

Comparison of Ion Chromatography and Isotachophoresis for the Determination of Selected Anions in Atmospheric Wet Deposition Samples

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Abstract

Isotachophoresis using coupled capillaries (ITP) and ion chromatography (IC) – two analytical procedures for the determination of anions in atmospheric wet deposition – were compared. Both techniques were used for the determination of anions in small volume samples of dew, rime and fog deposition. IC was characterized by lower limits of detection and quantitation. The wide dynamic range of the technique made it suitable for accurate and precise determination of the analytes in samples of widely different matrices and analyte levels. No significant differences were observed between the two techniques in terms of time- and labor intensity.

Keywords: anions, dew, fog, ion chromatography, isotachophoresis, rime

Introduction

Interest in atmospheric deposition has been growing in recent years because of the large load of pollutants carried by the atmosphere. A number of new research programs have been established in different countries focused not only on atmospheric precipitation, but also on atmospheric deposition.

Analysis of atmospheric deposition samples is a complicated and difficult process. Proper sampling methods are particularly important, as errors committed at this stage cannot be estimated and/or corrected. The choice of the sampling device is determined by the form and manner in which the deposition reaches the surface of the Earth [1].

The analysis is complicated by a number of factors, including the specific composition and non-uniformity of the samples, low analyte levels, decreasing analyte concentrations with increasing amount of deposition, and possible interactions between the analytes in the collected samples. In addition, the volumes of the deposition samples are usually very small, hence the analytical procedure used must be suitable for low volume samples. An optimal analytical procedure should be fast, simple, inexpensive, accurate, precise and sensitive.

Most important is to introduce the proper system of quality assurance and quality control (QA/QC) if one wants to have reliable analytical data which could be considered as a source of information. Analytical procedures and techniques have to be metrologically characterized and evaluated [2].

Table 1. Operating parameters of the IC and ITP units.

Method	Column	Eluent/Electrolyte	Injection volume [ml]	Flow rate [ml/min]	Analysis time [min]	Recommended sample volume use for analysis [ml]
IC	IonPac AS9-HC, 2 x 250 mm	9.0 mM Na ₂ CO ₃	0.0075	0.25	15-20	2
ITP		terminating electrolyte: 5·10 ⁻³ M citric acid	1		25-30	4
	Teflon preseparation column, 90 mm, i.d. 0.8 mm	leading electrolyte: 8·10 ⁻³ M HCl 3.8·10 ⁻³ M BisTrisPropane 1.5·10 ⁻³ M β-alanine 0.1 % hydroxyethylcellulose pH = 3.68				
	Teflon analytical column, 160 mm, i.d. 0.3 mm	leading electrolyte: 2·10 ⁻³ M HCl 1.5·10 ⁻³ M β-alanine 0.1 % hydroxyethylcellulose pH = 3.54				

Ion chromatography (IC) is considered the standard method for ion analysis in environmental matrices [3]. IC is an accurate and precise technique, but it suffers from some intrinsic limitations, such as low efficiency and selectivity due to difficulties with developing gradient elution methods, and the high price of the analytical columns, especially ones tailored for specific applications.

All capillary electrophoretic methods use an electric field applied on a capillary to achieve separation. Individual methods differ in separation principles, but two CE methods have an identical separation principle, namely, capillary isotachopheresis (ITP) and capillary zone electrophoresis (CZE). Both methods are based on the same physico-chemical property, i.e. effective mobility. The practical difference between the two techniques lies in a different arrangement of the buffering electrolyte [4]. CZE represents separation in a continuous electrolyte (background electrolyte), whereas ITP is an example of a technique employing a discontinuous electrolyte system [5] (leading and terminating electrolyte, respectively).

Capillary electrophoresis (CE) has been shown to be a viable alternative to ion chromatography (IC) for the determination of inorganic ions and other low-molecular-mass ionic solutes [6-20]. CE offers a number of advantages over IC, including simplicity, low operating costs, greater separation efficiency, unique selectivity and a high degree of matrix independence, all of which make it ideal for the analysis of ionic solutes in very complex matrices such as wet deposition samples [21-25]. In contrast, IC, like any chromatographic method, is a dilution method where the original sample concentration is constantly decreased by various effects such as free diffusion, Eddy diffusion, and mass transfer. For detection, however, an inherent weakness of CE compared to liquid chromatographic methods

becomes apparent. This is in opposition to IC, which effectively uses conductivity detection employing high equivalent conductivity as an inherent property of inorganic analytes [26].

