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Short communication

Auto-regeneration of anion-trap columns for improved determination of silica by ion chromatography

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Abstract

The goal of this project was to develop an automated method to regenerate the ATC-3 trap columns that are used on the DX-800 on-line ion chromatography silica systems. The old method of regenerating the ATC-3 trap columns was to physically remove the trap columns from the silica system once every 2 weeks and manual regenerate them. A new automated regeneration method was developed by re-plumbing the silica system to allow 300 mM NaOH to run as the eluent. This regenerates the trap column automatically once every 24 h. The data have shown that regenerating the ATC-3 trap columns once per day improves the R.S.D. values for 250 ng/l silica analysis from 26.0 to 8.7%. The length of useful lifetime for the silica concentrator column was increased by an average of 9 months.

Keywords: Instrumentation; Regeneration; Trap column; Silica

1. Introduction

The function of the anion-trap column is to strip trace anion contaminates from the eluent and help extend the life of the concentrator, guard and separator columns [1]. Before this project, the ATC-3 trap columns were physically removed from the silica systems once every 2 weeks for manual regeneration. Using a DQP pump, 100 ml of 300 mM sodium hydroxide was pumped at a flowrate of approximately 1.0 ml/min through the trap column. The trap column was then installed back onto the ion chromatography (IC) system and rinsed with 20 ml of 10 mM sodium hydroxide–10 mM boric acid eluent [1] to equilibrate it prior to running samples. This process involved 2.5 man-hours, increased the risk of contamination, and provided inconsistent silica results. To solve these problems an auto-regeneration system was installed on the silica IC system that automatically regenerates the ATC-3 trap column once per day.

2. Experimental

All data in this experiment was generated on a Dionex (Sunnyvale, CA, USA) DX-800 on-line IC system using an IonPac anion-trap column ATC-3 (35 mm × 4 mm). The IC system uses an eluent of 10 mM sodium hydroxide–10 mM boric acid at a flowrate of 0.30 ml/min and a 20 mM molybdate post-column reagent at a flowrate of 0.15 ml/min. The post-column reagent is made by adding 9.68 g of sodium molybdate to approximately 1800 ml of deionized water and then adding 25 ml of concentrated nitric acid. After vigorously mixing the solution, add 10 ml of sodium dodecyl sulfate and then top off the solution with deionized water to 2000 ml.

All standards are 50 mg/l silica solutions from Hach Chemical Co. (Loveland, CO, USA). A quality control (QC) solution is made by a diluting the Hach silica standard in 1800 ml of deionized water to generate a solution with a concentration of 250 μg/l. During a QC sample on the DX-800 on-line IC, the method takes the 250 μg/l QC solution and dilutes it by 1000 with deionized water to produce a finial QC concentration of 250 ng/l.

