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Elemental Analysis of Organic Compounds with the Use of Automated CHNS Analyzers

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Abstract—Results of many years' worth of studies on the determination of carbon, hydrogen, and nitrogen on automated CHNS analyzers were summarized. Catalytic oxide compositions were selected that allow for the analysis of synthetic and natural organic compounds and materials of any elemental composition and structure: polycyclic, condensed aromatic, heterocyclic, carbonaceous (graphites and carbons), organometallic, organo-element, etc.

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New compounds and materials, which appear as a result of the development of organic chemistry, such as different polycyclic, nitrogen containing, heterocyclic (imidazoles, oxazoles, triazoles, pyrroles, stable nitroxyl radicals, nitrones, pyrimidines, quinoxalines, etc.), polyhalogen-containing aromatic including poly-fluoroaromatic, organometallic, carbonaceous, etc., are characterized by a complex elemental composition and structure, a variety of properties, thermal and chemical stability, incombustibility, volatility, hygroscopicity, instability in light, etc. A necessary condition of their reliable identification and the verification of their purity is still the determination of the elemental composition of the compound performed by methods of organic elemental analysis. Its problems in the determination of carbon, hydrogen, and nitrogen in complex hardly degradable compounds by both classical and automated methods are well known. For example, in the determination of nitrogen in heterocyclic compounds containing nitrogen in the cycle or in groups such as azide, nitrile, nitro, nitron, and amine groups in concentrations >30%, the incomplete conversion of different forms of nitrogen to elemental nitrogen was observed and, hence, underestimated results were obtained. Of significant difficulty is the analysis of polyfluorinated compounds, e.g., polyfluoroaromatic compounds, fluoropolymers, fluoroclastomers, lubricants, fluorine-containing graphites, polyfluorocarbons, etc. Many of these compounds contain peripheral >CF₂ and -CF₃ groups along with the C-F bonds constituting a rigid fluorinated framework, and different fluorinated graphites contain, e.g., intercalated halogen fluorides ClF₂, ClF₃, and BrF₃ or alkali fluorides. The elemental analysis of these fluorides is complicated by the aggressiveness of fluorine and HF formed during the course of the decomposition, which leads to the corrosion of analytical instruments. Many fluorine-containing compounds

require more stringent decomposition conditions, because they are thermally stable, and the removal of fluorine as an interfering element from the combustion zone.

The major elements of an organic substance, namely, carbon, hydrogen, and nitrogen, are commonly determined using commercially available CHN and CHNS analyzers, in which the organic substance undergoes oxidative decomposition and the subsequent reduction of nitrogen and sulfur oxides with the formation of the final products: carbon dioxide, water, elemental nitrogen, and sulfur dioxide. Currently, both old types of analyzers (Carlo Erba, models 1106 and 1108; Hewlett-Packard, model 185; Perkin Elmer, model 240; etc.) and new types (Euro EA 3000; Perkin Elmer 2400, series II; CE 440; Vario EL III; etc.) are used in the practice of organic elemental analysis. It is known that the oxidants and catalysis developed and proposed in classical methods of elemental CHN analysis also find application in automated analyzers.

Co₃O₄, the product of the thermal decomposition of silver permanganate [1], Cr₂O₃ [2, 3], NiO [4], WO₃ [5, 6], V₂O₅ [7], SnO₂ [8, 9], CeO₂ [10], MgO [11], or their mixtures are used as additives to samples including hardly combustible substances containing, e.g., B, P, Si, and alkali metals; oxide additives improving the combustion of pyrimidines and triazoles (Co₃O₄-V₂O₅) [12], and polycyclic and organometallic compounds (V₂O₅, MnO₂, K₂Cr₂O₇, etc.) were also proposed [13–15]. The reagents added to a portion of a sample were calculated for a single effect and are removed before the analysis of the next sample. These are oxygen donors, oxidation catalysts, and absorbers of the interfering compounds. The functions of these reagents can hardly be separated.