Conductivity detection – used universally in combination with IC – has been considered for CZE of rainwater [13]. The construction of conductivity detectors for CZE and its coupling with the capillary and maintenance, however, is more laborious than for IC, due to the reduced dimensions. Interference of the high separation electric field and fouling of the small electrodes exposed to the samples can cause additional problems. Fortunately, by increasing the frequency of the excitation signal during the conductivity measurements (oscillometry), the electrodes can be installed outside of the capillary, overcoming all these problems [13, 27].

Analytical capillary isotachopheresis (ITP) is a powerful tool for the separation of compounds exhibiting very small differences in net electrophoretic mobility [28-30]. ITP is a simple, sufficiently sensitive and inexpensive method (cheaper than CZE) and therefore suited for routine analysis. The technique offers a number of features such as the ability to preconcentrate dilute samples and the capacity to handle a wide variety of concentrations, which can be of benefit when performing practical analytical measurements. However, to perform an isotachopheretic separation it is necessary to use a discontinuous electrolyte system comprising of a leading electrolyte and a terminating electrolyte. The leading electrolyte must contain an ion with a higher mobility than the sample species and the terminating electrolyte an ion with a lower mobility than the sample species. The latter requirement can usually be realized easily but the former can cause problems if there is a requirement to analyze ions which exhibit high electrophoretic mobility.

Table 2. Calibration curve parameters, limits of detection (LOD) and quantitation (LOQ), as well as measurement ranges for IC and ITP.

Analyte	IC				ITP				LOQ _{ITP} / LOQ _{IC}
	Range	Regression coefficient r	LOD	LOQ	Range	Regression coefficient r	LOD	LOQ	
	[mg/l]		[mg/l]		[mg/l]		[mg/l]		
F ⁻	0.5-1.5 0.033-0.1	0.998 0.996	0.011	0.033	0.74-1.5	0.904	0.25	0.74	16
Cl ⁻	20-90 0.03-0.1	1.000 0.990	0.0090	0.030	22-90	0.995	7.3	22	733
NO ₂ ⁻	0.5-2 0.16-0.5	0.990 0.996	0.055	0.16	0.39-2.0	0.991	0.13	0.39	2
NO ₃ ⁻	10-40 0.042-0.1	1.000 0.993	0.014	0.042	2.1-40	0.992	0.68	2.1	53
PO ₄ ³⁻	1-2 0.12-0.2	0.984 0.998	0.041	0.12	0.76-2.0	0.984	0.25	0.76	6
SO ₄ ²⁻	25-125 0.048-0.1	1.000 0.990	0.016	0.048	1.6-125	0.999	0.55	1.6	33

This paper presents a comparison of the two analytical techniques for the determination of F⁻, Cl⁻, NO₃⁻, NO₂⁻, SO₄²⁻ and PO₄³⁻ anions in the deposition samples mentioned above. Basic validation parameters of the techniques were determined, including linearity, repeatability, LOD and LOQ (uncertainty, limit of detection and limit of quantitation, respectively). The article also describes the use of IC and ITP for the analysis of inorganic ions in samples of fog, rime and dew collected in the city of Gdańsk, Poland.

Experimental

Sampling

Samples were collected between August 2005 and January 2006 in the Gdańsk-Sopot-Gdynia Tricity (fog deposition in Gdańsk Osowa, rime in Sopot and dew in Gdynia). Samples of dew and rime were collected using dedicated samplers of known geometry exposed to the atmosphere. Dew samples were collected using a sampler based on the design described by Muselli [31]. The collecting surface of this sampler (2,000 by 2,000 mm) was made of rigid polyethylene foil mounted on a wooden frame. The sampler was mounted at a 30-degree angle to promote the flow of condensation droplets to a collector (groove) and subsequently to a collection vessel (50 ml flask). Droplets remaining on the collection surface were transferred to the collector using a polyethylene scraper.

Fog samples were collected using a passive wet deposition collector placed 180 cm above ground. The receptor consisted of a network of 130 lines stretched in the form of an inverted cone, covered on top with a round hood. The total collecting area was 90 cm². Rime samples were collected using a passive dry deposition collector consisting of a duralumin rod of 400 mm length and 30 mm diameter, placed 2 m above ground.

Atmospheric deposition samples were collected during or immediately after a precipitation event. They were stored at low temperature without chemical preservatives because the analysis was performed either directly on-site, or immediately after the samples were delivered to the laboratory. Because the samples were usually highly contaminated with solids (sand, leaves, etc.), they had to be pre-filtered (0.45 µm, Millex[®]-HV).

