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Optimization of the ion chromatographic quantification of airborne fluoride, acetate and formate in the Metropolitan Museum of Art, New York

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ABSTRACT

Ion chromatographic (IC) methods have been compared in order to achieve an optimal separation of fluoride, acetate and formate under various elution conditions on two formerly introduced analytical columns (i and ii) and a novel one (iii): (i) an IonPac AS14 (250 mm × 4 mm I.D.), (ii) Allsep A-2 (150 mm × 4.6 mm I.D.), and (iii) an IC SI-50 4E (250 mm (length) × 4 mm (internal diameter - I.D.)). The IC conditions for the separation of the anions concerned were optimized on the IC SI-50 4E column. A near baseline separation of these anions was attained on the IonPac AS14, whereas the peaks of fluoride and acetate could not be resolved on the Allsep A-2. A baseline separation for the three anions was achieved on the IC SI-50 4E column, when applying an eluent mixture of 3.2 mmol/L Na_2CO_3 and 1.0 mmol/L $NaHCO_3$ with a flow rate of 1.0 mL/min. The highest precision of 1.7, 3.0 and 2.8% and the best limits of detection (LODs) of 0.014, 0.22 and 0.17 mg/L for fluoride, acetate and formate, respectively, were obtained with the IC SI-50 4E column. Hence, this column was applied for the determination of the acetic and formic acid contents of air samples taken by means of passive gaseous sampling at the Metropolitan Museum of Art in New York, USA. Atmospheric concentrations of acetic and formic acid up to 1050 and 450 $\mu g/m^3$, respectively, were found in non-aerated showcases of the museum. In galleries and outdoors, rather low levels of acetic and formic acid were detected with average concentrations of 50 and 10 $\mu g/m^3$, respectively. The LOD data of acetate and formate on the IC SI-50 4E column correspond to around 0.5 $\mu g/m^3$ for both acetic and formic acid in air samples.

1. Introduction

Indoor air pollution has become a major concern in the deterioration of Cultural Heritage (CH) objects for the past decades. To minimize the damage to CH items, concentrations of harmful air pollutants in a CH environment should be kept as low as possible [1–4]. Acidic compounds, such as acetic and formic acid, have been shown to be dangerous for certain works of art displayed in CH environments (e.g., in museums and churches). Hence, they present a major problem for curators and conservators [3–5]. Inorganic materials including lead, bronze, copper, shells, limestone and ceramics can suffer irreversible damages (e.g., formation of efflorescence on the surface) by the attack of organic acids. The sources of these acids are well established, including wood and wooden products [6–8]. As wood and/or wooden composites (e.g., medium density fiberboard) are often used as construction materials for indoor environments, it is necessary to monitor the concentrations of gaseous acetic and formic acid in the indoor air of buildings displaying valuable CH items.

Atmospheric gaseous pollutants can be sampled based on molecular diffusion through a static air layer, or permeation through a membrane, with no active movement of air through the sampler at a physically controlled rate [4,9]. By one type of diffusion tube, four important gaseous pollutants can be sampled simultaneously, i.e., NO_2, SO_2, CH_3COOH and HCOOH. Additionally, hydrogen fluoride (HF) is sampled. The important advantages of using diffusion tubes are that there is no need for an electrical vacuum pump and that the dimensions of the tubes are quite small (60 mm (length) × 4.8 or 5.8 mm (internal diameter – I.D.)). Therefore, they can be used discretely in a narrow space without disturbance of the CH environment, e.g., within museum showcases.

The diffusion cartridges are desorbed in Milli-Q water, so the resulting anions can be analyzed with ion chromatography (IC). IC is an efficient analytical method for the determination of inorganic

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and low molecular mass organic ions present in aqueous samples at low, even μg/L, levels [10,11]. This technique is based on the affinity of ions in the mobile phase for oppositely charged ions on the stationary phase. The selectivity of the chromatographic column is of great importance, especially, for separating ions with very close retention times as in the case of fluoride–acetate and acetate–formate critical pairs. The availability of columns with stationary phases of different type, particle size and selectivity provides a range of options for solving an analytical separation task as mentioned above.

