

MILLENNIUM APPLICATION

BRIEF

TOPIC: Why Cleanliness is Important in Low Level (ppb) Anion Analysis

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Introduction:

Analysis of low level anionic impurities by CIA or IC is challenging due to the possibilities of contamination. Contamination can come from many sources such as hands, lab air, and labware used for sample or standard preparation. With a few precautions low level anion analysis by CIA or IC can be easily achieved. Some general precautions include the following:

- 1: Wear, non-talc, gloves when preparing or handing samples and standards.
- 2: Use only plastic volumetric flask and plastic sample vials.
- 3: Be sure to clean all vials and labware by soaking them in high purity water. (at least overnight)
- 4: If possible, devote all volumetric flask to low level analysis (this prevents any cross contamination from other analysis)
- 5: Do not leave samples or standards open and sitting exposed to the lab environment (keep the lids on them or the autosampler cover on them)
- 6: Do not clean the labware with detergents. (rinse, clean and soak in high purity water only)

Some examples of the effects of contamination are shown in the following figures. Figure 1 is an electropherogram of a water blank run using the procedures listed above (top) and the sample if gloves were not worn and the sample is touch, inadvertently, while loading the vials or transferring to vials. (middle) As can be seen a high level of chloride and sulfate has been introduced into the sample by accidental contact. A 14 ppb standard is shown in the bottom for comparison purposes. Approximate contamination of the sample was 1 ppm of chloride.

Figure 2 is an electropherogram demonstrating the effects of exposure to lab air has on samples. The top electropherogram is of a water blank run immediately and the same water blank after sitting out exposed to the lab air (uncovered on the bench) for approximately 30 minutes. A standard at 2 ppb is shown in the bottom. Once again fairly high (ppb) levels of chloride, sulfate and nitrate have been introduced by exposure to the lab air. This contamination will vary greatly depending on the location of the lab (i.e. urban versus rural or suburban) as well as the air ventilation system used in the lab. Samples that were covered did not suffer these effects. The take home message from this is that by following a few simple precautions low level anion analysis is possible.

EXPERIMENTAL:

Anion Analysis

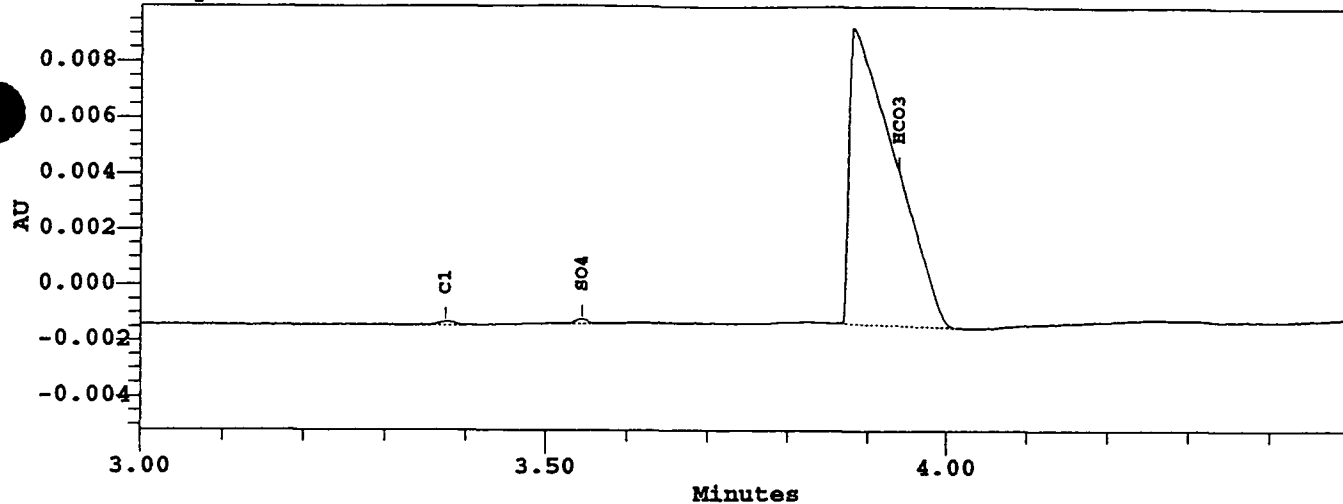
System:	Q4000E
Electrolyte:	Modified Chromate/OFM pH=11.2
Capillary:	75um X 60cm
Injection:	Electromigration -5kV, 45 seconds.
Run Voltage:	-15kV
Detection:	Indirect UV at 254nm
Data:	Millennium 2010 Chromatography Manager, Ver. 2.1 with CIA Option

FIGURES:

