Rajmund MICHALSKI
Jan Długosz Academy, Częstochowa
r.michalski@ajd.czest.pl
Jolanta JANISZEWSKA
Institute for Sustainable Technology – National Research Institute, Radom
jolanta.janiszewska@itee.radom.pl

ION CHROMATOGRAPHY – A METHOD OF CHOICE FOR THE DETERMINATION OF IONIC SPECIES IN WATERS AND WASTE WATERS

Key words

Ion chromatography, water analysis, wastewater analysis, inorganic ions, standardization.

Abstract

Water and wastewater are an important part of chemical analysis. The development of new methods and improvement of existing ones is a major task for analytical chemists. There is a need for reliable identification and monitoring of many substances present in water and wastewater in order to prevent contamination and protect the environment. Substances occurring in different types of water may be classified as biological, chemical (both inorganic and organic), physical, and radiological impurities. Inorganic anions and cations are the most frequently determined in many laboratories in routine analysis. Ion chromatography is an attractive technique for the determination of ionic species, especially for laboratories that need to determine numerous anions and cations in several thousand samples, but do not have the throughput to justify the purchase of large automatic analysers, usually based on colorimetric

procedures. Ion chromatography eliminates the need to use hazardous reagents, which are often integral to wet chemical methods. The demands from regulators for justifiable analytical results and from laboratories for validated methods provide the necessity to standardize ion chromatographic methods.

Introduction

Water is considered as one of the basic substances supporting life and the natural environment, a primary component for industry, a consumer item for humans and animals, and a vector for domestic and industrial pollution. Various directives provide a framework for the control of aquatic substances, the quality of bathing, surface and drinking water, and acceptable concentration in wastewaters. The number of chemicals determined in water has grown exponentially in the past 60 years; however, very few have been studied or have documented proof of their health effects. Nearly half of the monitored parameters are being measured for operational reasons (e.g., iron, ammonium, pH, chloride, dissolved organic carbon) and for reasons of customer satisfaction (e.g., colour, taste, and total hardness) [1].

Of the health-related compounds, a number of the elements and small groups of organic compounds, in the most of countries, are being measured due to legislation. They include the metals, e.g., chromium, antimony, manganium, arsenic, aluminium, magnesium, cadmium, lead, copper, nickel, mercury, iron as well as inorganic anions (e.g., fluoride, nitrite, nitrate, cyanide, bromate) and some organic compounds (e.g., benzo(a)pyrene, trihalomethanes, chlorobenzenes, pesticides). The United States Environmental Protection Agency (US EPA) has established regulations and methodology for inorganic contaminants under the Safe Drinking Water Act. Fluoride, nitrate, and nitrite are classified as primary pollutants, since they can cause adverse health effect. High level of fluoride causes skeletal and dental fluorosis, while nitrite and nitrate can cause methemoglobulinemia, which can be fatal to infants. Other common inorganic anions, like chloride and sulphate, responsible for water taste, odour, colour, and certain aesthetic effects, are considered secondary contaminants.

The determination of common inorganic anions (F^-, Cl^-, NO_2^-, NO_3^-, SO_4^{2-}, and PO_4^{3-}) and cations (Na^+, K^+, Mg^{2+}, and Ca^{2+}) was usually carried out using chemical wet methods such as gravimetry, titration, turbidimetry, and colorimetry. Many of these methods suffer from interferences and limited sensitivity, and they can be labour intensive and are often difficult to automate. Although performance criteria (trueness, precision, and the limit of detection) can be specified for analytical methods, but there are still problems with obtaining of similar results in different laboratories. In turn, organic substances are usually determined by chromatographic methods, which can help to separate

and determine polar and non-polar components; acidic, neutral and alkaline compounds; organic and inorganic substances; monomers, oligomers and polymers. It is necessary to use an appropriate chromatography type, which depends on the physicochemical properties of the examined sample and its components. Gas chromatography (GC) and liquid chromatography (LC) can be used to separate and determine approx. 20% and 80% of the known compounds, respectively.