Chemicals

Standard solutions of chloride, nitrite, nitrate, sulfate, fluoride and phosphate anions (1,000 mg/l) were obtained from Merck (Germany). All other standard solutions were prepared from sodium salt solutions of the same concentration. BisTrisPropane and β-alanine were from Sigma Aldrich (USA). HCl and citric acid were from POCH (Poland). Hydroxyethylcellulose was from SERVA (Germany). All chemicals were of analytical grade. Water was purified using the Milli-Q[®] Millipore system.

Methods

IC experiments were carried out using a DX 500 IC system (Dionex Corporation, USA) consisting of a chromatographic module, a gradient pump and a conductivity detector. Detection was performed with the anion suppressor (ASRS[®]-ULTRA, 2 mm) in the recycle mode. Data collection and processing were carried out using a PeakNet 4.3 chromatographic data system (Dionex).

We used an ItaChrom EA 101 isotachopheric analyzer from Villa Labeco s.r.y. (Spisska Nova Ves, Slovakia). The instrument was equipped with a Teflon pre-separation column and an analytical column, both with conductivity detection. The current in the pre-separation column was 250 µA, and in the analytical column 7.5 µA. The isotachopherograms were evaluated with the help of a PC software

Table 3. Repeatability and uncertainty of the results obtained for standard solutions and real samples using IC and ITP.

Standard solution								
Ion	Concentration [mg/l]	Ion chromatography		Isotachophoresis		CV _{ITP} / CV _{IC}	U _{ITP} / U _{IC}	
		Repeatability* CV [%]	Uncertainty U [%]	Repeatability* CV [%]	Uncertainty U [%]			
Cl ⁻	20	0.32	4.1	0.53	21	1.7	5.1	
	40	0.15	1.8	0.19	9.2	1.3	5.1	
	60	0.15	1.2	0.15	5.9	1.0	4.9	
	75	0.12	1.0	0.32	5.0	2.7	5.0	
	90	0.060	0.90	0.15	4.5	2.5	5.0	
F ⁻	1.0	1.2	4.3	7.8	29	6.5	6.7	
	1.25	3.1	4.9	5.0	23	1.6	4.7	
	1.5	0.59	3.1	4.0	21	6.9	6.8	
NO ₂ ⁻	0.5	3.5	25	7.0	24	2.0	1.0	
	1.0	2.7	11	6.6	12	2.4	1.1	
	1.5	1.8	7.6	3.4	7.6	1.9	1.0	
	2	1.9	6.6	2.1	6.1	1.1	0.9	
NO ₃ ⁻	10	0.71	4.2	1.4	22	2.0	5.2	
	20	0.32	1.8	2.1	9.9	6.5	5.5	
	30	0.14	1.2	0.59	6.4	4.2	5.3	
	40	0.070	1.0	0.77	5.6	11	5.6	
PO ₄ ³⁻	1	0.74	9.8	4.0	19	5.5	1.9	
	1.5	4.9	7.9	3.3	11	0.68	1.4	
	2.0	2.4	5.7	2.3	9.8	0.95	1.7	
SO ₄ ²⁻	25	0.55	4.3	1.1	9.8	2.1	2.3	
	50	0.37	2.0	1.0	4.5	2.8	2.2	
	75	0.32	1.3	0.52	2.9	1.6	2.2	
	100	0.27	1.0	0.32	2.2	1.2	2.2	
	125	0.44	1.0	0.45	2.0	1.0	2.0	
Real samples								
	IC	ITP						
Cl ⁻	4.88	<LOQ	0.40	0.80	-	-	-	-
NO ₃ ⁻	13.3	22.1	0.67	1.2	0.93	13	1.4	11
PO ₄ ³⁻	0.57	0.53	2.6	5.2	7.4	37	2.8	7.2
SO ₄ ²⁻	14.3	13.6	0.31	0.70	2.0	19	6.4	27

*Calculated as the average of the coefficients of variation (CV) obtained for 2 measurement series consisting of 3 replicates at each concentration level.

package supplied with the analyzer (KasComp Ltd., Slovakia). Table 1 lists the operating parameters of both instruments.

Results and Discussion

Quality Control

A comparison of the values of the validation parameters, costs, and time- and labor intensity of ion chromatography and isotachophoresis was carried out to evaluate the

usefulness of these techniques in the analysis of anions in atmospheric deposition and to facilitate the choice of a more suitable technique. Table 2 lists the values of the calibration curve parameters, limits of detection (LOD) and quantitation (LOQ), as well as the dynamic ranges of both techniques. The concentration ranges used for the determination of the calibration curves were established according to the guidelines described in the STN 757430 Slovak standard [32]. The values of the regression coefficients indicate that the calibration curves were linear for both techniques in the concentration range examined.

