Table 1.2 Principal Applications of Instrumental Methods of Analysis

Molecular Analysis

Nuclear magnetic resonance (NMR) spectroscopy

Qualitative analysis: NMR is one of the most powerful methods available for determining the structure of molecules. It identifies the number and type of protons and carbon atoms in organic molecules, for example, distinguishes among aromatic, aliphatic, alcohols, and aldehydes. Most importantly, it also reveals the positions of the nuclei in the molecule relative to each other. For example, NMR will distinguish between CH_3—CH_2—CH_2OH and CH_3—CHOH—CH_3. It does not provide the MW of the compound. NMR is also applied to compounds containing heteroatoms such as sulfur, nitrogen, fluorine, phosphorus, and silicon.

Quantitative analysis: NMR is useful at % concentration levels, but trace levels (ppm) are becoming attainable with reasonable accuracy.

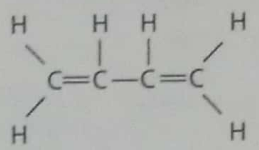
Infrared (IR) spectroscopy and Raman spectroscopy

Qualitative analysis: IR readily identifies organic functional groups present in molecules including groups containing heteroatoms—O, S, N, Si, halides. The IR spectrum is a fingerprint for a given compound, making it a very useful qualitative method. Classical mid-IR spectroscopy cannot be done on aqueous solutions. It does not give the MW of the compound. Raman spectroscopy complements IR spectroscopy and is useful for aqueous samples.

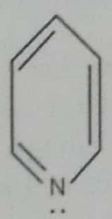
Quantitative analysis: IR is used routinely for the quantitative analysis of organic compounds, particularly at % concentration levels. It is used for liquid, solid, and gaseous samples. The related field of Raman spectroscopy complements IR.

Ultraviolet (UV) absorption spectroscopy

Qualitative analysis: UV absorption can be used for identifying functional groups and the structures of molecules containing unsaturated bonds (π electrons), such as



and aromatics and lone-pair electrons, such as those in pyridine:



lone pair

It does not indicate MW or give useful information on saturated bonds (σ bonds). NMR and IR have almost entirely replaced UV absorption spectroscopy for organic compound identification.

Quantitative analysis: UV absorption is used routinely for the quantitative determination of unsaturated compounds such as those found in natural products. The method is subject to spectral overlap and therefore interference from other compounds in the sample.

UV fluorescence

Qualitative analysis: UV fluorescence is used for the determination of unsaturated compounds, particularly aromatics. It does not indicate MW but gives some indication of the functional groups present. It is much more sensitive than UV absorption.

Quantitative analysis: UV fluorescence is a very sensitive method of analysis (10^{-8} g/g or 10 ppb), but it is subject to many kinds of interference, both from quenching effects and from spectral overlap from other compounds.

UV and visible (UV/VIS) spectrophotometry

Qualitative analysis: Organic or inorganic reagents are used for specific tests for many elements or compounds by forming a compound that absorbs at specific wavelengths. The products may or may not be colored. If the compounds are colored, analysis may be carried out visually (colorimetric analysis by eye), but the use of a spectrometer is more accurate.

Quantitative analysis: Sensitive and selective methods have been developed for most elements and many functional groups. It is used extensively in routine analysis of water, food, beverages, industrial products, etc.

Table 1.2 (continued) Principal Applications of Instrumental Methods of Analysis

X-ray diffraction (XRD)

Qualitative analysis: XRD is used for the measurement of crystal lattice dimensions and to identify the structure and composition of all types of crystalline inorganic and organic materials.

Quantitative analysis: XRD is used for the determination of percent crystallinity in polymers, the composition of mixtures, mixed crystals, soils, and natural products.

X-ray absorption spectroscopy

Qualitative analysis: X-ray absorption reveals the contours and location of high atomic weight elements in the presence of low atomic weight matrixes or holes in the interior of solid samples (voids). Examples are bone locations in the human body, the contents of closed suitcases, old paintings hidden under new painting on a canvas, and voids in welded joints and opaque solid objects.

Organic mass spectrometry (MS)

Qualitative analysis: MS can be used to identify the MW of organic and inorganic compounds, from very small molecules to large polymers and biological molecules (>100,000 Da). MS is a powerful tool in the determination of the structure of organic compounds. Fragmentation patterns can reveal the presence of substructure units within the molecule.