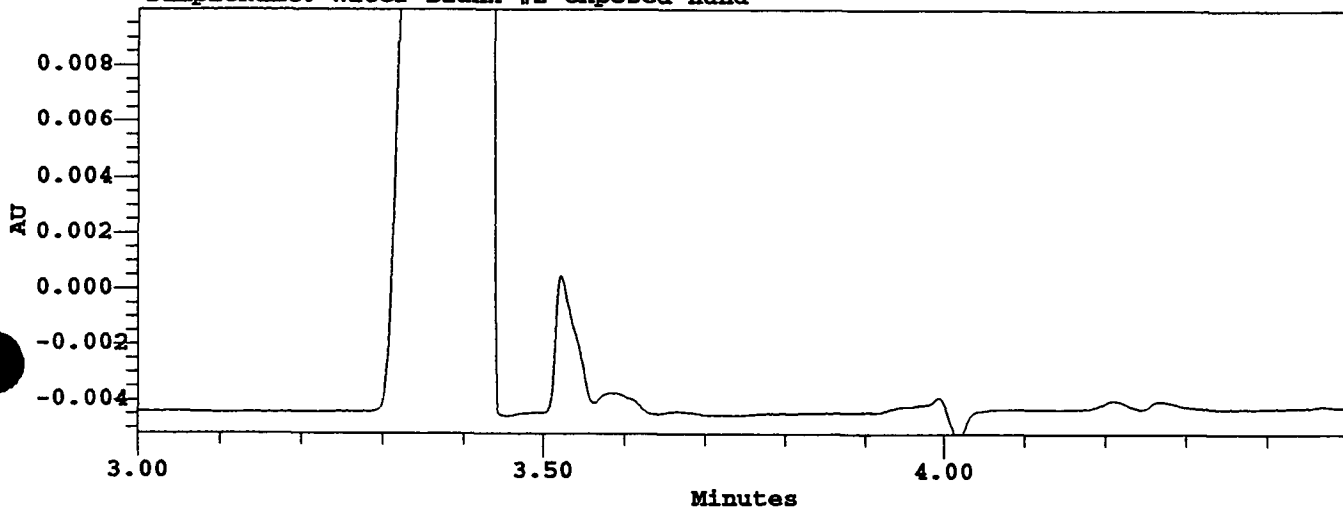
Figure 1: Electropherogram of a water blank run properly (top) and exposed to an ungloved hand (middle) as compare to a ppb level standard

Figure 2: Electropherogram of water blank run as is (top) and after exposed (not covered) to lab air for 30 minutes (middle) as compared to a low level (ppb) standard.

