

## Performance Monitoring Protocol (QA/QC) for the Ion Chromatography (IC) System

### 1 Scope

This document addresses the performance monitoring (QA/QC) of the Ion Chromatography (IC) system. This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Explosives (chemistry) and Chemistry Unit general physical and chemical analysis.

### 2 Principle

The IC system is a high performance liquid chromatography (HPLC) pump and a conductivity detector. The instrument can be configured to analyze anions or cations. When an ion elutes from the column and passes the detector, it produces a change in conductivity which is recorded as a peak in the chromatogram.

This performance monitoring protocol is based upon the manufacturer's recommendations. The definitions and guidelines for following this protocol are outlined in the "General Instrument Maintenance Protocol."

### 3 Equipment/Materials/Reagents

- a. Instrumentation - Dionex ICS-2000, ICS-2100, or Dionex Integriion HPLC pump and suppressed conductivity detector, Dionex AS or AS-AP programmable autosampler, and Chromeleon Software (or equivalent)
- b. Instrumentation - Waters e2695 Separations module, Waters 432 Conductivity Detector, and Empower Software (or equivalent)
- c. Columns:
  - IonPac CS12A Analytical Column (Dionex or equivalent)
  - IC-Pak C M/D Analytical Column (Waters or equivalent)
  - IonPac AS19 Analytical Column (Dionex or equivalent)
  - IonPac AS22 Analytical Column (Dionex or equivalent)
  - IonPac AG19 Guard Column (Dionex or equivalent)
  - IonPac AG22 Guard Column (Dionex or equivalent)
  - IonPac CG12A Guard Column (Dionex or equivalent)
- d. Nitric Acid (HNO<sub>3</sub>) (Reagent Grade)

- e. Ethylenediaminetetraacetic Acid (EDTA) (Reagent Grade)
- f. Thermo Dionex EGC III or EGC 500 KOH RFIC Eluent Generator (Potassium Hydroxide), or equivalent
- g. Thermo Dionex EGC III or EGC 500 MSA RFIC Eluent Generator (Methanesulfonic Acid), or equivalent
- h. Thermo Dionex EGC 500 K<sub>2</sub>CO<sub>3</sub> Eluent Generator (Potassium Carbonate), or equivalent
- i. Deionized Water, 18.2 MΩ·cm Milli-Q or equivalent
- j. Sodium, Ammonium, Potassium, Magnesium, Calcium, Chloride, Nitrite, Chlorate, Nitrate, Sulfate, Thiocyanate, Thiosulfate, Oxalate and Perchlorate Standards for IC (1000 ppm) (Fluka or equivalent)
- k. Calcium threonate (>98% purity) (Fluka or equivalent)
- l. Syringe - 250 μL (Dionex or equivalent)

## 4 Standards and Controls

### 4.1 Anions Testmix

The Testmix is used to assess daily operating performance and continued integrity of the system.

To prepare:

Pipette 5 mL of each liquid component of the anion standard for IC (chloride, nitrite, chlorate, nitrate, sulfate, oxalate, thiosulfate, thiocyanate, and perchlorate) and 5mg of each solid component (calcium threonate) into a 250 mL volumetric flask. Dilute to volume with deionized 18.2 MΩ·cm water. Shelf life is two years when stored refrigerated in a plastic bottle. This preparation may be appropriately scaled.

### 4.2 Cations Testmix

The Testmix is used to assess daily operating performance and continued integrity of the system.

To prepare:

Pipette 5 mL of each cation standard for IC (ammonium, potassium, sodium, calcium and magnesium) into a 250 mL volumetric flask, and dilute to volume with deionized 18.2

MΩ·cm water. Shelf life is two years when stored refrigerated in a plastic bottle. This preparation may be appropriately scaled.

## 5 Sampling

Not applicable.

## 6 Procedures

### 6.1 Daily Checks

The following steps will be performed daily. Enter the appropriate information in the QA/QC log for tracking purposes.

- a. Record the remaining disk space on the hard drive. Use Windows to verify that the hard disk has at least 100 MB of free space. Do not use if less than 100 MB remain.
- b. Check the level of deionized water in the reservoir and make sure there is sufficient volume to complete the sequence.
- c. Check the level of the waste container. Empty if necessary.
- d. For the Waters Cations system, set an appropriate base and sensitivity range.
- e. Perform an analysis of the appropriate Testmix. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable, print the chromatogram.
- f. If all requirements are within specification, prepare the documentation as outlined in the "General Instrument Maintenance Protocol." If any requirements fail, contact the appropriate instrument support personnel.