The most widely applied, commercially available column for the analysis of inorganic anions and low molecular mass organic acids is the IonPac AS4A-SC. This type of column was also adapted for the separation of acetate and formate using compounds of different affinity as eluents, at various concentrations and flow rates [12–14]. Secondly, the IonPac AS14 column has been widely applied for the analysis of inorganic anions, like F^-, Cl^-, $NO_2{}^-$, Br^-, $NO_3{}^-$, $SO_4{}^{2-}$, and $PO_4{}^{3-}$ [11,15]. According to the manufacturer, this column can also be used for the separation of inorganic anions and low molecular mass organic acids. Another column is the Allsep A-2, which has been studied in an earlier work for its separation characteristics for acetate and formate [16]. It was possible to separate these anions on both columns. Additionally, the separation of fluoride from acetate and formate was investigated. However, on the Allsep A-2 column, significant influence of the fluoride concentration was found, thus it was impossible to achieve good separation between fluoride and acetate. This is of certain importance for samples having high fluoride content, as can be the case for a polluted CH environment.

A third type of IC columns, appeared on the market in 2007, is the IC SI-50 4E. As will be shown in this article, this column offers a promising possibility for complete baseline separations of fluoride, acetate and formate. To the best of the authors' knowledge, this column has not yet been optimized and applied for the separation of the anions concerned.

The main aim of this study is to compare the separation of fluoride, acetate and formate on three different types of analytical anion-exchange columns, i.e., an IonPac AS14, an Allsep A-2 and an IC SI-50 4E. Additionally, the experimental conditions for the separation of these anions are optimized using the IC SI-50 4E column. As the retention behaviour of analytes depends mainly on the eluent concentration, mixtures of $NaHCO_3$ and Na_2CO_3 at varying concentrations and flow rates were selected. Much attention was paid on the influence of the fluoride content of the samples, since it has very similar retention behaviour to acetate. Poor accuracy and reproducibility for both of these ions were reported in the literature [17–19]. The optimized chromatographic conditions are subsequently applied to determine the acetic and formic acid contents of air samples taken in galleries and showcases of the Metropolitan Museum of Art, New York, USA.

2. Experimental

2.1. Air sampling and sample processing

Radiello® passive diffusion tubes (Fondazione Salvatore Maugeri, Padova, Italy) were used for the sampling of acetic acid, formic acid, NO_2, SO_2 and HF, according to the recommendation of the manufacturer [20]. The adsorbing cartridges of the tubes are made from microporous polyethylene and coated with triethanolamine (TEA). The exposure time of the cartridges was one week. During sampling, the pollutant gases were adsorbed on the adsorption cartridge of the diffusion tubes, i.e., bound in the form of fluoride (F^-), acetate (CH_3COO^-) and formate ($HCOO^-$). Since any airborne particle containing these species is not able to pass through the diffusive barrier of the sampler, this method is selective for gaseous compounds [21]. Each cartridge was leached for 1 h in 5 mL ultrapure water (Milli-Q, Millipore, Billerica, USA), and afterwards the leachates were analyzed by means of IC.

The sampling rates for acetic and formic acid, determined in a dynamic exposure chamber at a constant temperature of 298 K and a relative humidity of 50%, were found to be 97.3 ± 3.1 mL/min and 91.2 ± 3.9 mL/min, respectively [22]. The application of temperature correction was not necessary, since the storerooms of the museum are all air-conditioned at around 22 °C. Theoretically, a temperature increase of 5 °C should not cause more than 2.6% alteration in diffusivity [4]. Blank values of 1.61 ± 0.05 μg formate and 1.26 ± 0.20 μg acetate were obtained, corresponding to LODs for both acids of around 0.5 $\mu g/m^3$ for air samples. Sampling linearity tests for these acids with the use of Radiello diffusive samplers have already been reported [22,23].

2.2. Apparatus and columns

All measurements were carried out on a Dionex model DX-120 ion chromatograph equipped with an AS-50 (Dionex, Sunnyvale, CA, USA) autosampler. A 20 μL aliquot of each sample/standard was loaded into the eluent-stream. The ions were detected by suppressed conductivity of the eluent using an ASRS-ULTRA self-regenerating suppressor (Dionex).

For the separation of the concerned ions, three columns were studied:

(i) an IonPac AS14 (250 mm × 4 mm I.D.) analytical column with an AG14 (50 mm × 4 mm I.D.) guard column produced by Dionex. The resin of the IonPac AS4A-SC (250 mm × 4 mm I.D., Dionex, Sunnyvale, CA, USA) column is composed of 13 μm ethylvinylbenzene (EVB) cross-linked with 55% divinylbenzene (DVB) substrate, aminated with a latex containing hydroxyl group. The IonPac AS14 column (250 mm × 4 mm I.D.) is also packed with highly crosslinked (55%) EVB–DVB substrate particles of 9 μm diameter. Its anion exchange layer is functionalized with quaternary ammonium groups grafted to the surface, having an ion exchange capacity of 65 μeq/column.