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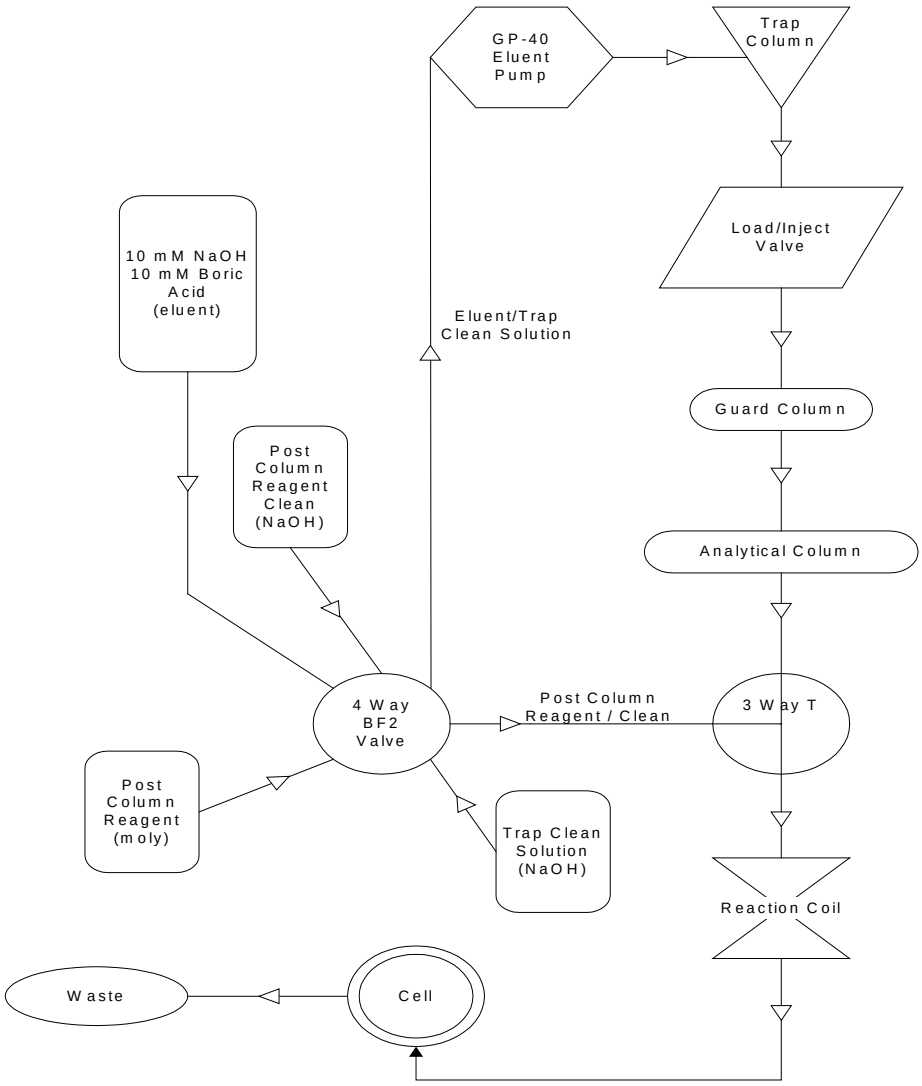

Fig. 1. Flowchart of silica system with on-line trap column clean.

To solve the problems associated with the manual way of regenerating the trap columns, a new method was developed that automatically regenerates the trap columns once per day. This was accomplished by first preparing 2 l of "regeneration solution" consisting of 300 mM sodium hydroxide. Next, plumb the system as per Figs. 1 and 2 to allow the 300 mM sodium hydroxide to run as the eluent during a post-column reagent clean cycle. During a post-column clean cycle, the chromatography software switches a BF-2 valve (Fig. 2) allowing the "regeneration solution" to run as the eluent. The "regeneration solution" was then run through the ATC-3 trap, concentrator, guard, and analytical columns for approximately 60 min at a flowrate of 0.30 ml/min. When the post-column clean cycle is complete, the chromatography software switches the BF-2 valve back to its original position. A blank is then run to allow the system to acclimate back to the eluent prior to running samples.

3. Results and discussion

The benefits of automatically regenerating the anion-trap columns include more consistent silica results, less instrument downtime, less risk of contamination to the system, lower operational costs due to less man-hours involved and increased concentrator column life. The manual way of regenerating the trap columns produced an R.S.D. value of 26.0% on a 250 ng/l silica QC over a 9-month period. Once the automatic trap column regeneration method was installed an R.S.D. value of 8.7% was produced on the same 250 ng/l silica QC over a 12-month period. From Fig. 3 it is easy to see how much more consistent the silica results are with the automatic regeneration method installed. Regenerating the trap columns manually involved approximately 2.5 man-hours per system every 2 weeks. With our four systems, this is equivalent to 20 man-hours of extra work each month. The automatic regeneration method requires