In 1975, Small *et al.* [2] described a novel ion-exchange chromatographic method for the separation and conductometric detection of ionic species. They employed a low-capacity ion-exchange stationary phase for the separation of analyte ions, in conjunction with the second ion-exchange column and conductivity detector, which allowed for continuous monitoring of ionic species of sample components. In September 1975, ion chromatography had its public presentation at the meeting of the American Chemical Society, where Dionex showed the first commercially available instrument [3]. Ion chromatographic methods have been developed especially for drinking and wastewater analyses; however, other applications (e.g., rainwater and swimming pool water) are of interest as well.

Ion exchange is the primary separation mode used in modern ion chromatography, although other approaches used for separation of inorganic anions and cations include ion interaction, ion exclusion, and chelation chromatography. Determined ions are being separated on the ion-exchange column, and these ions together with the eluent are being moved to the suppressor. In the suppressor, the conductance of the eluent is lowered (so-called "suppressed") and the conductance of the sample ions is increased, leading to a large increase in the detected ion signal to noise ratio [4]. The conceptualization of suppressed conductivity by Small *at al.* [3] was the seminal idea in the development of ion chromatography. A development and usage of suppression devices for the conductometric detection of inorganic ions by ion chromatography was described by Haddad *et al.* [5].

Over the past years, large varieties of stationary phases with different selectivity and capacity have been developed for both anion-, and cation-exchange chromatography. The stationary phases used in ion chromatography have been usually polystyrene-divinylbenzene (PS-DVB), polymethacrylate and polyvinyl resins. Recently, there is an increasing interest in using porous monolithic stationary phases for high-performance separation of inorganic and organic ions [6].

A variety of eluents can be used for anion separation, although bicarbonate solutions are the mainstay eluents in suppressed ion chromatography. The ideal eluent seems to be hydroxide, since, after suppression, it forms water that has virtually zero conductance, and therefore provides the perfect conductivity

baseline. However, hydroxide eluent is difficult to use because it readily absorbs carbon dioxide and forms carbonate [7].

Through the choice of the stationary phase and eluent composition, the selectivity can be modulated but the eluent must meet requirements of the detection system. Although the conductivity detector is still the most popular, other types of detection can be applied for different analytes. These include the following methods: electrochemical (amperometric, pulsed and integrated amperometric, potentiometric), photometric (UV/VIS, indirect photometric following post column derivatisation, chemiluminescence), and fluorescence. Ion chromatography plays a very important role in hyphenated techniques used in species analysis [8]. Even though speciation analysis with hyphenated methods has tremendously developed over the last decades, it is still a relatively new field of the analytical chemistry. Its further development depends on many factors such as the development of new sample preparation, separation and detection methods, and the availability of new certified reference materials. Hyphenated methods offer great possibilities and their main advantages include extremely low limits of detection and quantification, and very good precision and repeatability of determinations [9].

Ion chromatography can be used for the determination of ionic solutes such as inorganic anions, inorganic cations (including alkali metals, alkaline earth metals, transition metals and rare earth metals), carboxylic, phosphonic and sulfonic acids, detergents, carbohydrates, low-molecular-weight organic bases, and ionic metal complexes. It is applicable to the determination of ions in many sample matrix types, although the determination of inorganic ions in water continues to be the most widely used application. Ion chromatography offers several advantages over conventional methods of inorganic analysis [10] as follows:

- A short time of analysis;
- Sensitivity on the $\mu g \, L^{-1}$ level;
- High selectivity in samples with complex matrix;
- Simple water sample pretreatment;
- Small sample volume;
- simultaneous determination of anions and cations, or inorganic and organic ions, and species analysis (e.g., $NO_2^-/NO_3^-/NH_4^+$; $SO_3^{2-}/SO_4^{2-}/S^{2-}$; $H_2PO_4^-/HPO_4^{2-}/PO_4^{3-}$; Br^-/BrO_3^-; $Cl^-/ClO_2^-/ClO_3^-/ClO_4^-$; $Cr(III)/Cr(VI)$; $Fe(II)/Fe(III)$); and,
- The use of cheap, safe and environment friendly chemicals.