(ii) an Allsep A-2 column (150 mm × 4.6 mm I.D.) with a GA-1 (7.5 mm × 4.6 mm I.D.) guard column, produced by Alltech (Deerfield, IL, USA). The traditional 100 mm Allsep A-2 column has been adapted to a custom-type 150 mm stainless steel column. The column is packed with a high capacity, methacrylate-based anion exchanger with quaternary amine functional groups. This material is hydrophilic and stable in the pH range of 2–11.

(iii) an IC SI-50 4E column (250 mm × 4 mm I.D.) analytical column with an IC SI-90G (10 mm × 4.6 mm I.D.) guard column, produced by Shodex. This column has a particle size of 5 μm and consists of an anion exchanger made from polyvinylalcohol gel chemically bound to quaternary ammonium groups.

Data acquisition, construction of calibration curves and peak integration were carried out with the aid of the Peaknet® Dionex software package, version 6.11.

2.3. Reagents

All reagents were of analytical grade. The dissolution of reagents and the dilutions were made with Milli-Q water. Standard solutions of acetate and formate were prepared by dilution of their stock solutions (each 1000 μg/mL, Merck, Darmstadt, Germany). Standards of fluoride were prepared by dilution of the stock solution (20 μg/mL, Dionex). The LODs correspond to 3σ confidence levels of blank solutions of acetate, formate and fluoride, which were prepared from unexposed cartridges of

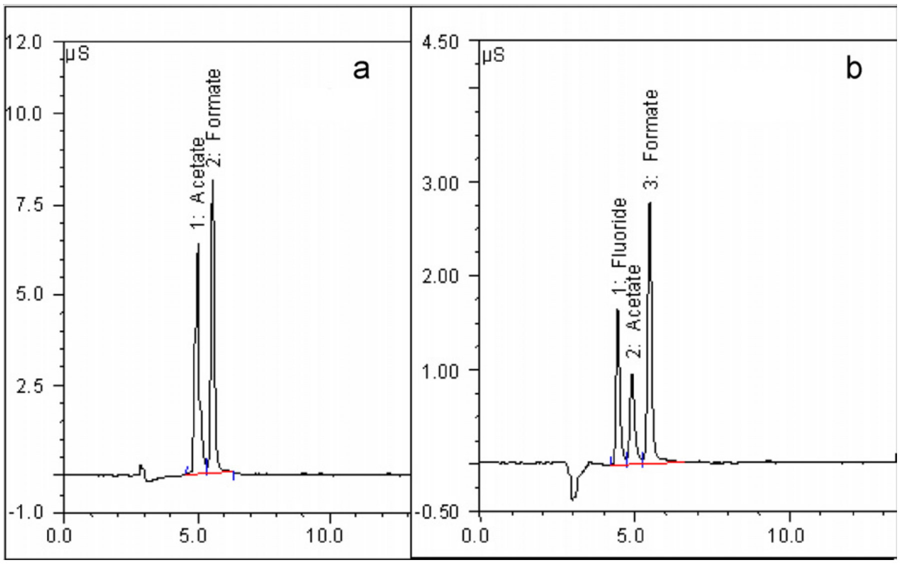

Fig. 1. Baseline separation of chromatographic peaks of fluoride, acetate and formate applied in a standard solution on an IC SI-50 4E column; (a) 200 mg/L acetate and 50 mg/L formate; (b) 1 mg/L fluoride, 10 mg/L acetate and 5 mg/L formate.

diffusive samplers using the same procedure as for the samples.

Eluents containing mixtures of Na_2CO_3 and $NaHCO_3$ at various concentrations were prepared by dissolving Na_2CO_3 (Merck) and $NaHCO_3$ (Merck) powders. Accurate weighing of chemicals was carried out on a Chyo Model JL-180 (Chyo Balance Corp., Kyoto, Japan) electronic analytical balance. The calibration curves were constructed with five standards. Each standard/sample was measured in triplicate and average concentrations and their standard deviations (SDs) were calculated.

