Determination of Nitrate and Sulfate Collected on Air Filters

PREFACE

Since the initial development of this Application Note in May 1978, there have been several updates in suggested IC conditions. These updates have improved the speed and resolution of IC. The latest recommended conditions from Dionex are listed below. See Figure 3.

Column:	IonPac® AS14 Analytical and AG14 Guard
Eluent:	3.5 mM Na_2CO_3/1.0 mM $NaHCO_3$
Flow Rate:	1.2 mL/min
Suppressor:	Anion Self-Regenerating Suppressor (ASRS), recycle mode
Detection:	Conductivity
Injection Volume:	50 µL

The remainder of this Note is similar to when it was originally published.

INTRODUCTION

The classical method of analysis for nitrate and sulfate is wet chemistry. This report describes a comparison between IC and wet chemistry methods for the determination of nitrate and sulfate collected on filters used in air pollution monitoring studies. The filters were provided by J. Mulik of the United States Environmental Protection Agency (U.S. EPA) in Research Triangle Park, North Carolina.

RECOMMENDED EQUIPMENT

Dionex DX-120 or
DX 500 Ion Chromatography System consisting of:
    GP40 Gradient Pump
    CD20 Conductivity Detector or
        ED40 Electrochemical Detector

Autosampler
PeakNet Chromatography Workstation

PROCEDURE

Four 20- x 1.5-cm strips of labeled filter paper were submitted for analysis. Each contained a known weight of nitrate and sulfate. Similar strips were analyzed by wet chemistry techniques for nitrate and sulfate.

Filter strips 6054 and 6052 contained 465 mg of sulfate and 1381 mg of nitrate determined by known addition. The following procedure was used to extract and analyze the strips for nitrate and sulfate: Immerse each strip in 50.0 mL of a buffer (or eluent) at 50 ± 3 °C for 7 minutes, agitating the solution during this period. (*Note*: More recently the eluent used has been 0.003 M $NaHCO_3$/ 0.0024 M Na_2CO_3 and the extractions were performed by letting the filter strips stand for l0 minutes or sonicating them for 10 minutes at room temperature without heat. Decant the buffer and inject directly into a Dionex Ion Chromatograph.

The strips were allowed to stay in contact with the buffer for an additional 4 hours at room temperature (covered to prevent evaporation) and then analyzed to ensure that all of the nitrate and sulfate had been extracted.
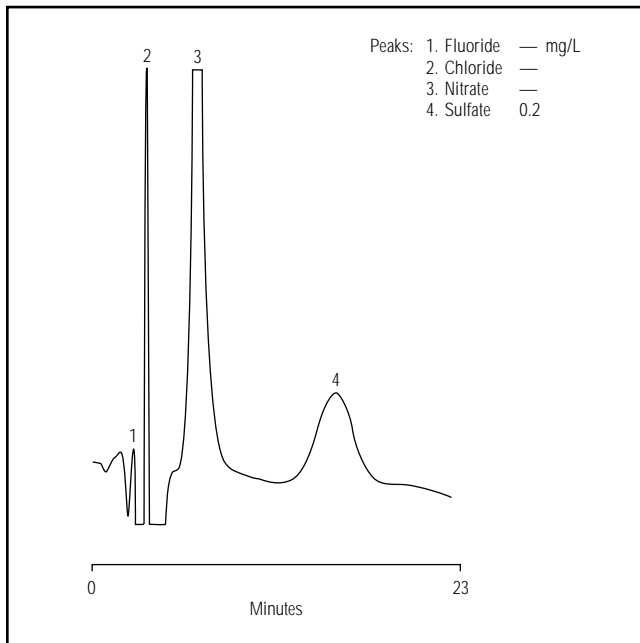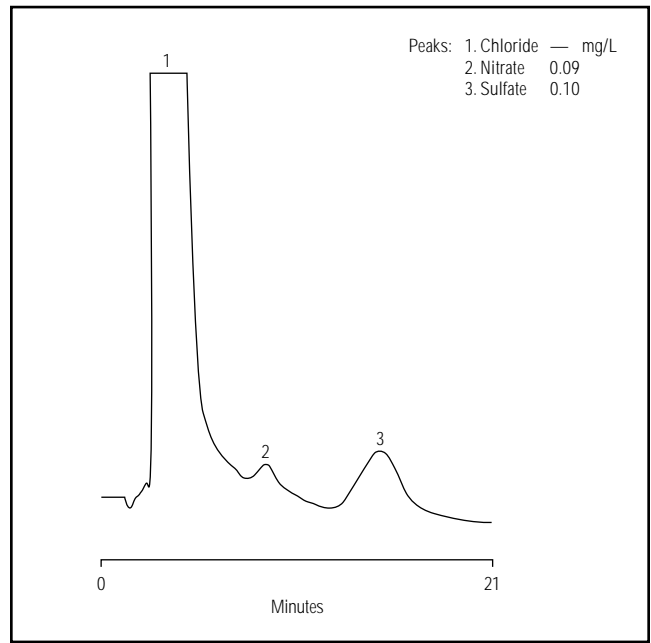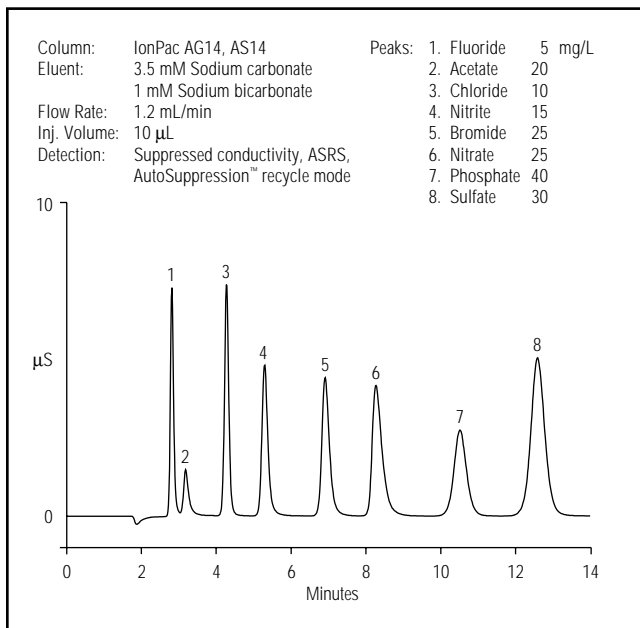
Figure 1   *EPA Standard*