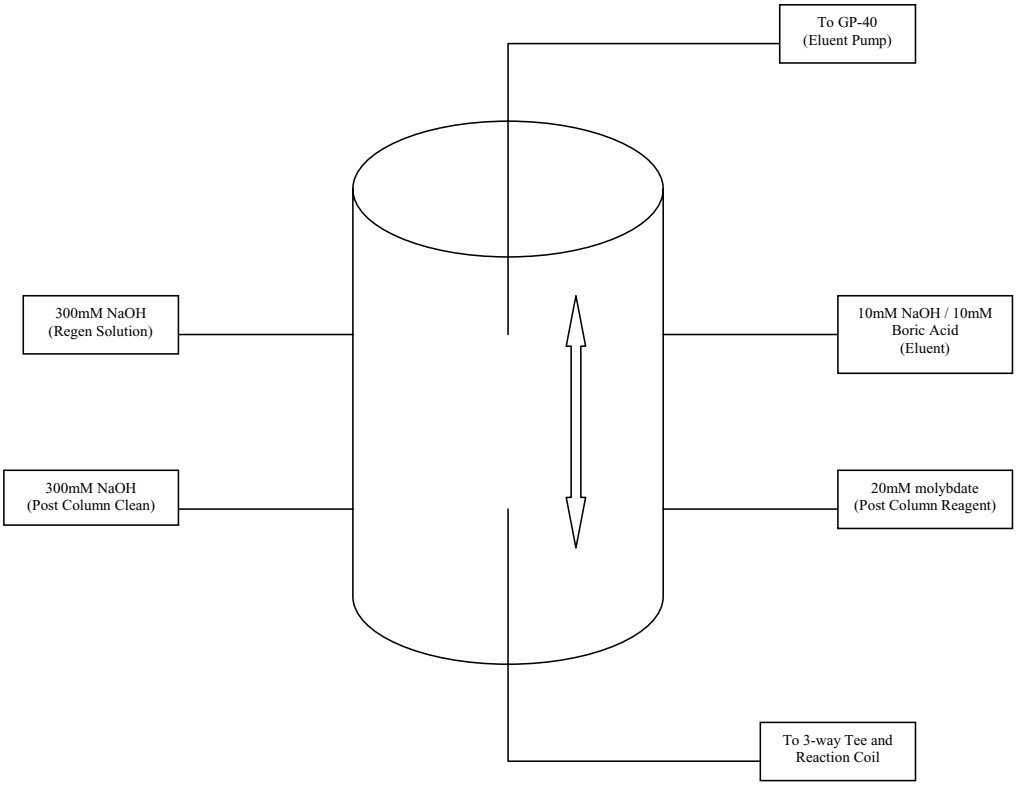

Fig. 2. BF-2 valve configuration.

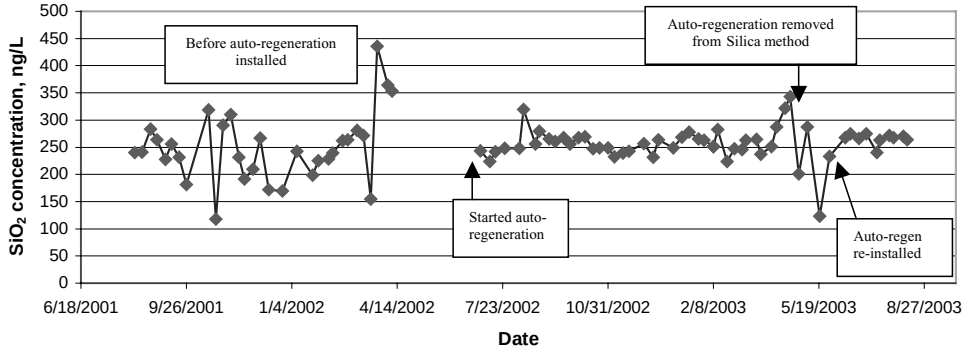

Fig. 3. SiO_2 250 ng/l QC before and after ATC-3 auto-regeneration. Dates as month/day/year.

no extra time per month and frees time to work on other projects. Another benefit of the auto-regeneration method is extending the life of the concentrator column by an average of 9 months. The reduction in man-hours and extended concentrator column life reduces the overall operational cost associated with the silica analysis.

4. Conclusion

The silica on-line IC system has been modified to auto-regenerate the ATC-3 anion-trap columns by switching a BF-2 valve and running 300 mM NaOH as the eluent once per day. This modification decreased R.S.D. values for the silica analysis by 17.3% thus providing more consistent and reproducible silica results. Due to this minor modification we now have a more cost-effective silica analysis by reducing the time spent on maintenance by 20 h per month and extending the concentrator column life by an average of 9 months.

References

[1] IonPac ATC-3 (35 × 4 mm) 2-mm and ATC-3 (24 × 9 mm) 4-mm anion-trap columns Quickstart, Document No. 031837-06, Dionex, Sunnyvale, CA, 2003, p. 1.