2.4. Optimization of the separation procedure

For the optimization of the separation on the IC SI-50 4E column, a series of standards with varying acetate and formate content was prepared as follows (all in mg/L): 1 + 1, 10 + 5, 50 + 25, 100 + 50, 200 + 100, and 400 + 100, respectively. In order to get the best separation of the three ions, the following eluents were tested (all in mmol/L): (I) 0.8 $NaHCO_3$/2.0 Na_2CO_3; (II) 0.8 $NaHCO_3$/2.4 Na_2CO_3; (III) 1.0 $NaHCO_3$/2.8 Na_2CO_3; (IV) 1.0 $NaHCO_3$/3.2 Na_2CO_3; and (V) 1.2 $NaHCO_3$/3.6 Na_2CO_3. The eluent flow rate was studied in the range of 0.7–1.2 mL/min.

3. Results and discussion

3.1. Comparison of separation conditions on various columns

A nearly baseline separation of acetate and formate is attained on an IonPac AS14 column when using an eluent of 2.0 mmol/L $Na_2B_4O_7$ with a flow rate of 1.0 mL/min [16]. Under these conditions, retention times of 8.8 min for acetate and 9.4 min for formate are obtained. For the Allsep A-2 column, the optimal separation of acetate and formate is achieved with the use of a mixture of 1.5 mmol/L $NaHCO_3$ and 1.2 mmol/L Na_2CO_3 buffer solution as an eluent at a flow rate of 1.3 mL/min. Under these conditions, retention times of 4.1 min and 5.1 min are observed for acetate and formate, respectively [16].

In this study, a complete baseline separation of both anions is achieved using an IC SI-50 4E column and an eluent of 3.2 mmol/L Na_2CO_3 plus 1.0 mmol/L $NaHCO_3$. This eluent composition is also recommended by the manufacturer of the column. The optimal eluent flow rate on the separation of fluoride, acetate and formate is found to be 1.0 mL/min. Under these conditions, retention times of 5.0 min for acetate and 5.6 min for formate can be observed (Fig. 1a).

For the columns IonPac AS14 and IC SI-50 4E, the separation of the two chromatographic signals is achieved for concentrations not higher than 100 mg/L and 50 mg/L of acetate and formate, respectively. At higher concentrations, due to the broadening of peaks and their overlapping, the resolution is decreasing. In the case of the Allsep A-2 column, it is possible to obtain a good resolution of the peaks, even up to 500 mg/L and 100 mg/L of acetate and formate, respectively [16].

As mentioned before, poor reproducibility for fluoride and acetate has been reported in the literature [17,18]. With the use of Radiello diffusion tubes, HF is also sampled concurrently with acetic and formic acid. As it was found in a previous study, on the IonPac AS14 column the fluoride, acetate and formate peaks could be almost baseline separated [16]. However, the separation is not

Table 1

Comparison of the analytical performance of IonPac AS14, Allsep A-2 and IC SI-50 4E columns for fluoride, acetate and formate under optimized chromatographic conditions.

Parameter	IonPac AS14			Allsep A-2			IC SI-50 4E		
	Fluoride	Acetate	Formate	Fluoride	Acetate	Formate	Fluoride	Acetate	Formate
LOD in solutions (mg/L)	0.045	0.61	0.46	0.030	0.68	0.51	0.014	0.22	0.17
UL (mg/L)	100	200	100	100	300	100	100	500	100
RSD (%)	5.3	4.0	5.0	5.4	3.5	4.7	1.7	3.0	2.8
R^2	0.9992	0.9989	0.9982	0.9991	0.9993	0.9992	0.9996	0.9991	0.9997
Retention time (min)	8.2	8.8	9.4	3.7	4.1	5.1	4.4	5.0	5.6

Abbreviations: LOD, limit of detection; UL, upper limit of the linear range of calibration; RSD, relative standard deviation; *R*, correlation coefficient of the calibration curve (linear fitting).

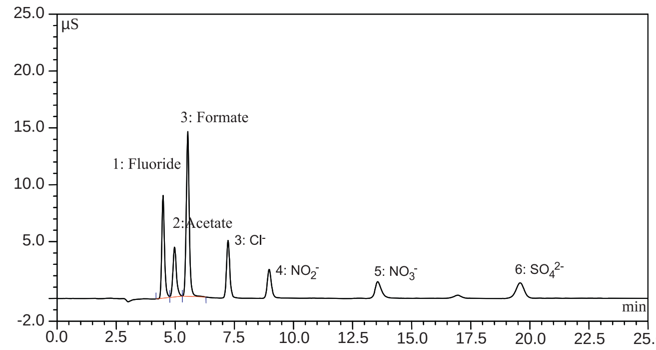

Fig. 2. Separation of fluoride, acetate and formate on an IC SI-50 4E column for a typical air sample taken at the Metropolitan Museum of Art in New York.

efficient on the Allsep A-2 column in terms of resolving the peaks of these anions, even under optimized conditions as above. Hence, the separation of fluoride from acetate and formate on the IC SI-50 4E column is investigated in details.