Acceptance of ion chromatography for the anions analysis was very rapid, primarily due to the lack of alternative methods that could determine multiple anions in a single analysis. However, the situation regarding the analysis of cations was quite different, due to many rapid and sensitive spectroscopic

techniques, such as AAS, ICP-AES, ICP-MS, as well as polarography and stripping voltammetry.

More than 25 years ago, capillary electrophoresis appeared as a promising substitute for the ion chromatography mainly because of its higher speed of separation. Comparison of ion chromatography and capillary electrophoresis has shown that these techniques can be considered as complementary rather than competitive [11]. Nevertheless, until now, there are no international standards applied for capillary electrophoresis methods. Comparison of the advantages and limitations of ion chromatography and capillary electrophoresis is given in Table 1.

Table 1. Advantages and limitations of ion chromatography and electrophoresis methods

Ion chromatography	
Advantages	Limitations
• Wide range of applications • Different detection modes • Repeatability • Standards methods	• The limited speed of analysis • Higher cost compared to capillary electrophoresis

Electrophoresis methods	
Advantages	Limitations
• Speed of analysis • High separation efficiency • Good tolerance to high pH • Low running costs	• Instability • Poor traceability and repeatability • A limited number of applications

Regulators and clients expect to receive meaningful and comparable results from the laboratory. Legislators generally define which of different validated methods should be applied to analyse water and wastewater samples. In general, standard methods can be chosen to serve as a reference method, but the laboratory serving a public client should apply reference methods. The best methods for inorganic anions determinations should meet the following requirements: the determination of target ions with a limit of determination on 10% of maximum acceptable concentration, simple sample treatment, a short time of analysis, the low cost of single analysis, and method availability. Ion chromatography methods meet these requirements, and they can be used for routine applications in routine laboratories.

Standardization on the international level is the responsibility of International Standardization Organization (ISO). ISO Technical Committee 147, founded in 1971, is responsible for the standardization in the field of water quality. During the standardization process, the draft standard methods had to go through a validation procedure, including checks for trueness, precision,

recovery, and finally an inter-laboratory trial before publishing as a standard method.

The normative part of an analytical standard method in ISO includes at least the following clauses: scope, normative references, interferences, principle, essential minimum requirements, reagents, apparatus, quality requirements for the separator column, sampling, and sample pre-treatment, procedure, calculation, expression of results and test reports. Standard methods can be adopted as a recommendation on a voluntary basis by any laboratory around the world. Governments can decide to incorporate existing standards into their national standards.

Considering that several individual wet chemistry methods for common inorganic anions or cations could be replaced by one fast and reliable IC procedures, it is not surprising that ion chromatography quickly became accepted worldwide by regulatory bodies to be used for the analysis of anions and cations in water and wastewater. Ion chromatography can be considered a well-established, mature technique for the analysis of anions and cations and many organizations, such as ISO, US EPA, ASTM (American Society for Testing and Materials), or AOAC (Association of Official Analytical Chemists) have their standards or regulatory methods of analysis based upon it [12]. Recently, several ion chromatography standards concerning ion determination in water and wastewater have been published. Only one standard concerns cation determination. All ISO ion chromatography standards are based on suppressed conductivity detection, although two of them (ISO 10304 part 3, and part 4) allow using UV/Vis and amperometry detection mode. US EPA published 7 ion chromatography methods, out of which 3 (Methods: 317.0, 321.8, and 326.0) concern determination of oxyhalide disinfection by-products (bromate, chlorite, and chlorate). It is noteworthy that two US EPA Methods (314.1, and 332.0) published the analysis of perchlorate, which is a new challenge in analytical chemistry [13]. After the publication of US EPA, and particularly ISO ion chromatography standards the number of laboratories applying this technique have increased dramatically. For those laboratories, ion chromatography is a reliable and economic supplement for existing wet chemical methods.

