



<b>ATF-LS-E14</b> <b>Ion Chromatography - Cation Analysis</b>	Published Online: <b>March 2018</b>
Authority: Technical Leader	
Unofficial Copy; May Not Be Most Current Version	Page: 1 of 4

## I. Scope:

This procedure is applicable for determination of inorganic and organic cations commonly found in aqueous extracts of explosives and explosive residues. Ion Chromatography follows basic liquid chromatography principles using an ion exchange column. The unique resin inside the column allows for ionic species within an aqueous sample to be separated. These cations are then detected using electrically suppressed conductivity.

## II. References:

Bender, E.C. (1989). Indirect photometric detection of anions for the analysis of low explosives, *Crime Lab. Digest*, 16, 78-83.

Bender, E.C., Crump, J., Midkiff, C.R. (1993). The instrumental analysis of intact and post blast water gel and emulsion explosives, *Advances in Analysis and Detection of Explosives*, 179-188.

Hargadon, K.A. and McCord, B.R. (1992). Explosive residue analysis by capillary electrophoresis and ion chromatography, *J. Chromatogr.*, 602, 149-154.

Kolla, P. (1991). Trace analysis of salt based explosives by ion chromatography, *Forensic Science Int.*, 50, 217-226.

McCord, B.R., Hargadon, K. A., Hall, K.E., Burmeister, S.G. (1994). Forensic Analysis of Explosives using Ion Chromatographic Methods, *Analytica Chimica Acta*, 288, 43-56.

Reutter, D.J., Buechele, R.C., and Rudolph, T.L. (1983). Ion chromatography in bombing investigations, *Anal. Chem.*, 55, 1468A – 1472A.

Reutter, D.J., and Buechele, R.C. (1983) Ion Chromatography of Explosives and Explosive Residues, *Proc. 1st Int. Symp. on Analysis and Detection of Explosives*, FBI Academy, Quantico, VA, pp. 199-207

Rudolph, T.L. (1983). The characterization of some low explosive residues by ion chromatography, *Proc. 1st Int. Symp. on Analysis and Detection of Explosives*, FBI Academy, Quantico, VA, pp. 213-219.

Small, H., *Ion Chromatography*, Plenum Press, New York, 1989

Small, H., Stevens, T.S., and Bauman, W.C. (1975). Novel ion exchange chromatographic method using conductimetric detection, *Anal. Chem.*, 47, 1801-1809.

Small, H. (1983). Modern inorganic chromatography, *Anal. Chem.*, 55, 235A – 242A.

### **III. Apparatus/Reagents:** Basic System Components (or equivalent)

Dionex ICS3000 Ion Chromatography system. See instrument parameter sheet for specifics on cation IC analysis;

See IC/MS protocol for information on anion IC analysis

All reagents will be HPLC grade or better.

Disposable plasticware must be used whenever possible.

All standards will be made using 18 MegaOhm deionized water. Cations - At a minimum, the standard will contain the following: sodium, ammonium, potassium, magnesium, calcium, strontium and barium.

### Sampling and Sample Prep

18 MegaOhm deionized water will be used for all extractions. All extracts and control samples will be filtered before injection into the system using disposable syringe filters rated for IC analysis. Plastic sample vials should be used whenever possible.

### **IV. Safety Precautions:**

Handling of eluents and eluent constituents require the standard proper protections against exposure to strong acids and bases. Additionally, other chemicals may pose health or environmental risks. The samples are primarily aqueous and may pose a chemical threat due to the possible ionic components of the extracts (e.g. cyanates). Additionally, bodily fluids and/or biological organisms may be present and vital in samples from certain post blast cases. Care must be exercised in preventing exposure to skin, eyes, or ingestion.

### **V. Procedures:**

1. Sample extracts will be prepared using 18-megohm deionized water. They should be filtered, and diluted to an appropriate level as determined by a conductivity meter.
2. Each sample set will have a standard run at the beginning (after the initial water blank) and end of the sequence. Longer sequences may require additional standards. The standard component peaks should show peak height, shape & resolution comparable to the previously run standard. The primary operator will be notified if there is a significant change in any of these criteria. Retention time windows should be no more than 5% of the retention time for each species. If any standard component peaks fall outside the retention time windows, these windows will be reset. Windows may also be adjusted to reflect

- smaller changes in retention times.
3. Each sample set should include a materials control, which is representative of all the procedures used in preparing the sample. Additionally, water blanks should be run immediately before a standard, materials control, or sample extract (with the exception of the sample following a clean materials control).
  4. Sample chromatograms should show baseline resolution between the components of interest. In addition, the peak shapes and retention times of the resultant peaks should be comparable to the standard chromatogram.

Non-routine exam – allowance for variation: Changes to any of the IC methods may be necessary for unique examinations of ionic species that may not be normally encountered yet need to be examined for any given case. Columns, eluents, and instrumental parameters may be changed to perform a unique analysis. These changes and procedures must follow sound scientific procedures and be fully documented.

#### **VI. Quality Assurance/Quality Control:**

Possible sources of error: interfering/co-eluting ions, matrix effects, temperature effects

Logs must be maintained that record:

- operator use
- standards
- service/maintenance

Any in-lab service, such as changing columns, or other maintenance, must be recorded and logged.

Primary Operators should perform maintenance to correct any of the following:

- Loss of resolution (should be 1.5 or above)
- Loss of retention
- Loss of detector response
- Abnormal peak shapes
- Abnormal operating pressures

#### **Glossary**

Auto regenerator - supplies the counter ions for chemical suppression

Capacity - the maximum concentration of sample that can be loaded onto a column before overloading.

Chemical suppression - used to reduce the eluent background to near zero while simultaneously converting the sample components to their acid or hydroxide forms which have higher conductances than the salt forms of ions to reduce the current

Chromatography - the separation of components in a mixture by partitioning ions between a mobile phase and a stationary phase.

Degassing - removing CO<sub>2</sub> which interferes with analytes on the column by using an inert gas.

Efficiency – a measure of peak dispersion as components move through a column.

Eluent - the fluid that is pumped through the column as the mobile phase

Gradient – the composition of the mobile phase changes during the analysis

Guard column - small version of separator columns which should be the same as a separator column so that any contaminant will be retained on the guard column.

HPLC: High performance liquid chromatography. Refers to an externally pressurized column as opposed to gravitational or osmotic modes.

Ion: Negatively (anion) or positively (cations) charged chemical species.

Isocratic - using the same mobile phase throughout

Resolution - the measure of separation between two peaks.

$R = \frac{T_2 - T_1}{[(W_1 + W_2)/2]}$ : where T is the retention time and W is the width of the peak at the base.

Selectivity - a measure of the affinity of a sample ion for the stationary phase

18 MegOhm deionized water: Water that has been treated to remove conductive ions whereas the resistance of the product is 18 megaohms or more; also known as “ultrapure water”.