The peak resolution depends on the amount of analyte (anion) injected on an IC column. At higher anion concentrations, the peak may be broadened and thus possibly overlapping with a subsequent peak of another ion with close retention time. Therefore, a series of standard solutions at constant acetate and formate contents (10 and 5 mg/L, respectively) and with increasing fluoride content (1, 2, 5, 10, or 15 mg/L) was prepared and analyzed to study the effect of fluoride on the determination of acetate and formate. At each fluoride concentration, complete baseline separation of fluoride from acetate, as well as acetate from formate is achieved on this column (Fig. 1b). This means that the amount of fluoride present in samples does not have any influence on the peak of acetate. Consequently, all three chromatographic signals could be resolved well within a fairly short analysis time of around 6 min (Fig. 1b). Similar analysis time was also observed for the Allsep A-2 column, whereas for the IonPac AS14 the analysis time was much longer (around 10 min).

3.2. *Analytical performance*

The analytical performance data for all three columns are listed in Table 1. The best LODs for fluoride, acetate and formate are obtained on the IC SI-50 4E column: 0.014, 0.22 and 0.17 mg/L, respectively. The LOD values achieved for the IonPac AS14 and the Allsep A-2 are more than twice higher (i.e. worse), compared to those for the IC SI-50 4E column. The precision of the determination of formate, expressed as the relative standard deviation (RSD), is approximately two times better (2.8%) on the IC SI-50 4E than on the other two columns (around 5%). In the case of acetate, the RSDs are similar for all three columns ranging between 3 and 4%, although the lowest RSD is observed for the IC SI-50 4E column (Table 1). For fluoride, the RSD is also found to be much lower (1.7%) on the IC SI-50 4E than on the other two columns (around 5%). The LOD and RSD values are lower, or commensurable with the data reported in the IC literature [12,17,18].

The retention times on the Allsep A-2 and on the IC SI-50 4E are only half of the ones obtained on the IonPac AS14. The shorter retention times obtained for the former two columns are similar with those data reported in the literature [12]. However, a twice higher sensitivity of the determinations, expressed as conductivity, is noted when applying the IonPac AS14 column in comparison to the Allsep A-2 and the IC SI-50 4E. The upper limit of the linear calibration range is found to be the highest for the IC SI-50 4E column followed by the IonPac AS14 and the Allsep A-2 columns (Table 1). These analytical performance parameters are comparable for formate, but better for acetate than those data reported in the literature [12]. Based on the above results, the IC SI-50 4E column is selected for further studies.

3.3. *Application of the method to ambient air samples*

The concentrations of gaseous acetic and formic acid were investigated in the atmospheric air of the Metropolitan Museum of Art in New York and compared to the results obtained at several museums worldwide [3,7,24]. In the Metropolitan Museum of Art, a part of the samples were taken in the Luce Centre of the American Wing, where American Art is on display, including furniture, glass and metal objects. Another series of samples was taken in the Egyptian Wing, where art originating from ancient Egyptian dynasties is displayed. For comparison, samples were also taken outdoors, on the roof of the museum near the air inlets of the air-conditioner.

Based on the above results and taking into account the possible presence of fluoride in the atmospheric air, the samples taken in the Metropolitan Museum of Art were analyzed for their acetate and formate contents using the IC SI-50 4E column. Fig. 2 shows a baseline separation for all the investigated anions as well as for