The application of ion chromatography with suppressed conductivity detection for the simultaneously separation and detection of common inorganic anions in water and wastewater samples was the aim of the work presented below.

1. Experimental

1.1. Apparatus

The 761 Compact IC System from Metrohm AG (Herisau, Switzerland) consisting of a MSM II (Metrohm Suppressor Module) suppressor and conductometric detector was used. Anion separation was carried out on Metrosep A Supp 5 (150 mm × 4 mm and 250 mm × 4 mm) columns from Metrohm. The volume of a sample injection loop was 20 µL. The IC Net 2.3 Metrodata (Metrohm) software was used for data acquisition and evaluation of chromatograms.

Filters of 0.45 µm pore size (MCE, Fisherbrand) were used for sample filtration throughout the experiments.

1.2. Reagents

All reagents used in the work were of analytical grades. Water purified in a Millipore Elix3/Simplicity UV system (a specific resistance >18.2 MΩ cm) was used in all experiments.

Standard solutions of inorganic (fluorides, chlorides, nitrates(III), bromides, nitrates(V), phosphates and sulphates) anions prepared from high purity sodium salts, dedicated to IC (from Fluka) and containing 1000 mg L^{-1} of each anion, were used. Multi-ionic working solutions containing inorganic anions within the following concentration (mg L^{-1}) ranges: F^- (0.4-10), Cl^- (0.8-75), NO_2^- (0.4-10), Br^- (0.4-10), NO_3^- (0.6-50), PO_4^{3-} (0.8-75), and SO_4^{2-} (0.6-25) were used for the calibration procedure and the identification of the analytes in the examined samples.

Sodium carbonate (Na_2CO_3) and sodium hydrogen carbonate ($NaHCO_3$) from Fluka were used for the preparation of the eluent: 3.2 mmol L^{-1} Na_2CO_3 + 1.0 mmol L^{-1} $NaHCO_3$.

1.3. Water and wastewater samples examined

Various types of drinking waters (medicinal, tap, Oligocene, well and mineral) were examined. Medicinal (*Henryk, Jan, Józef, Słotwinka, Wielka Pieniawa, Zuber*) and mineral (*Cisowianka, Kryniczanka, Muszynianka i Staropolanka 2000*) waters were bought at the local market. The samples of tap and Oligocene waters were collected in Warsaw, while well waters in the rural area of Mazovia.

Test samples of waters were collected in 30 ml polyethylene bottle (brand-clean, washed with deionised water and dried). All samples and standard solutions were stored in the refrigerator in the dark. An example of

chromatogram of chloride, nitrate, sulphate, and chromate obtained for industrial wastewater is given on Figure 1.
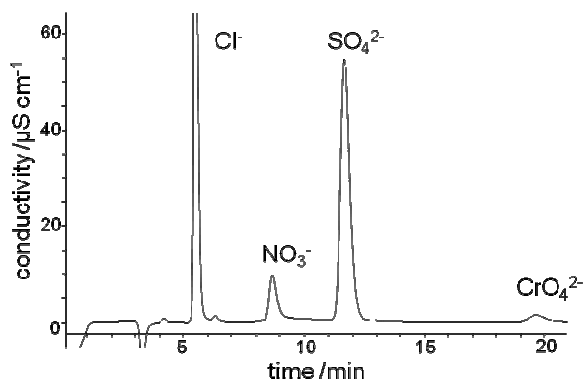

Fig. 1. Chromatogram of common inorganic anions and chromate in industrial wastewater (IonPac AS14 Dionex column, 5.0 mmol L^{-1} Na_2CO_3 + 1.0 mmol L^{-1} $NaHCO_3$ eluent)

