Analytical Chemistry Supplement

Context

Classroom and laboratory experiences in analytical chemistry at the undergraduate level should present an integrated view of methods and instrumental techniques, including their theoretical basis, for solving a variety of real chemical problems. Students should receive a coherent treatment of the various steps of the analytical process, including: problem definition, selection of analytical method, sampling and sample preparation, validation of analytical method, data collection and interpretation, and reporting. The problem-oriented role of chemical analysis should be emphasized throughout the student's experience. Such experiences provide an excellent introduction to the analytical process while engaging students in relevant societal problems requiring modern chemical analysis.

Conceptual Topics

The student should emerge from an undergraduate program of study having been exposed to a systematic treatment of the entire sequence of steps of the analytical process, including:

Definition of Analytical Requirements

- What is the analyte?
- What is the nature of the sample?
- What information is needed (qualitative, quantitative)?
- What level(s) of analyte(s) is (are) expected?
- For quantitative analysis, what is the detection threshold, and what is the required precision and accuracy?

Selection of Analytical Method

- Criteria: information content, specificity, limit of detection, interferences, dynamic range, sampling methods (gas, liquid, solid), sample preparation (solid phase extraction, digestion, etc.), accuracy, speed, ease of use, cost, temporal and spatial resolution, regulations (FDA, EPA, GLP, ISO)

- Capabilities and Limitations of Analytical Methods:

    - Chemical and Biological Reactions for Analysis and their Properties: Reaction stoichiometry, equilibrium chemistry, reaction rate, labeling (fluorescent, radiochemical), biospecific reactions (enzymes, antibodies, DNA)

    - Instrumental Methods: Instrument components and principles of their operation in the following areas:

        - Spectroscopy (UV-vis, fluorescence, atomic absorption, ICP-AES, IR, Raman, x-ray, NMR)
        - Separations (GC, HPLC, electrophoresis, ion chromatography, affinity chromatography)
        - Mass spectrometry including the distinction and utility of different ionization methods (e.g., EI, CI, ESI, MALDI) and mass analyzers (e.g., quadrupole, TOF, ion trap)
        - Electrochemistry (ion selective electrodes, amperometry, voltammetry)
        - Hyphenated techniques (GC-MS, LC-MS)
        - Thermal methods (TGA, DSC)

Signal Measurement and Processing Concepts:

- Basic electronics, signal/noise ratio, signal transducers, signal processing (filtering, Fourier transform)

Sampling and Sample Preparation

- Sampling approaches (random, stratified, etc.) power analysis, sample stability and storage
- Analyte pre-concentration and separation from complex matrices, elimination or reduction of interferences, derivatization/solubilization

Troubleshooting

- Identification and correction of problems when executing a method

Validation of Method:

- Choice of suitable standards, instrument calibration (standard addition, internal and external standards), use of surrogates (tracers), standard reference materials
- Collection and interpretation of data
- Statistical analysis (hypothesis testing, outliers, confidence intervals, errors, analysis of variance), accessing and employing databases

Reporting:

- Record-keeping, report writing, and oral presentation

Practical Topics

The laboratory experience needs to reflect the entire “analytical process” and not focus only on the measurement step. Problems to which students are exposed should reflect the diversity of analytical problem-solving scenarios:

- Biological and chemical systems including materials analysis and characterization
- Major to trace components
- Various physical states of matter
- Chemical speciation
- Comparison and selection of analytical methods for:
    - Qualitative analysis
    - Quantitative analyses reflecting a range of accuracy, precision, dynamic range, limit of detection, and limit of quantitation

The lab experience should include diverse approaches that reflect the wide range of analytical tools available (equilibrium-based methods, kinetic-based methods, physical properties) using various families of instrumentation including spectroscopy (atomic and molecular), separations, mass spectrometry and electrochemistry.

Illustrative Modes of Coverage

Analytical chemistry is typically taught in a two-course sequence – one at the foundation level, the other at an in-depth level. If a two-course sequence is used, both courses should include laboratory work and coverage of chemical/biological and instrumental methods of analysis. A foundation course in analytical chemistry should include an introduction to basic concepts and instrumental methods, with the goal of providing a systematic treatment of the entire sequence of steps of the analytical process. The laboratory would focus on problem solving approaches reflective of contemporary analysis requirements. The in-depth course and associated lab will build upon basic concepts developed in the foundation course. Laboratories associated with an in-depth course should incorporate more complex problem-solving and decision-making than that occurring in the foundation course.

An approach in which analytical chemistry is distributed throughout the curriculum is acceptable as long as the analytical process is taught. Carefully designed courses in environmental or forensic chemistry and biochemistry may provide some components of the analytical curriculum. The choice of problems for analysis affords an opportunity for students to understand and address the application of chemistry to broad societal concerns. Examples of such problems include environmental assessment, screening for controlled substances and explosives, materials characterization, toxicology, food safety, and detection of pathogens.

While spectroscopic characterization of newly isolated or prepared substances, which is typically included in organic and inorganic laboratories, are important components of the undergraduate curriculum, these experiences cannot be substituted for teaching the analytical process as described.

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