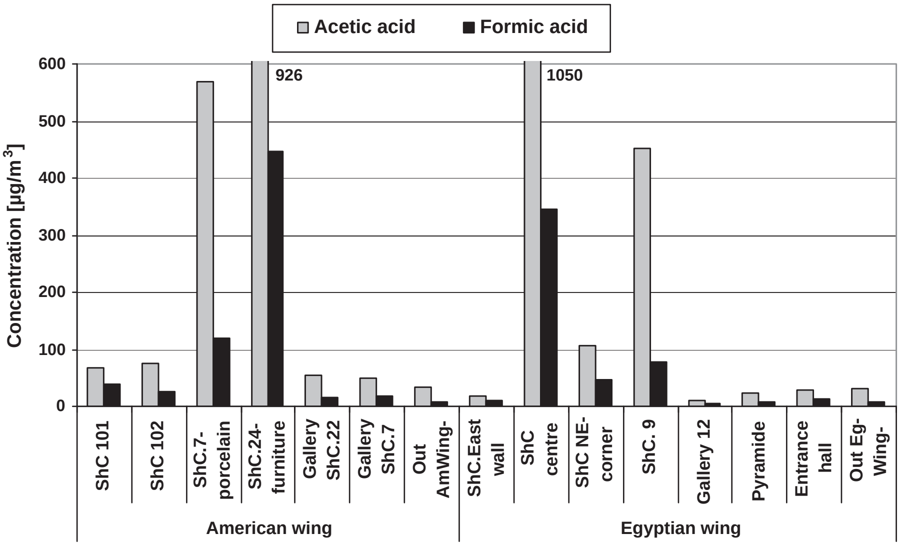

Fig. 3. Concentrations of acetic and formic acid in air samples taken at various locations in and outside of the Metropolitan Museum of Art (ShC – showcase).

Table 2
Concentrations of acetic and formic acid in showcases of European and American museums.

Sampling site	Concentration (μg/m^3)		Reference
	Acetic acid	Formic acid	
The Burrel Collection, Glasgow, UK	2723–11,384	273–335	[7]
Royal Museum of Scotland, Edinborough, UK	708–1768	648–1570	[7]
Santa Ana Museum, CA, USA	1371	373	[7]
Musical Instruments Museum, Brussels, Belgium	80–600	15–250	[3]
Plantin-Moretus Museum, Antwerp, Belgium	113–3215	16–438	[3]
Metropolitan Museum of Art, New York, USA	18–1050	16–450	This work

other anions, such as NO_2^-, NO_3^-, and SO_4^{2-} present in the air samples of the museum.

High organic acid levels were detected inside the non-aerated showcases, both in the Egyptian and the American Wing (Fig. 3). Values of up to 1050 μg/m^3 of acetate and 450 μg/m^3 of formate were found. These concentrations, however, are commensurable to those observed in showcases of European and American museums, displaying wood and/or wooden items [3,7]. For example, in the Burrel Collection in Glasgow, as well as in the Royal Museum of Scotland similar, or even more elevated levels of acetic and formic acid have been reported (Table 2). On the other hand, showcases without objects of art made of wood or wooden composites did not show any significant accumulation of these organic acids inside (e.g., showcases 101 and 102 in the American Wing, Fig. 3), as these showcases are made of metal and glass. In the galleries and in the outdoor air, rather low concentrations of organic acids were found, with average concentrations of 50 μg/m^3 of acetate and 10 μg/m^3 of formate.

4. Conclusions

In this work, the IC separation of fluoride, acetate and formate was studied and compared by the application of a novel (IC SI-50 4E) and two formerly introduced (IonPac AS14 and Allsep A-2) analytical columns. By applying these methods, it was possible to determine the level of organic acids in indoor air samples of buildings displaying works of art. Optimal conditions for the complete baseline separation of all of the three anions on the IC SI-50 4E were found to be as the following: an eluent containing a mixture of 3.2 mmol/L Na_2CO_3 and 1.0 mmol/L $NaHCO_3$ with a flow rate of 1.0 mL/min. It was concluded that good separation of acetate and formate could be achieved on all three columns. However, on the IC SI-50 4E, the best analytical performance was obtained, in terms of the LODs, RSDs and dynamic ranges. With the use of the optimized elution conditions for this column, baseline chromatographic separation of fluoride and acetate, as well as acetate and formate peaks was achieved within a fairly short analysis time.

With the application of the optimized method based on the use of the IC SI-50 4E column, the levels of acetic and formic acid could be more accurately and precisely quantified in the indoor air of museums, for instance, the Metropolitan Museum of Art, implying even the presence of a high fluoride content in their atmosphere. The results also show that acids can promote the accumulation of other dangerous compounds emitted by the showcases themselves or by the works of art. Hence, depending on which pollutants are especially dangerous for the displayed works of art, the storage in showcases can improve, but can also deteriorate the conditions for conservation. Analytical chemical techniques, such as the IC methodology elaborated in this study, can help in the understanding of common conservation problems originating from atmospheric pollution.

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