of atoms in molecules.

Qualitative analysis determines the identity of the analyte whereas quantitative analysis determines the concentration of analyte present. The concentration ranges listed (major, minor trace, and ultratrace) are those in common use.

Table 1.1 Key to technique selection chart.

Location of analyte	
B	Sample from bulk of material in question
S	Sample from or on surface of material in question
Physical state of sample	
G	Gas
L	Liquid (includes liquids dissolved in liquid solvents)
S	Solid (includes solids with gases adsorbed on surface)
Ds	Solid dissolved in appropriate liquid solvents
Dg	Gas dissolved in appropriate liquid solvent
Amount of sample	
L	Macro > 1 milligram
S	Micro \leq 1 milligram
Estimated sample purity	
P	Pure (> 99%) element or compound
Sm	Simple mixture of up to six major components
M	Complex mixture
Fate of sample	
D	Destructive analysis
N	Nondestructive analysis
Elemental information	
El	Total analysis (element present in all chemical forms in sample)
Sp	Speciation (determination of individual elemental oxidation or chelation states)
Is	Isotopic and mass analysis
Molecular information	
Cp	Compounds present in sample
Io	Polyatomic ionic species present in sample
Fg	Functional group analysis
St	Structural analysis (structural and stereochemical determinations)
MW	Molecular weight determination
Pp	Physical property
Analysis type	
Ql	Qualitative analysis
Qt	Quantitative analysis
Analyte concentration	
Mj	Major component determination (> 10 wt %)
Mn	Minor component determination (10 to 0.1 wt %)
Tr	Trace component determination (1 to 1000 ppm or 0.0001 to 0.1 wt %)
Ul	Ultratrace component determination (< 1 ppm or < 0.0001 wt %)

Table 1.2 Techniques

Technique	Sample					Information Desired				Reference
	Location	Physical State	Amount	Purity	Form	Elemental	Molecular	Analytic Type	Analyte Concentration	
Separation techniques										
Gas chromatography	B	G, L, S, Ds	L, S	Sm, M	N, D ¹	El ²	Gp, Fn ¹	Q ¹ , Q ^t	Wj, Wn, Tr, U ²	8
High performance liquid chromatography	B	L, Ds	L, S	Sm, M	N, D ¹		Gp, lo, St	Q ¹ , Q ^t	Wj, Wn, Tr, U ²	9
Ion chromatography	B	L, Ds	L, S	Sm, M	N, D ¹	Sp	Gp, lo, Fn	Q ¹ , Q ^t	Wj, Wn, Tr, U ²	12
Supercritical fluid chromatography	B	L, Ds	L, S	Sm, M	N, D ¹		Gp	Q ¹ , Q ^t	Wj, Wn, Tr, U ²	11
Capillary electrophoresis	B	L, Ds	L, S	Sm, M	N	Sp	Gp, lo, St	Q ¹ , Q ^t	Wj, Wn, Tr, U ²	10
Planar chromatography	B	L, Ds	L, S	Sm, M	N		Gp, lo	Q ¹ , Q ^t	Wn, Tr ²	13
Optical spectroscopic techniques:										
Qualitative										
Infrared spectrometry (dispersive and Fourier transform)	B, S	G, L, S, Ds	L, S	P, Sm	N		Gp, Fn, St	Q ¹ , Q ^t	Wj, Wn, Tr	15
Raman spectrometry	B, S	G, L, S, Ds	L, S	P, Sm	N		Gp, Fn, St	Q ¹ , Q ^t	Wj, Wn, Tr	16
Nuclear magnetic resonance spectrometry	B	L, S, Ds	L, S	P, Sm	N		Gp, Fn, St	Q ¹ , Q ^t	Wj, Wn	17
X-ray spectrometry	B	S	L	P, Sm	N	El	Gp, St	Q ¹ , Q ^t	Wj, Wn, Tr	18

Table 1.2 (continued)