2. Results and discussion

2.1. Calibration and method validation

Figure 2 presents the example of the synthetic anion mixtures chromatograms used for the calibration procedure. The concentrations ranges were chosen according to the expected concentration of anions in analysed samples. All calibration solutions were analysed in triplicate under optimized chromatographic conditions.
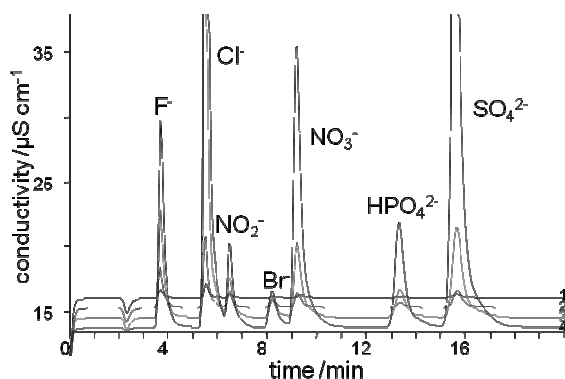

Fig. 2. The ion chromatograms of the used calibration solutions (1 – 0.4 mg L^{-1} F^-, NO_2^- and Br^-, 0.8 mg L^{-1} Cl^-, PO_4^{3-} and SO_4^{2-} and 0.6 mg L^{-1} NO_3^-; 2 – 2 mg L^{-1} F^-, NO_2^- and Br^-, 4 mg L^{-1} Cl^- and PO_4^{3-} and 3 mg L^{-1} NO_3^- and SO_4^{2-}; 3 – 5 mg L^{-1} F^-, NO_2^- and Br^-, 20 mg L^{-1} Cl^- and PO_4^{3-}, 15 mg L^{-1} NO_3^- and 10 mg L^{-1} SO_4^{2-}; 4 – 10 mg L^{-1} F^-, NO_2^- and Br^-, 75 mg L^{-1} Cl^-, PO_4^{3-}, 50 mg L^{-1} NO_3^- and 25 mg L^{-1} SO_4^{2-}; Metrosep A Supp 5 Metrohm column (length 150 mm), 3.2 mmol L^{-1} Na_2CO_3 + 1.0 mmol L^{-1} $NaHCO_3$ (0.7 mL min^{-1}) eluent)

The validation process was performed by testing different parameters affecting the measurements of common inorganic anions. Such parameters included the method of relative standard deviations (RSD), method detection and quantification limits (Table 2), and analysis of spiked samples and recovery. The limit of detection (LOD) and the limit of quantification (LOQ) were calculated as six and ten times, respectively, the ratio between the standard deviation of the regression and the slope of the calibration line.

Table 2. Limits of detection (LOD), limits of quantification (LOQ) and relative standard deviations of the results obtained for synthetic anion mixtures

ion	LOD	LOQ	RSD
	mg L^{-1}		/%
F^-	0.02	0.06	0.6–2.8
Cl^-	0.01	0.03	0.1–1.9
Br^-	0.01	0.03	0.7–4.5
NO_2^-	0.10	0.30	0.5–2.3
NO_3^-	0.02	0.06	0.5–4.7
PO_4^{3-}	0.05	0.15	0.6–2.7
SO_4^{2-}	0.01	0.03	0.4–2.8

2.2. The examination of water samples

Figure 3 presents the examples of chromatograms of medicinal, well, tap, and Oligocene waters. The results obtained for examined water samples are given in Table 3. As can be seen from the results, the majority (53.3%) of well water samples and some (*Henryk, Słotwinka and Zuber*) medicinal waters contained higher concentrations of nitrite and nitrate than the admissible concentrations of these anions in waters intended for human consumption [18] and medicinal waters [17], respectively. In the examined tap, Oligocene and mineral water samples nitrites were not detected under the conditions used. Exceeding the legal standards was also noted for sulphate in well waters.

The highest fluoride concentrations contained medicinal (up to 0.69 mg L-1) and Oligocene (up to 0.68 mg L-1) waters. Having regard to the possibility of the occurrence of fluorides in various foods (e.g., tea [20, 21]), the daily intake of drinking water and acceptable daily intake of fluorides (0.1 mg for children under one year of age and 4.0 mg for adults [22]), must use caution when choosing a drinking water especially for young children and infants.