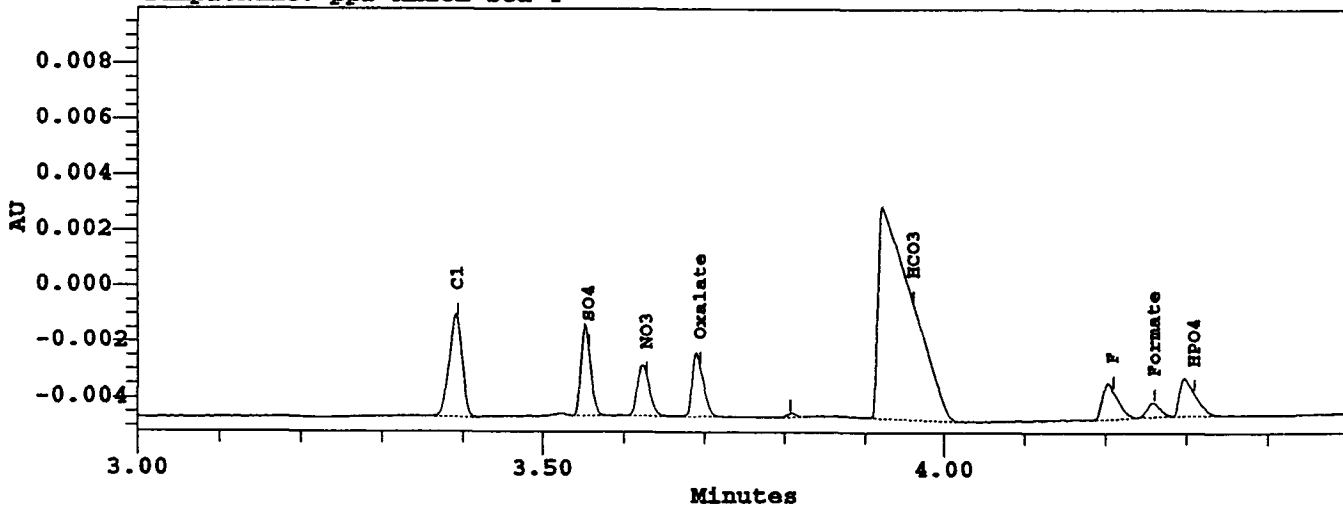
SampleName: Water Blank



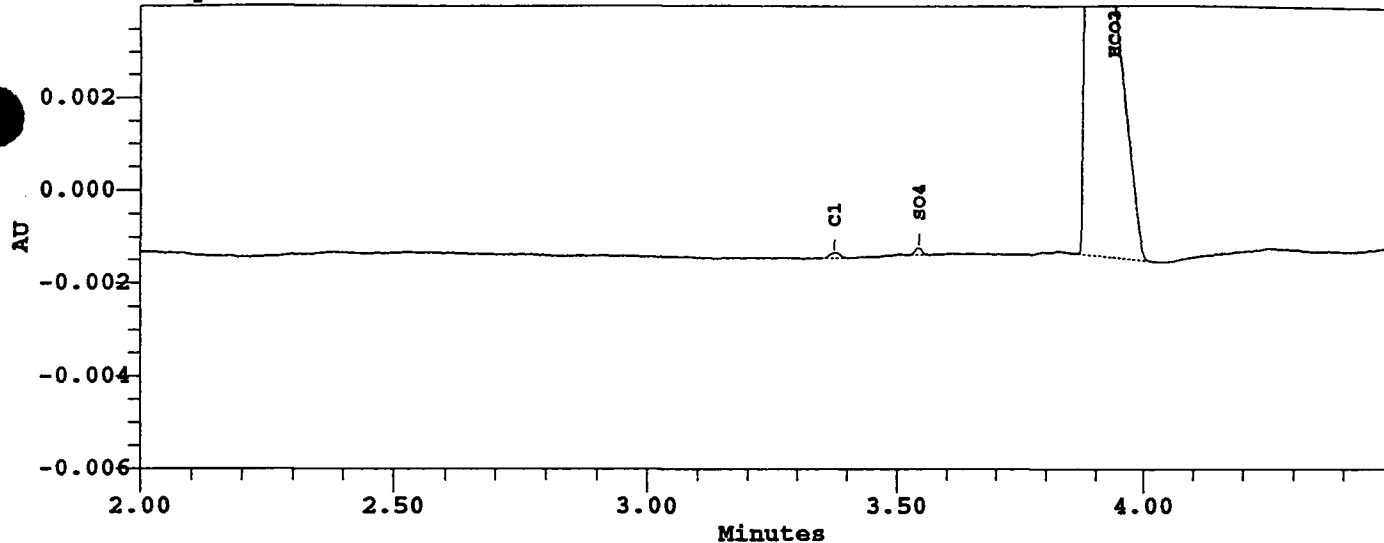
SampleName: Water Blank #2-exposed hand



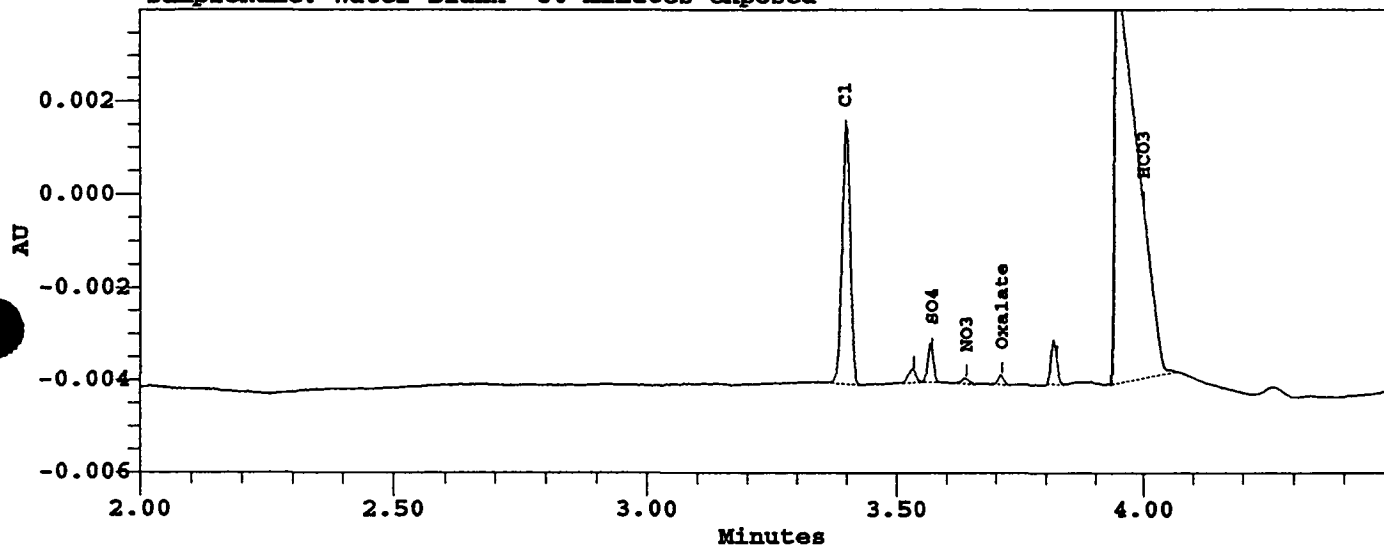
SampleName: ppb Anion Std 4



SampleName: Water Blank



SampleName: Water Blank- 30 minutes exposed



SampleName: ppb Anion Std1