Technique	Sample					Information Desired			Reference (in parentheses)	
	Location	Physical State	Amount	Purity	Foto	Elemental	Molecular	Analysis Type		Analyte Concentrations
Optical spectroscopic techniques:										
Quantitative										
Atomic absorption spectrometry	B	L,Ds	L,S ²	Sm,M	D	El		Qt	Mj,Mh,Tr,U	20
Inductively coupled plasma atomic emission spectrometry	B	L,Ds	L	Sm,M	D	El		QLQt	Mj,Mh,Tr,U ⁶	21
Inductively coupled plasma mass spectrometry	B	G,L,Ds	L	Sm,M	D	El,Is		QLQt	Mj,Mh,Tr,U	22
Atomic fluorescence spectrometry	B	L,Ds	L,S	Sm,M	D	El ⁷		Qt	Mj,Mh,Tr,U	23
Ultraviolet/visible spectrometry	B	G,L,S,Ds	L,S	P,Sm	N	El ⁸ ,Sp	Cp,lo	QLQ ⁹	Mj,Mh,Tr	25
Molecular fluorescence spectrometry	B	G,L,S,Ds	L,S	Sm	N		Cp ¹⁰	Qt	Mj,Mh,Tr,U	26
Chemiluminescence spectrometry	B	G,L,Ds	L,S	Sm,M	D	El ⁷	Cp ¹¹	Qt	Mj,Mh,Tr,U	27
X-ray fluorescence spectrometry	B	L,S,Ds	L	Sm,M	N	El		QLQt	Mj,Mh,Tr,U	24
Mass spectrometry (MS)										
Electron ionization MS	B	G,L,Ds	L,S	P,Sm ¹²	D	El,Is	Cp,Fn,lo,St	QLQt	Mj,Mh,Tr	29,30
Chemical ionization MS	B	G,L,Ds	L,S	P,Sm ¹²	D	El,Is	Cp,Fn,St,Mh ¹²	QLQt	Mj,Mh,Tr	29,30
High resolution MS	B	G,L,S,Ds	L,S	p ¹²	D	El,Is	Fn,St,Mh ¹¹	Ql		30
Gas chromatography mass spectrometry	B	G,L,Ds	L,S	Sm,M	D		Cp,Fn,St	QLQt	Mj,Mh,Tr,U	31
Fast atom bombardment MS	B,S	L,S	L,S	P,Sm	D	El,Is	Cp,Fn,St,Mh	QLQt	Mj,Mh,Tr,U	32
High performance liquid chromatography MS	B	L,Ds	L,S	Sm,M	D	El,Is	Cp,Fn,St,Mh	QLQt	Mj,Mh,Tr,U	33
Laser MS	B,S	S,L,Ds	L ¹² ,S	P,Sm	D	El,Is	Cp,Fn,St,Mh	QLQt	Mj,Mh,Tr,U	34

Table 1.2 (continued)

Technique	Sample					Information Desired			Reference Chapter(s)	
	Location	Physical State	Amount	Purity	Photo	Elemental	Molecular	Analytic Type		Analyte Concentration
Electrochemical techniques										
Ampometric techniques	B	L, Ds, Dg	L	Sm ¹⁶	N	El, Sp	Cp, Io, Fn	Qt	Mj, Mn, Tr, U	36
Voltammetric techniques	B	L, Ds, Dg	L	Sm ¹⁶	N	El, Sp	Cp, Io, Fn	Qt, Ql	Mj, Mn, Tr, U	37
Potentiometric techniques	B	L, Ds, Dg	L	P, Sm	N	El, Sp	Cp, Io	Qt	Mj, Mn, Tr	38
Conductometric techniques	B	L, Ds	L	P, Sm ¹⁶	N	El, Sp	Cp, Io	Qt	Mj, Mn, Tr	39
Microscopic and surface techniques										
Atomic force microscopy	S	S	L	P, Sm	N	El	Cp	Ql ¹⁷		41
Scanning tunneling microscopy	S	S	L	P, Sm	N	El, Sp	Cp, Io	Ql ¹⁷		41
Auger electron spectroscopy	S	L ¹⁵ , S	L	P, Sm	N	El		Ql, Qt	Mj, Mn, Tr	42
X-ray photoelectron spectroscopy	S	L ¹⁵ , S	L	P, Sm	D	El ¹⁸ , Sp		Ql, Qt	Mj, Mn, Tr	43
Secondary ion MS	S	L ¹⁵ , S	L	P, Sm	D	El, Is	Cp, Io	Ql, Qt	Mj, Mn, Tr	32, 34, 44

