

Guidelines

Xenoic™

Passivation of stainless steel-based HPLC and IC instruments

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Scope

This document gives general recommendations regarding procedures for passivation of stainless steel-based analytical liquid chromatography instruments (HPLC and IC) for use in ion chromatographic analysis using conductivity detection with eluent suppression by XAMS Membrane Suppressor and ASUREX Automatic Regenerator. Note that the applicability of these procedures must be verified against the recommended usage conditions in the manuals from each manufacturer of the chromatographic equipment.

Preparations



Remove all columns from the system before performing any passivation.

1. First verify against the user manuals that the passivation procedure intended to be used is compatible with your instrument components.
2. Before performing any of the listed passivation procedures, make sure you first disconnect the column, and replace it with a union and then wash the entire system with purified water to remove any previous eluents.
3. Use existing stainless-steel tubing to connect the different system modules from degasser to pump(s), to gradient mixer and to column compartment's heat exchanger (if present), to injector or autosampler, to detector, and finally to waste.
4. Ensure you will collect the waste from the passivation solutions in a controlled manner and dispose the waste according to local regulations.
5. If possible, place the injector in the inject position such that the entire loop is flushed, but do not perform any injections as a part of, or during, the passivation procedures.

All chemicals used should be of analysis grade or better and the water should be purified to contain less than 10 ppb total organic contaminants and deionized to have a resistivity of 18.2 MΩ/cm.

Procedures



Wear protective clothes, glasses and gloves when performing passivation.

Make sure you wear protective clothes, glasses and gloves when performing any of the passivation procedures.

The passivation procedure that typically achieves the best results with of lowest amount of dissolved metal ions, is by consecutive washing with dilute formic acid followed by phosphate solution and water. A faster procedure, however, involving much more aggressive chemicals, is by washing with a strong nitric acid solution and water.

If a gradient system with multiple pump heads is used, increase the total flow rate accordingly and make sure every pump head operates with equal percentage of the total flow rate.

Phosphate passivation

1. Prepare a 1000 ml 80:20 acetonitrile/water solution containing 1% formic acid, by mixing 200 ml of acetonitrile with 800 ml of water containing 1% formic acid in a solvent bottle and connect the pump supply line(s) to the solvent bottle.
2. Set the column compartment and heat exchanger to minimum 60 °C, or 80-95 °C if possible. Be sure to close the column compartment to keep a stable temperature.
3. Using a flow rate of 0.4 ml/min per pump, flush the system for a minimum of 4 hours or overnight if possible.
 - △ Note: Do not allow the pump to run out of solvent and shut off by making sure that there is enough solvent to pump at the set flow rate for the desired time duration, (e.g. 0.4 ml/min pumps 384 ml in 16 hours).
4. After flushing the system, add 5 mmol/l phosphate (e.g. NaH₂PO₄ 0.6 g/L) to the remaining solvent mixture or one freshly prepared as described in step 1.
5. Change the column compartment's temperature to 40 °C.
6. Using a flow rate of 0.4 ml/min per pump, flush the system overnight, or if possible, over the weekend.
 - △ Note: Do not allow the pump to run out of solvent and shut off by making sure that there is enough solvent to pump at the set flow rate for the desired time duration, (e.g. 0.4 ml/min pumps 384 ml in 16 hours).
7. Replace the solvent bottle containing passivation solution with one containing purified water and wash the entire system using a flow rate of 0.4 ml/min per pump for a minimum of 30 minutes to remove the passivation solution.

Nitric acid passivation



Avoid exposure to fumes and ensure proper ventilation when handling nitric acid.

1. Prepare a 50 ml 52% nitric acid solution by slowly adding 40 ml of concentrated nitric acid (65%) to 10 ml purified water in a solvent bottle and connect the pump supply line(s) to the solvent bottle.
 - △ Note: Do not use more diluted nitric acid since this is more corrosive to stainless steel and may damage your instrument components.
2. Using a flow rate of 0.4 ml/min per pump, flush the system for 30 minutes.
3. Immediately after stopping the nitric acid flow, connect the pump supply line(s) to another solvent bottle containing at least 500 ml purified water and flush the system for 30 minutes, at a flow rate of 0.6 ml/min per pump.
4. Replace the solvent bottle with another one containing 1000 ml purified water and flush the system for a minimum of 4 hours, at a flow rate of 0.4 ml/min per pump.
5. If possible, wash the rear of the pump pistons with purified water after about 30 minutes of flushing the system with water.

Suggested reading

- Kenneth E. Collins, Carol H. Collins, Celso A. Bertran, LCGC North America, 18(6), 2000, 600-608.
- Kenneth E. Collins, Carol H. Collins, Celso A. Bertran, LCGC North America, 18(7), 2000, 688-692.

Notes