### 6.2 As Needed Checks and Maintenance

- a. Fill the needle wash reservoir (if applicable).
- b. Replace the Eluent Generator.
- c. Replace the guard column.
- d. Replace the analytical column.

## 7 Instrumental Conditions

### 7.1 Dionex Cations

Mobile Phase: Methanesulfonic acid (20mM), supplied from Eluent Generator  
Pump Mode: Isocratic  
Flow Rate: 1.0 mL/min  
Column: Dionex IonPac CS12A 4x250mm with IonPac CG12A Guard  
4x50mm  
Column Temperature: 30°C  
Inj Volume: 25 µL  
Acquire Time: 15 minute minimum

### 7.2 Waters Cations

Mobile Phase: 3.0 mM Nitric Acid (HNO<sub>3</sub>) / 0.1 mM EDTA  
Pump Mode: Isocratic  
Flow Rate: 1.0 mL/min  
Column: Waters IC-Pak Cation M/D 3.9x150mm  
Column Temperature: Ambient  
Inj Volume: 10 µL  
Acquire Time: 15 minute minimum

### 7.3 Dionex Anions (Potassium Hydroxide Method)

Mobile Phase: Potassium Hydroxide (gradient 20-80 mM)  
Pump Mode: Multi-step gradient (20 mM at 0 min, 20 mM at 2 min, 30 mM at 9 min, 80 mM at 13 min, 80 mM at 21min, 20 mM at 21.1 min, 20 mM at 25 min) ), supplied from Eluent Generator  
Flow Rate: 1.0 mL/min  
Column: Dionex IonPac AS19 4x250mm with IonPac AG19 Guard  
4x50mm  
Column Temperature: 30°C  
Cell Temperature: 35°C  
Inj Volume: 25 µL  
Acquire Time: 25 minute minimum  
Suppressor: Non-carbonate specific suppressed conductivity

### 7.4 Dionex Anions (Potassium Carbonate Method)

Mobile Phase: Potassium Carbonate (10mM), supplied from Eluent Generator  
Pump Mode: Isocratic  
Flow Rate: 1.5 mL/min

Column:	Dionex IonPac AS22 4x250mm with IonPac AG22 Guard 4x50mm
Column Temperature:	35°C
Cell Temperature:	35°C
Inj Volume:	25 µL
Acquire Time:	16 minute minimum
Suppressor:	Non-carbonate specific suppressed conductivity

## 8 Decision Criteria

Verify the results of the Testmix.

- a. In order for the instrument to be considered in good operating condition, all components from the appropriate Testmix should generate well-resolved, Gaussian-shaped peaks with baseline separation.
- b. A SNR of 3:1 will be the minimum response necessary to consider a response a peak.
- c. The retention times of the appropriate Testmix components should not deviate by  $\pm 5\%$  compared to the previous run of the appropriate Testmix.

## 9 Calculations

Not applicable.

## 10 Measurement Uncertainty

Not applicable.

## 11 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of this instrument.

## 12 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal

protective equipment should be used when handling any chemical and when performing any type of analysis.

### **13 References**

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual*.

"Liquid Chromatograph General Maintenance Protocol" (Inst 003) *Instrument Operation and Systems Support SOP Manual*.

*FBI Laboratory Safety Manual*.

Rev. #	Issue Date	History
0	04/25/16	New document, previously existed in the Explosives Unit.
1	04/29/16	Added 'or equivalent' to section 3 for the eluent generators. Section 6 removed the detector settings for the Dionex system.
2	10/04/18	Updated Section 1 Scope to include applicable disciplines/categories of testing. Added calcium and magnesium to Sections 3 j and 4.2. Updated heading in Section 5. Added 'appropriate instrument personnel' to Section 6.1 f. Changed from 10 min to 15 min in Sections 7.1 and 7.2. Updated 'Instrument Operation and Systems Support' in Section 13 and header.
3	07/15/20	Updated wording in Section 2. Added Dionex Integrion and AS-AP Autosampler to section 3a. Added thiosulfate and oxalate to Sections 3j and 4.1. Added calcium threonate to Sections 3k and 4.1. Added EGC 500 to Sections 3f and 3g. Updated heading in Sections 5 and 6.2. Removed "Explorer program" from Section 6.1a. Added Suppressor information to Sections 7.3 and 7.4. Added unit name to the title of each approver.

**Approval**

Redacted - Signatures on File

Explosives Unit-Chemistry  
Technical Leader

Date: 07/14/2020

Explosives Unit Chief

Date: 07/14/2020

Research & Support Unit-  
IOSS Manager

Date: 07/14/2020

Chemistry Unit-General  
Chemistry Technical Leader

Date: 07/14/2020

Chemistry Unit Chief

Date: 07/14/2020