Analytical Chemistry Supplement

Context

Classroom and laboratory experiences in analytical chemistry at the undergraduate level should present an integrated view of chemical, biological 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. Not only do such experiences provide an excellent introduction to the analytical process they also provide the opportunity to introduce undergraduate students to 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?
- If quantitative analysis is desired, 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 major 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 including EI, CI, ESI, MALDI)
        - 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 consistent with question being addressed, analyte concentration and separation from complex matrices, elimination or reduction of interferences, derivatization/solubilization
- Validation of Method:
  - Choice of suitable standards, instrument calibration (standard addition, internal and external standards), use of surrogates (tracers)
  - Collection and interpretation of data
  - Statistical analysis (errors, analysis of variance, hypothesis testing), 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. The problems to which students are exposed should reflect the diversity of analytical problem-solving:

- Biological, materials, environmental, and chemical systems
- Major to trace components
- Various physical states of matter
- Chemical speciation
- Qualitative and quantitative analyses reflecting a range of accuracy and precision

The lab experience should provide experience in a diverse set of 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, and electrochemistry.

Illustrative Modes of Coverage

Typically analytical chemistry is taught in a two-course sequence; an introduction to basic concepts followed by a course focused on instrumental analysis. The latter course generally has a physical chemistry pre-requisite. If such 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 both 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. An approach in which analytical chemistry is distributed throughout the curriculum is acceptable as long as the "analytical process" is taught. In such an approach, general chemistry can serve to introduce some experiences in analytical chemistry. 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, 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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