Our results confirm the good quality of tap, mineral and Oligocene waters in terms of the content of inorganic anions and indicate the need to pay more attention to the quality of water known as medicinal and water from domestic wells.

Precision of the method used, evaluated as the coefficient of variation (CV) of the results obtained for sixfold well water sample analysis, was in the range of 0.7% for chlorides to 6.1% for fluorides. The accuracy of obtained results was evaluated by a standard addition method. The recoveries (%) of the anions determined in the work were in the ranges of: 96.8–104.2 (F^-), 99.3–107.4 (Cl^-), 99.0–110.3 (NO_2^-), 93.6–108.3 (Br^-), 91.5–119.9 (NO_3^-), 95.2–99.9 (PO_4^{3-}) and 100.1–116.4 (SO_4^{2-}).
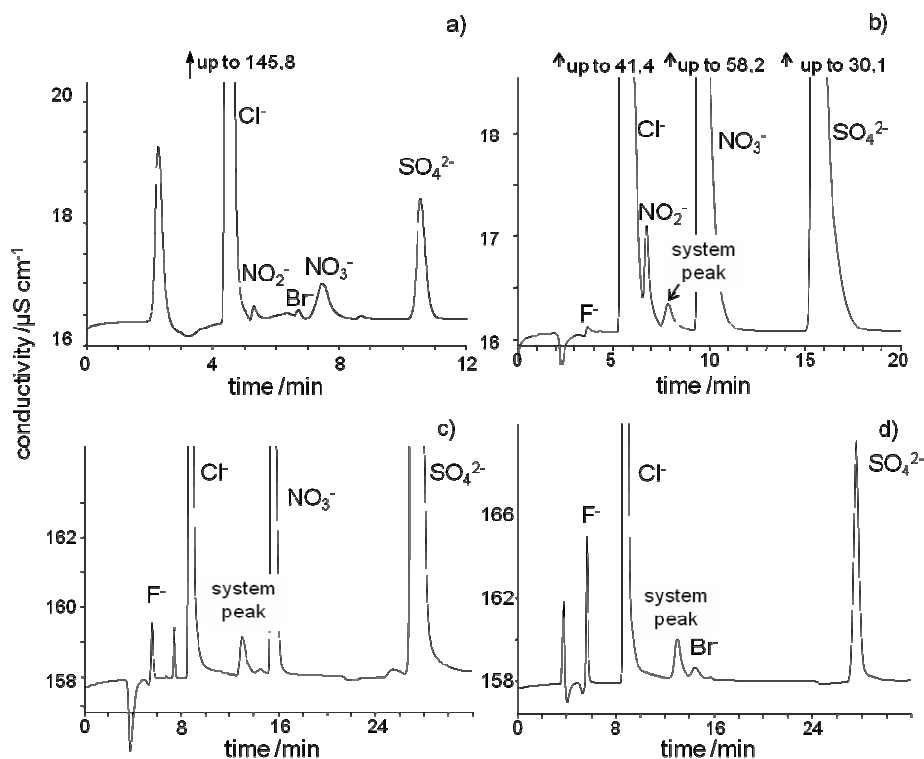

Fig. 3. The ion chromatograms of examined water samples: (a) medicinal, (b) well, (c) tap, and (d) Oligocene (Metrosep A Supp 5 Metrohm column, 3.2 mmol L^{-1} Na_2CO_3 + 1.0 mmol L^{-1} $NaHCO_3$ (0.7 mL min^{-1}) eluent and different column length: a, b 150 mm and c, d 250 mm)

Table 3. Concentration ranges of determined anions in examined water samples and the maximum admissible concentrations of these ions