Table 4. Results of determination of the analytes in certified reference materials.

Ion	IC [mg/l]	Recovery [%]	ITP [mg/l]	Recovery [%]	Analytical Reference Material Rain-97 [mg/l]
Chloride	0.640 ± 0.0454	122	<LOQ	-	0.526 ± 0.0942
Nitrate (N)	2.26 ± 0.075	108	<LOQ	-	2.09 ± 0.196
Sulfate	5.74 ± 0.046	109	6.6 ± 2.7	125	5.28 ± 0.730
					Reference Material No 409 [mg/l]
Chloride	3.91 ± 0.035	97.5	<LOQ	-	4.01 ± 0.071
Nitrate (N)	1.09 ± 0.075	100	<LOQ	-	1.09 ± 0.014
Sulfate	5.14 ± 0.036	101	5.6 ± 2.7	110	5.11 ± 0.067

In Table 3 results of comparison of the reproducibility of both techniques in the analysis of standard solutions and real samples. The coefficients of variation (CV [%]) for chloride, nitrite and sulfate anions in the individual measurement series were comparable for both techniques. In addition, expanded uncertainty (the interval around the measurement result in which the expected value should be present at a given probability level) was also estimated.

Expanded uncertainty is affected by the uncertainty related to calibration and by reproducibility of the measurements. The large uncertainties observed for chloride ions at 20 mg/l and fluoride ions at 1 mg/l in isotachopheresis were related to the high limits of detection of these ions in this technique. The ability to analyze nitrate, chloride and sulphate simultaneously is difficult to realise using isotachopheresis because all three of these anions possess high