Peaks: 1. Fluoride — mg/L
2. Chloride —
3. Nitrate —
4. Sulfate 0.2

Figure 2   *Sample 5163*

Peaks: 1. Chloride — mg/L
2. Nitrate 0.09
3. Sulfate 0.10

Column:	IonPac AG14, AS14	Peaks:	1. Fluoride	5	mg/L
Eluent:	3.5 mM Sodium carbonate		2. Acetate	20	
	1 mM Sodium bicarbonate		3. Chloride	10	
Flow Rate:	1.2 mL/min		4. Nitrite	15	
Inj. Volume:	10 μL		5. Bromide	25	
Detection:	Suppressed conductivity, ASRS,		6. Nitrate	25	
	AutoSuppression™ recycle mode		7. Phosphate	40	
			8. Sulfate	30	

Figure 3   *Anion standards chromatographed on the IonPac AS14 anion-exchange column*

CONDITIONS

Eluent:	1.8 mM Na_2CO_3/1.7 mM $NaHCO_3$
Flow Rate:	2.0 mL/min
Column:	IonPac AS1 (3 x 250 mm)
Detection:	Conductivity
Suppressor:	AMMS

Table 1 gives the results of the analysis for nitrate and sulfate. Notice that it would be straightforward to determine fluoride and chloride in addition to nitrate and sulfate.

The data clearly show that determination of sulfate by IC is more accurate than by wet chemistry methods. It is known that chloride ion interferes to give a positive variance in the barium chloranilate analysis[1]. Sample 6054 contains over 138 mg/L chloride, suggesting one source of error. The sulfate results for samples 5162 and 5163 indicate an even larger error for the wet chemistry method, as the concentration of sulfate is reduced. The nitrate determination by IC compares favorably with wet chemistry, particularly at the higher concentration. No increase in nitrate or sulfate levels was observed even after soaking the filters in buffer for four additional hours. This, along with the good overall experimental reproducibility, implies that the extraction is complete.

CONCLUSION

IC offers a convenient and more accurate method of sulfate analysis than wet chemical methods. Also, nitrate analysis by IC is comparable in accuracy and faster than wet chemistry methods (total analysis time was 20 minutes). Since fluoride, chloride, nitrate, sulfate, and other ions such as nitrite, phosphate, and acetate may be simultaneously analyzed, IC represents a powerful analytical tool for the rapid and accurate analysis of anions for air pollution.

REFERENCE

1. Personal communication, Jim Mulik, U.S. EPA.

Table 1 Analysis of Filter Extracts						
	Nitrate Concentration (mg/L)			Sulfate Concentration (mg/L)		
Samples	IC[1]	Wet Chemistry[2]	Known	IC[1]	Wet Chemistry[2]	Known
6052, 6054	25.4 ± 0.6[3]	27.5	27.6	9.25 ± 0.5[3]	13.6	9.3
5162, 5163	0.09 ± .005	0.45	—	0.1 ± .01	11.6	—

[1] Average of the average of each sample. Each individual analysis was repeated at least three times giving a relative standard deviation of 3% for 6054 and 6052. The RSD for 5162 and 5163 was not calculated but was higher due to poorer signal to noise. Only one analysis per sample was made in this case.

[2] Error analysis is unknown.

[3] No increase in concentration was observed after 4 hours of soaking the filter in buffer.