Table 1.2 (continued)

Technique	Sample				Information Desired			Reference Chapter(s)		
	Location	Physical State	Amount	Purity	Form	Elemental	Molecular		Analytic Type	Analyte Concentration
Techniques for polymers¹⁹										
Size exclusion chromatography	B	L, D, S ²⁰	L	Sm	D		Mw	Qt	Mj, Mn	46
Low angle laser light scattering	B	L, D, S ²¹	L	P, Sm	N		Mw	Qt	Mj, Mn	47
Light scattering particle size techniques	B	L, D, S, Dg	L	Sm	D		Pp	Qt	Mj, Mn	47, 48
Pyrolysis techniques	B, S	D, S, L	L	Sm, M	D		Cp, Fv, St	Ql, Q ²²	Mj, Mn, Tr	49
Thermal techniques	B	S, L, Ds	L	P, Sm	D		Cp, Pp	Ql, Qt	Mj, Mn	50
Mechanical property techniques	B	S, L, Ds	L	P, Sm	D, N		Cp, Pp	Ql, Qt	Mj, Mn	51

1. Depends on the characteristics of the detector.
2. Can determine elemental composition with microanalytical plasma-optical emission detector.
3. Depends on type of detector and standardization procedure used.
4. Complements infrared spectroscopy; can analyze aqueous samples.
5. For selected elements with electrothermal atomizer.
6. For selected elements.
7. Applicable to nontransition metals and some transition metals.
8. Applicable to many transition elements.
9. Dilution with appropriate spectroscopic solvent often required for liquid solutions.
10. Many compounds that do not fluoresce can be derivatized to compounds that do.
11. Limited to compounds that can be made to undergo a reaction that generates chemiluminescence.
12. MS technique can serve as detector for separation technique with appropriate interface between output of separation technique and input of MS.
13. Intensity of parent peak (P) and P-peaks make molecular weight determination easier than with electron ionization. Chemical ionization spectrum contains fewer peaks than electron ionization spectrum.
14. Can determine molecular mass and formula with high accuracy and precision.
15. High boiling liquids can be handled.
16. Can serve as detectors for ion and liquid chromatographic separation techniques.
17. Provide high resolution images of surfaces allowing qualitative and semiquantitative analysis.
18. Sensitive to all elements except hydrogen.
19. Many techniques from the above section may be used to obtain information from polymer and other macromolecular samples. The techniques in this section are used exclusively for polymer and macromolecular samples.
20. Dilution with appropriate chromatographic grade solvent required.
21. Dilution with appropriate solvent required.
22. Chromatography and mass spectrometry are used to separate and analyze pyrolysis products.

Summary

The major components of analytical science (analytical methods applied across many disciplines) are shown in Fig. 1.2. Samples originate from many sources. Some preparation of samples prior to measurement is usually required. Tasks associated with this step range from simple dilution to complex separations. Samples may be brought to the measuring device or the measurement may be made at the sample site. The measurement step involves the transformation of chemical and physical properties of the sample into data in the form of electronic signals. The conversion of this data into information is the third major component of a method. This final step includes mathematical operations, management of the original data and resulting information, and report generation. These components are generic to any process involving measurements of physical or chemical properties.