Commonly, in both classical and automated methods, the sample is not completely oxidized in the pyro-

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Elemental Analysis Frank Vanhaecke, José Luis Todolí, Gerhard Schlemmer, Michael W. Hinds, 2025-06-02 Elemental Analysis is an excellent guide introducing cutting edge methods for the qualitative and quantitative analysis of elements Each chapter gives an overview of a certain technique such as AAS AFS ICP OES MIP OES ICP MS and XRF Readers will benefit from a combination of theoretical basics operational principles of instruments and their applications New extended section on supply of liquid samples for AAS ICP OES and ICP MS

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Undergraduate Instrumental Analysis Thomas J. Bruno, James W. Robinson, George M. Frame II, Eileen M. Skelly Frame, 2023-07-31 Analytical instrumentation is crucial to research in molecular biology medicine geology food science materials science forensics and many other fields Undergraduate Instrumental Analysis 8th Edition provides the reader with an understanding of all major instrumental analyses and is unique in that it starts with the fundamental principles and then develops the level of sophistication that is needed to make each method a workable tool for the student Each chapter includes a discussion of the fundamental principles underlying each technique detailed descriptions of the instrumentation and a large number of applications Each chapter includes an updated bibliography and problems and most chapters have suggested experiments appropriate to the technique This edition has been completely updated revised and expanded The order of presentation has been changed from the 7th edition in that after the introduction to spectroscopy UV Vis is discussed This order is more in keeping with the preference of most instructors Naturally once the fundamentals are introduced instructors are free to change the order of presentation Mathematics beyond algebra is kept to a minimum but for the interested student in this edition we provide an expanded discussion of measurement uncertainty that uses elementary calculus although a formula approach can be used with no loss of context Unique among all instrumental analysis texts we explicitly discuss safety up front in Chapter 2 The presentation intentionally avoids a finger wagging thou shalt not approach in favor of a how to discussion of good laboratory and industrial practice It is focused on hazards and remedies that might be encountered in the use of instrumentation Among the new topics

introduced in this edition are Photoacoustic spectroscopy Cryogenic NMR probes and actively shielded magnets The nature of mixtures in the context of separations Troubleshooting and leaks in high vacuum systems such as mass spectrometers Instrumentation laboratory safety Standard reference materials and standard reference data In addition the authors have included many instrument manufacturer s websites which contain extensive resources We have also included many government websites and a discussion of resources available from National Measurement Laboratories in all industrialized countries Students are introduced to standard methods and protocols developed by regulatory agencies and consensus standards organizations in this context as well

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Undergraduate Instrumental Analysis, Sixth Edition James W. Robinson,Eileen M. Skelly Frame,George M. Frame II,2004-12-02 Completely rewritten revised and updated this Sixth Edition reflects the latest technologies and applications in spectroscopy mass spectrometry and chromatography It illustrates practices and methods specific to each major chemical analytical technique while showcasing innovations and trends currently impacting the field Many of the chapters have been individually reviewed by teaching professors and include descriptions of the fundamental principles underlying each technique demonstrations of the instrumentation and new problem sets and suggested experiments appropriate to the topic About the authors JAMES W ROBINSON is Professor Emeritus of Chemistry Louisiana State University Baton Rouge A Fellow of the Royal Chemical Society he is the author of over 200 professional papers and book chapters and several books including Atomic Absorption Spectroscopy and Atomic Spectroscopy He was Executive Editor of Spectroscopy Letters and the Journal of Environmental Science and Health both titles Marcel Dekker Inc and the Handbook of Spectroscopy and the Practical Handbook of Spectroscopy both titles CRC Press He received the B Sc 1949 Ph D 1952 and D Sc 1978 degrees from the University of Birmingham England EILEEN M SKELLY FRAME recently was Clinical Assistant Professor and Visiting Research Professor Rensselaer Polytechnic Institute Troy New York Dr Skelly Frame has extensive practical experience in the use of instrumental analysis to characterize a wide variety of substances from biological samples and cosmetics to high temperature superconductors polymers metals and alloys Her industrial career includes supervisory roles at GE Corporate Research and Development Stauffer Chemical Corporate R D and the Research Triangle Institute She is a member of the American Chemical Society the Society for Applied Spectroscopy and the American Society for Testing and Materials Dr Skelly Frame received the B S degree in chemistry from Drexel University Philadelphia Pennsylvania and the Ph D in analytical chemistry from Louisiana State University Baton Rouge GEORGE M FRAME II is Scientific Director Chemical Biomonitoring Section of the Wadsworth Laboratory New York State Department of Health