Ion	Concentration range /mg L^{-1}					Admissible concentration / mg L^{-1}		
	Medicinal [14]	Tap	Oligocene [15]	Well [16]	Mineral	Medicinal [17]	Intended for human consumption [18]	Mineral [19]
F$^-$	<LOD–0.69	<LOQ–0.29	0.44–0.68	<LOQ–0.49	0.13–0.58	–	1.5	5.0
Cl$^-$	3.0– 1024	46.7–70.9	60.1–100.7	2.6–249.2	1.8–7.1	–	250	–
NO$_2$$^-$	<LOD–4.5	<LOD	<LOD	<LOD–2.28	<LOD	0.02	0.5	0.1
Br$^-$	<LOD–7.01	0.17–1.58	<LOD–0.63	<LOD–0.30	<LOD–3.10	–	–	–
NO$_3$$^-$	<LOD–16.3	3.3–17.6	<LOD–3.9	0.6–506.7	<LOD–3.1	10	50	50/10*
PO$_4$$^{3-}$	<LOD	<LOD	<LOD	<LOD–20.7	<LOD	–	–	–
SO$_4$$^{2-}$	3.4–63.8	25.8–104.8	1.3–19.2	12.7–288.6	0.7–26.3	–	250	–

* the maximum concentration of 10 mg L^{-1} concerns waters extracted from aquifers on Polish territory.

3. Conclusions

There is a need for reliable control and monitoring of many substances present in water and wastewater in order to prevent contamination and protect the environment. The majority of disposed wastes, including recyclable waste, are not environmentally safe. The presence in sewage sludge with excess levels of the potentially toxic elements such as chromium could affect the ability to recycle these residues in the future. Every day in hundreds of thousands of laboratories around the world, millions of analyses of different samples are carried out. The development of new methods and the improvement of existing ones are major tasks for analytical chemists. Advances in analytical instrumentation, detection systems, and separation techniques have provided analytical chemists the tools required to continually lower detection limits and improving methods reliability in many instances. Ion chromatography is an innovative analytical technique that has significantly improved investigations of ions in water and wastewater. The most routine ion chromatographic methods have been standardized over the last 20 years. The technique is applicable to the determination of ions in many sample matrix types, although the determination of inorganic ions in waters and wastewaters continues to be the most widely applied. The most important advantages of ion chromatography are the broad range of applications, well-developed hardware, many detection options, reliability (good accuracy and precision), high selectivity, high speed, high separation efficiency, good tolerance to sample matrices, and the low cost of consumables. Ion chromatography is widely accepted as standard reference methodology in water and wastewater analysis. Ion chromatography separation

will continue to be developed as more and more inorganic contaminants (e.g., perchlorate, chromate, cyanide) become regulated at lower and lower limits in the future.

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Chromatografia jonowa – metoda oznaczania jonowych składników wód i ścieków

Słowa kluczowe

Chromatografia jonowa, analiza wody, analiza ścieków, nieorganiczne jony, normalizacja/standardyzacja.

Streszczenie

Badania wód i ścieków stanowią istotny element analizy chemicznej. Rozwój nowych metod i ulepszanie istniejących to główne zadanie chemików analityków. Istnieje potrzeba ścisłej kontroli i monitorowania wielu substancji obecnych w wodzie i ściekach, żeby zapobiegać zanieczyszczeniu środowiska i chronić go. Substancje zawarte w różnych rodzajach wód mogą być klasyfikowane jako biologiczne, chemiczne (zarówno nieorganiczne, jak

i organiczne), fizyczne i zanieczyszczenia radiologiczne. W wielu laboratoriach w ramach rutynowej analizy najczęściej oznaczane są nieorganiczne aniony i kationy. Chromatografia jonowa jest atrakcyjną techniką, szczególnie dla laboratoriów, które muszą oznaczać wiele anionów i kationów w kilku tysiącach próbek, jednak nie posiadają tak dużej wydajności, żeby uzasadnić zakup dużych automatycznych analizatorów, zwykle opartych na procedurach kolorymetrycznych. Chromatografia jonowa eliminuje potrzebę stosowania szkodliwych odczynników, które są często integralną częścią klasycznej chemicznej analizy. Żądania ze strony jednostek nadzorujących uzasadnianie analitycznych wyników oraz ze strony laboratoriów zwalidowanych metod doprowadziły do konieczności standardyzacji metod oznaczania z zastosowaniem techniki chromatografii jonowej.