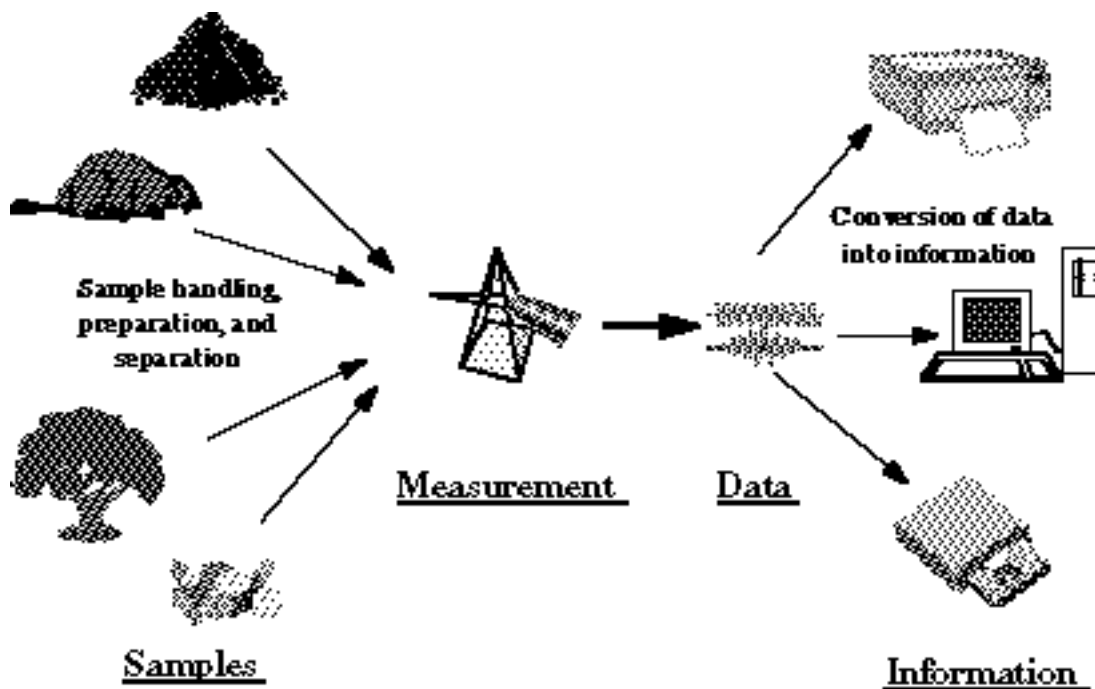
The chapters of this section address each of these components:

- Sample preparation (Chapters 2 and 3)
- Quality of measurements (Chapter 4)
- Acquisition and management of information (Chapter 5)
- Automation of the analytical process (Chapter 6)

Buyers' Guides for Analytical Instrumentation

The following guides are published annually and contain extensive information on the manufacturers and distributors of instrumentation, software, chemical reagents, and laboratory equipment, as well as firms providing analyses and consulting services.

Figure 1.2 The components of analytical science.



1. "LabGuide" published annually in August by *Analytical Chemistry*.
2. "Buyers' Guide" published annually in February by *American Laboratory*.
3. "Annual Directory" published in January by *Laboratory Equipment*.
4. Product reviews in the "A" pages of *Analytical Chemistry* focusing on a particular technique include general information on the selected technique and information on manufacturers.

Suggested Readings

The editor has found the following references especially useful in understanding the fundamentals of chemical analysis.

- ENKE, C., "Chemical Data Domains," *Analytical Chemistry*, 43, no. 1 (1971), 69A. Good paradigm for following transformations of data and information through a chemical analysis.
- HUBER, L., *Validation of Computerized Analytical Systems* Buffalo Grove, IL: Interpharm Press, Inc., 1995. This book is a guide to computer-controlled analytical instruments through the entire validation process from design through implementation, testing, and installation qualification to ongoing calibration and performance qualification.
- LAITINEN, H. A., AND W. E. HARRIS, *Chemical Analysis*, 2nd ed. New York: McGraw-Hill, 1975. Emphasizes the noninstrumental aspects underlying instrumental techniques.
- MILLER, J. C., AND J. N. MILLER, *Statistics for Analytical Chemistry*, 2nd ed. New York: Ellis Horwood, 1988. An excellent, understandable treatment of important topics in experimental design and statistical analysis of chemical data.
- SIGGIA, S., *Survey of Analytical Chemistry*. New York: McGraw-Hill, 1968. Presents a common-sense approach to analytical methodology. Unfortunately, it is out of print.
- TAYLOR, J. K., *Quality Assurance of Chemical Measurements* Chelsea, MI: Lewis Publishers, 1987. Good introduction to analytical process and validation.
- August review issue of *Analytical Chemistry*. In even-numbered years, reviews current developments in analytical techniques, and in odd-numbered years, covers application areas, such as air pollution, coatings, and foods.
- Articles in the "A" page section of *Analytical Chemistry* provide brief articles on recent advances in instrumentation and applications.

References

1. S. Siggia, *Survey of Analytical Chemistry* (New York: McGraw-Hill, 1968).
2. W. G. Hine, *Analytical Chemistry*, 46, no. 9 (1974), 1230A.