

## Anion Content in Charcoal Filters and in Water Samples Stored in Various Containers

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The anion content of natural water is related to the precursors of acid precipitation, mainly sulfur and nitrogen oxides (Tan et al. 1986). While cation contamination of environmental water samples due to supplies used in analytical procedures is well established (Robertson 1972, Hennig and Greenwood 1981), less is known about anion contamination. It was shown that anions (e.g. F, Cl, Br NO<sub>3</sub>, SO<sub>4</sub>, PO<sub>4</sub>) were introduced by specific filters or leached through membrane-type filters which are extensively used in water analyses (Butler et al. 1978, Jay 1985). Anion contamination of charcoal-filtered distilled-deionized water was evaluated, and anion content was measured following the storage of samples of standard anions.

## MATERIALS AND METHODS

Nanopure cartridge (series D0835, Sybron-Barnstead) was used for the washing study. Repeated washings were made by using 250-mL aliquots of eluent (0.0015 M NaHCO $_3$  and 0.0012 M Na $_2$ CO $_3$ ) on 10 g portions of Nanopure charcoal. The resultant leachates were analyzed for F , Cl , NO $_3$  , and SO $_4$  .

In the sample storage study, standards were prepared by diluting mixed stock solutions containing 998 mg L  $^{-1}$  F , 1019 mg L  $^{-1}$  Cl , 988 mg L  $^{-1}$  NO  $_3$  , and 1010 mg L  $^{-1}$  SO  $_4$  using serial dilution. Portions of each of these 4 mixtures (2 to 3 order dilution) were stored under the following conditions: a) 250 mL-Nalgene bottle at 6 C, b) 250-mL Nalgene bottle at 20 C, c) 250-mL glass bottle at 6 C, and d) 250 mL glass bottle at 20 C. The anion concentrations were measured after 1, 7, and 14 days by a Dionex 2110i ion chromatograph (IC) with a conductivity detector and a Spectra Physics SP 4100 integrator. A 4 x 50 mm anion precolumn (Dionex P/N 30986) and a 4 x 50 mm anion analytical column (Dionex P/N 30985) were operated in series. A suppressor column (9 x 100 mm) was also employed. The flowrate, injection volume, and pressure were 2 mL min  $^{-1}$ , 100  $\mu$ L, and 900 psi. Typical analyses of anion represented the average taken from 3 replicate chromatograms.

A standard mixture of the anions was digested by the Schoninger

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flask combustion technique. Subsequent anion contents were determined by standard wet titrimetric methods (ASTM 1976) and compared to those obtained by IC (Colaruotolo 1978). The relative % difference between methods was 1.8% for F , 2.3% for Cl , and 0.7% for  $\mathrm{SO}_{L}^{2}$ .

## RESULTS AND DISCUSSION

Figure 1 shows the effect of repeated washings of charcoal on anion content. The level of anions dropped below 0.4 mg L  $^{-}$  after 7 or 8 250-mL washings. Charcoal filters are commonly used to adsorb humic and fulvic acids from environmental samples (Marko-Varga 1984), and these filters are employed without con-sideration of anion contamination. This was particularly important in  $\rm F$  and NO  $_{3}$  analyses. For example, the contamination levels (first 3 washings ) of  $\rm F$  were 60% of precipitation (rain) and reservoir water, and those of NO  $_{3}$  were 20% of precipitation and 250% of reservoir water (Tan et  $^{3}$  al. 1986).

Table 1 shows the retention of anions in sample storage. There was no difference between storage of samples at 6 C and 20 C, and between storage in glass and Nalgene bottles. In addition, measurement of these mixed anions in solution did not change after 1, 7, and 14 days. These results are combined and summarized under each of the 4 concentration levels. Heydorn and Damsgaard (1982) reported that no significant adsorption or leaching took place with most aqueous solutions when polyethylene was used. Except for F, anion contents were not changed when stored at 4 C in high density polyethylene bottles for approximately 7 months (Jay and Judd 1985).

The loss in concentration was more apparent at the lower concentration levels. At concentration levels 0.5 to 1.0 mg L $^-$ , the relative standard deviations (RSD's) were 2.1 to 11% for these 4 anions. It was suggested that recalibration of these anions (0.5 to 10 mg L $^-$  range) improved the analytical precision (Jay and Judd 1985). At levels of 5 to 10 mg L $^-$ , the analytical precision was acceptable ( $\leq$  2.1% RSD) with sample storage, except for Cl $^-$ . The slopes for these anions were calculated, and the correlation coefficients were better than 0.9996. At the 95% confidence intervals of the slopes, all slopes calculated were within the gradient of 1 (Table 1), sug-gesting that anion adsorption-saturation was established below the 0.5 mg L $^-$  level.

In the study of anions derived from environmental samples, it is important to wash commercially available charcoal filters, and to recalibrate samples if the anion levels are expected to be below  $1.0~{\rm mg~L}^{-1}$ .

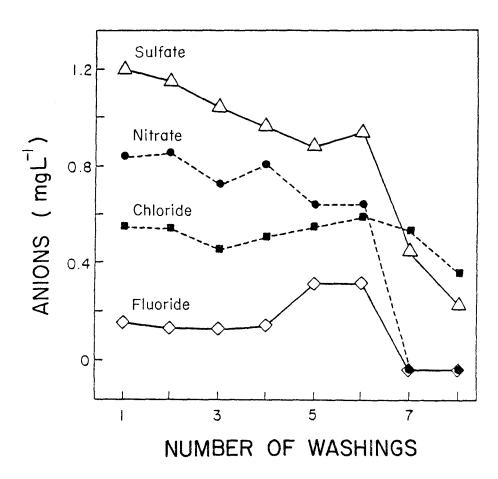


Figure 1. Effect of repeated washings (250-mL aliquots) of Nanopure charcoal (10 g) on anion content.

Table 1. Anion Retention in Sample Storage

Concentration (mg L )	F	C1	NO <sub>3</sub>	so <sub>4</sub> <sup>2</sup>
~0.5 ~1.0 ~5.0 ~10.0	0.49 (6.1) 0.95 (4.2) 4.95 (0.81) 9.94 (0.60)	0.53 (3.8) 0.97 (3.1) 4.8 (4.2) 10.1 (0.99)	0.49 (6.1) 0.97 (2.1) 4.94 (0.61) 10.1 (1.0)	0.56 (11) 1.04 (4.8) 4.9 (2.1) 10.0 (2.0)
slope	0.997±0.061	0.988±0.065	1.024±0.037	0.983±0.034

Results were presented in mg  $L^{-1}$  ±%RSD; n=12. 95% confidence intervals of slope.

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