Albany He has a wide range of experience in the field and has worked at the GE Corporate R D Center Pfizer Central Research the U S Coast Guard R D Center the Maine Medical Center and the USAF Biomedical Sciences Corps He is an American Chemical Society member Dr Frame received the B A degree in chemistry from Harvard College Cambridge Massachusetts and the Ph D degree in analytical chemistry from Rutgers University New Brunswick New Jersey Use of the Inductively-coupled Plasma for the Elemental Analysis of Organic Compounds David Lee Windsor,1977 A Practical Guide to Geometric Regulation for Distributed Parameter Systems Eugenio Aulisa,David Gilliam,2015-06-18 A Practical Guide to Geometric Regulation for Distributed Parameter Systems provides an introduction to geometric control design methodologies for asymptotic tracking and disturbance rejection of infinite dimensional systems The book also introduces several new control algorithms inspired by geometric invariance and asymptotic attraction for a wide range of dynamical control systems The first part of the book is devoted to regulation of linear systems beginning with the mathematical setup general theory and solution strategy for regulation problems with bounded input and output operators The book then considers the more interesting case of unbounded control and sensing Mathematically this case is more complicated and general theorems in this area have become available only recently The authors also provide a collection of interesting linear regulation examples from physics and engineering The second part focuses on regulation for nonlinear systems It begins with a discussion of theoretical results characterizing solvability of nonlinear regulator problems with bounded input and output operators The book progresses to problems for which the geometric theory based on center manifolds does not directly apply The authors show how the idea of attractive invariance can be used to solve a series of increasingly complex regulation problems The book concludes with the solutions of challenging nonlinear regulation examples from physics and engineering

Instrumental Analytical Chemistry James W. Robinson,Eileen M. Skelly Frame,George M. Frame II,2021-06-29 Analytical chemistry today is almost entirely instrumental analytical chemistry and it is performed by many scientists and engineers who are not chemists Analytical instrumentation is crucial to research in molecular biology medicine geology food science materials science and many other fields With the growing sophistication of laboratory equipment there is a danger that analytical instruments can be regarded as black boxes by those using them The well known phrase garbage in garbage out holds true for analytical instrumentation as well as computers This book serves to provide users of analytical instrumentation with an understanding of their instruments This book is written to teach undergraduate students and those working in chemical fields outside analytical chemistry how contemporary analytical instrumentation works as well as its uses and limitations Mathematics is kept to a minimum No background in calculus physics or physical chemistry is required The major fields of modern instrumentation are covered including applications of each type of instrumental technique Each chapter includes A discussion of the fundamental principles underlying each technique Detailed descriptions of the instrumentation An extensive and up to date bibliography End of chapter problems Suggested experiments appropriate to the technique

where relevant This text uniquely combines instrumental analysis with organic spectral interpretation IR NMR and MS It provides detailed coverage of sampling sample handling sample storage and sample preparation In addition the authors have included many instrument manufacturers websites which contain extensive resources The Journal of Industrial and Engineering Chemistry ,1916 American Journal of Pharmacy and the Sciences Supporting Public Health ,1920

Journal of Industrial and Engineering Chemistry ,1916 Industrial Laboratory ,1976 *Industrial Arts Index* ,1916 **Instrumental Organic Elemental Analysis** Ronald Belcher,1977 THE PERKIN ELMER MODEL 240 ELEMENTAL ANALYZER CARBON HYDROGEN AND NITROGEN THE PERKIN ELMER MODEL 240 ELEMENTAL ANALYZER OXIGEN OR SULPHUR THE CARLO ERBA ANALYZER CARBON HYDROGEN NITROGEN AND OXIGEN INSTRUMENTAL METHODS FOR THE DETERMINATION OF ORGANIC CARBON IN WATER THE DETERMINATION OF NITROGEN BY THE MERZ MODIFICATION OF THE DUMAS METHOD MICROCOULOMETRIC METHODS FOR THE DETERMINATION OF SULPHUR CHLORINE NITROGEN AND INDIVIDUAL COMPOUNDS American Journal of Pharmacy ,1920 *A Study of Seleno-organic Compounds and Their Analysis* Wilber Elmore Bradt,1926 Hygienic Laboratory bulletin. no. 115-120, 1918-20 ,1919 **Journal of Analytical Chemistry of the USSR.** ,1992 **A Text-book of Quantitative Chemical Analysis ...** Frank Julian,1902 The Preparation and Analysis of Organic Compounds Joseph Bernard Coleman,Francis Arnall,1926

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