Ion Chromatography (IC) System Performance Monitoring and Maintenance

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1 INTRODUCTION

This document addresses the performance monitoring and maintenance of the Ion Chromatography (IC) system. 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. The definitions and guidelines are outlined in IOSS-701.

2 SCOPE

This document applies to personnel using the associated instrument(s)/equipment in the following subdisciplines: Explosives Chemistry and General Chemistry.

3 EQUIPMENT

- Instrumentation
  - Thermo Dionex ICS-2100 or Integrion HPLC pump and suppressed conductivity detector, Dionex AS-AP programmable autosampler, and Chromeleon Software (or equivalent)
  - Waters e2695 Separations module, Waters 432 Conductivity Detector, and Empower Software (or equivalent)
- Materials
  - Columns:
    - IonPac CS12A Analytical Column (Dionex or equivalent)
    - IonPac CG12A Guard Column (Dionex or equivalent)
    - IonPac AS19 Analytical Column (Dionex or equivalent)
    - IonPac AG19 Guard Column (Dionex or equivalent)
    - IonPac AS22 Analytical Column (Dionex or equivalent)
    - IonPac AG22 Guard Column (Dionex or equivalent)
    - IC-Pak C M/D Analytical Column (Waters or equivalent)
  - Eluent Generators:
    - Thermo Dionex EGC III or EGC 500 KOH RFIC Potassium Hydroxide Eluent Generator (or equivalent)
    - Thermo Dionex EGC III or EGC 500 MSA RFIC Methanesulfonic Acid Eluent Generator (or equivalent)
    - Thermo Dionex EGC 500 K_2CO_3 Potassium Carbonate Eluent Generator (or equivalent)
  - Suppressors:
    - Cation Dynamically Regenerated Suppressor (CDRS)
    - Anion Dynamically Regenerated Suppressor (ADRS)
  - Nitric Acid (HNO_3) (Reagent Grade or equivalent)
  - Ethylenediaminetetraacetic Acid (EDTA) (Reagent Grade or equivalent)
  - Deionized Water, 18.2 MΩ·cm (Milli-Q or equivalent)

Redacted

- 250 µL syringe (Dionex or equivalent)
- General laboratory supplies

4 STANDARDS AND CONTROLS

- Anions Testmix

  The Anions Testmix is used to assess daily operating performance and continued integrity of an anions IC system. To prepare a 20 ppm solution, pipette 5 mL of each liquid component of the anion standard for IC Redacted a 250 mL volumetric flask and dilute to volume with 18.2 MΩ·cm deionized water. Shelf life is two years when stored refrigerated in a plastic bottle. This preparation may be appropriately scaled. Other concentrations may be prepared depending on instrument response.
  - If needed, a 1000 ppm stock solution can be prepared from solids by adding 100 mg of solid material to a 100-mL volumetric flask and diluting to volume with 18.2 MΩ·cm deionized water.

- Cations Testmix

  The Cations Testmix is used to assess daily operating performance and continued integrity of a cations IC system. To prepare a 20 ppm solution, pipette 5 mL of each cation standard for IC Redacted into a 250 mL volumetric flask and dilute to volume with 18.2 MΩ·cm deionized water. Shelf life is two years when stored refrigerated in a plastic bottle. This preparation may be appropriately scaled.

5 PROCEDURE

5.1 Daily Checks

The following steps will be performed daily. Enter the appropriate information in the instrument log.

A. Record the remaining disk space on the hard drive. 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, if applicable.

E. Perform an analysis of the appropriate testmix. Evaluate the results using the 'Acceptance Criteria' section. If the results are acceptable, print the chromatogram.
F. If all requirements are within specification, prepare the documentation as outlined in IOSS-701. If any requirements fail, contact the appropriate instrument support personnel.

5.2 As Needed Checks

The following steps will be performed as needed based on system performance. Indicate completion in the appropriate log. Refer to IOSS-701 for more information on instrument maintenance and documentation.

A. Fill the needle wash reservoir (if applicable).
B. Replace the eluent generator.
C. Replace the guard column.
D. Replace the analytical column.

6 INSTRUMENTAL CONDITIONS

6.1 Methanesulfonic Acid Cations

Mobile Phase:	Methanesulfonic acid (20mM), supplied from Eluent Generator
Pump Type:	Isocratic
Eluent Generator Mode:	Isocratic
Flow Rate:	1.0 mL/min
Column:	Dionex IonPac CS12A 4x250mm with IonPac CG12A Guard 4x50mm
Column Temperature:	30°C
Cell Temperature:	35°C
Injection Volume:	25 µL
Acquire Time:	15 minute minimum
Suppressor:	CDRS

6.2 Nitric Acid Cations

Mobile Phase:	3.0 mM Nitric Acid (HNO_3) / 0.1 mM EDTA
Pump Type:	Isocratic
Flow Rate:	1.0 mL/min
Column:	Waters IC-Pak Cation M/D 3.9x150mm
Column Temperature:	Ambient
Injection Volume:	10 µL
Acquire Time:	15 minute minimum

6.3 Potassium Hydroxide Anions

Mobile Phase:	Potassium Hydroxide (gradient 20-80 mM)
Pump Type:	Isocratic
Eluent Generator 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
Injection Volume:	25 µL
Acquire Time:	25 minute minimum
Suppressor:	ADRS

6.4 Potassium Carbonate Anions

Mobile Phase:	Potassium Carbonate (10mM), supplied from Eluent Generator
Pump Type:	Isocratic
Eluent Generator 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
Injection Volume:	25 µL
Acquire Time:	16 minute minimum
Suppressor:	ADRS

7 ACCEPTANCE CRITERIA

7.1 Testmix

A. In order for the instrument to be considered in good operating condition, all components from the testmix should generate well-resolved, Gaussian-shaped peaks with baseline separation.
B. An SNR of 3:1 is the minimum response necessary to consider a response a peak.
C. The retention times of the testmix components should not deviate by ±5% compared to the previous run of the testmix (unless LC maintenance has been performed, such as column replacement or new mobile phase).

8 LIMITATIONS

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

9 REVISION HISTORY

Revision	Issued	Changes
04	07/01/2022	Revised to match new format requirements. Section 3- Updated instrument models and added suppressors. Section 4- Added more detail to testmix solutions. Section 6- Added 'Eluent Generator Mode' for Dionex.
05	11/15/2022	Section 2- Updated Scope to remove location. Section 6- Changed from specific instruments to mobile phase based conditions. No other substantive changes to content.