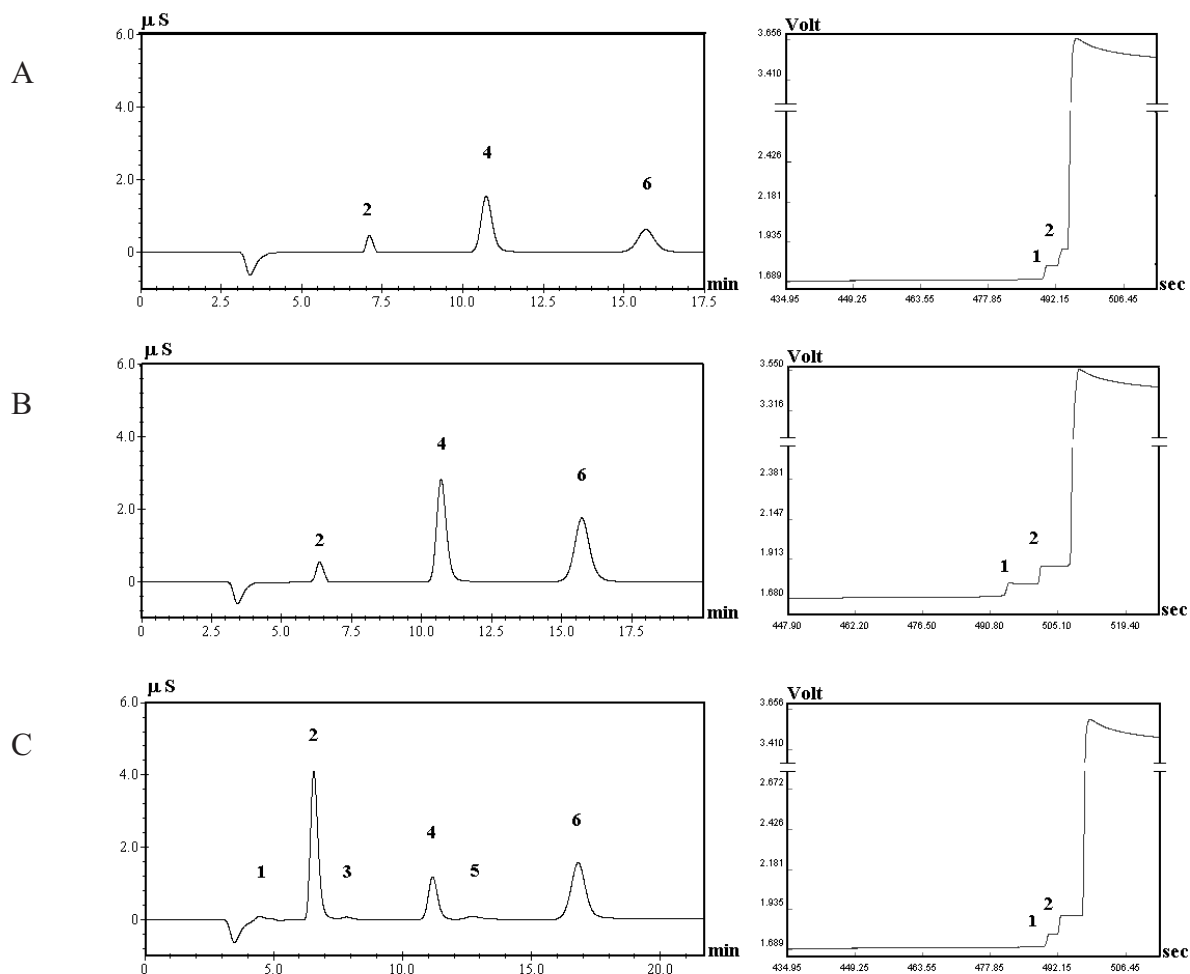


Fig. 1. Examples of chromatograms and isotachopherograms obtained in the analysis of anions in dew (A), rime (B) and fog deposition samples (C); 1 - F⁻, 2 - Cl⁻, 3 - NO₂⁻, 4 - NO₃⁻, 5 - PO₄³⁻, 6 - SO₄²⁻.

Table 5. Target anion concentrations in samples of atmospheric precipitation.

No.	Ion	Concentration [mg/l]	No.	Ion	Concentration [mg/l]	No.	Ion	Concentration [mg/l]
Dew			Rime			Fog deposition		
IC								
1	F ⁻	<LOQ	1	F ⁻	<LOQ	1	F ⁻	0.23
2	Cl ⁻	0.99	2	Cl ⁻	1.01	2	Cl ⁻	6.89
3	NO ₂ ⁻	<LOQ	3	NO ₂ ⁻	<LOQ	3	NO ₂ ⁻	0.24
4	NO ₃ ⁻	5.74	4	NO ₃ ⁻	10.57	4	NO ₃ ⁻	4.65
5	PO ₄ ³⁻	<LOQ	5	PO ₄ ³⁻	<LOQ	5	PO ₄ ³⁻	0.54
6	SO ₄ ²⁻	2.96	6	SO ₄ ²⁻	8.39	6	SO ₄ ²⁻	7.92
ITP								
1	NO ₃ ⁻	16.4	1	NO ₃ ⁻	21.7	1	NO ₃ ⁻	15.7
2	SO ₄ ²⁻	3.41	2	SO ₄ ²⁻	9.91	2	SO ₄ ²⁻	9.71

electrophoretic mobilities. Chloride has a higher effective mobility than nitrate or sulphate. In most electrolyte systems developed for anionic separations using isotachopheresis, chloride is employed as the leading ion. The most successful of these saw the use of dithionate as a leading electrolyte, which has a higher mobility than chloride, thus enabling the direct and simultaneous isotachopheretic determination of chloride and other anions [30, 33]. The data presented in Table 3 confirm the fact that the matrix composition has as a significant influence on the uncertainty values in the case of ion determination by means of the isotachopheresis technique.

Accuracy of the two techniques was evaluated using standard reference materials: Reference Material No. 409 (BCR-409, Institute for Reference Materials and Measurements, Belgium) and Analytical Reference Material Rain-97 (National Water Research Institute, Environment Canada). Mean concentrations of the ions determined using the techniques under study fell within the confidence intervals given by the manufacturers (Table 4).

Application of the Techniques to the Analysis of Real Samples

Following the validation experiments performed on standard solutions, both techniques were used to determine the target anions in real samples of atmospheric precipitation (rime, fog deposition and dew). Fig. 1 presents examples of chromatograms and isotachopherograms obtained in the study, while Table 5 lists the concentrations of the target anions in the samples analyzed.

The results obtained indicate that ion chromatography allows the determination of a broader spectrum of anions owing to the lower limits of detection of the technique toward the target anions.

Conclusions

Both ion chromatography and isotachopheresis allow the determination of anions in small volume samples. In this study, both techniques were compared in terms of the validation parameters. IC was characterized by lower limits of detection and quantitation. The higher LOD/LOQ values for isotachopheresis were most likely related to the presence of chloride ions in the electrolyte. The wide measurement range of IC allows accurate and precise determination of anions in samples of different matrices and analyte concentration levels. In addition, IC is characterized by lower expanded uncertainty of the results.

No significant differences between the two techniques were observed in terms of time- and labor intensity. The total analysis time was 15-20 min. for IC, and 25-30 min. for ITP; however, IC required an additional sample preparation step (filtration). In spite of the lower operating costs and the possibility of full automation, isotachopheresis has significant limitations (e.g. for the determination of chloride ions).

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