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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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