Quantitative analysis: MS is used extensively for the quantitative determination of the organic components of solid, liquid, and gas samples.

Thermal analysis (TA)

Qualitative analysis: TA is used to identify inorganic and some organic compounds using very small quantities of sample. It is also used to identify phase changes, chemical changes on heating, heats of fusion, melting points, boiling points, drying processes, decomposition processes, and the purity of compounds.

Quantitative analysis: TA can be used for the quantitative determination of the components of an inorganic sample, particularly at high concentration levels.

Gas chromatography (GC)

Qualitative analysis: GC can be used to separate the components of complex mixtures of gases or of volatile compounds. By comparison with known standards, it can identify components based on retention time.

Quantitative analysis: GC is an accurate method for quantitative analysis based on the area of the peak and comparison with standards. It is used extensively in organic, environmental, clinical, and industrial analysis. GC with MS detection (GC-MS) is a routine and powerful tool for quantitative analysis of organic compounds in environmental and biological samples.

Liquid chromatography (LC, high-performance LC [HPLC])

Qualitative analysis: LC is used for the identification of components of liquid mixtures, including polar compounds, ions, high MW components, and thermally unstable compounds. Identification is based on retention time and comparison with standards.

Quantitative analysis: LC is used for the quantitative determination of components in mixtures, especially for high MW or thermally unstable compounds. It is particularly useful for separating complicated mixtures such as natural products derived from plants or animals and biological samples such as urine and blood. Ion chromatography (IC) is used routinely in water analysis. LC with MS detection (LC-MS) is a routine and powerful tool for quantitative analysis of organic compounds in environmental and biological samples.

Capillary electrophoresis (CE)

Qualitative analysis: Used for the separation and identification of ions and neutral molecules in mixtures. Can be used for ions in aqueous solution and for organic ions.

Quantitative analysis: Quantitative determination of ions can be accomplished following separation, as in IC and LC.

Elemental analysis

Atomic emission spectrometry (AES), Optical emission spectrometry (OES)

Qualitative analysis: AES is an almost comprehensive method for qualitative elemental analysis for metals, metalloids, and nonmetals with the exception of some of the permanent gases. Its sensitivity range is great, varying from ppb to percent levels. Many elements can be detected simultaneously. Spectral overlap is the major limitation.

Quantitative analysis: AES is used extensively for the quantitative determination of elements in concentrations from percent levels down to ppb. Liquids, slurries, and solids can be analyzed using the appropriate instrumentation.

(continued)

Table 1.2 (continued) Principal Applications of Instrumental Methods of Analysis

Flame photometry (flame atomic emission spectrometry)

Qualitative analysis: Flame photometry is particularly useful for the determination of alkali metals and alkaline-earth metals. It provides the basis for flame tests used in qualitative analysis schemes.

Quantitative analysis: Flame photometry is used for the quantitative determination of alkali metals and alkaline-earth metals in blood, serum, and urine in clinical laboratories. It provides much simpler spectra than those found in other types of AES, but its sensitivity is much reduced.

Atomic absorption spectrometry (AAS)

Qualitative analysis: AAS is not used routinely for qualitative analysis, since with most instruments, it is only possible to test for one element at a time.

Quantitative analysis: AAS is a very accurate and sensitive method for the quantitative determination of metals and metalloids down to absolute amounts as low as picograms for some elements. It cannot be used directly for the determination of nonmetals.

X-ray fluorescence (XRF)

Qualitative analysis: XRF is useful for elements with atomic numbers greater than 4, including metals and nonmetals. For qualitative analysis, no sample preparation is required, and the method is generally nondestructive.

Quantitative analysis: XRF is used extensively for quantitative determination of elements in alloys and mineral samples, particularly of elements with high atomic weights. Sample preparation is complex for quantitative analysis.

Inorganic mass spectrometry (MS)

Qualitative analysis: Inorganic MS can identify elements, isotopes, and polyatomic ions in solutions and solid samples.

Quantitative analysis: Inorganic MS can determine elements at ppt concentrations or below. It is used for simultaneous multielement analysis for metals and nonmetals. Inorganic MS provides the isotope distribution of the elements. Special mass spectrometers are used for accurate isotope ratio measurements used in geology and geochemistry.