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Standard Operating Procedure for:

QuikChem 8500 flow injection analysis system

Operation for

Nitrate / nitrite in waters

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Contents

1. Purpose

The purpose of this SOP is to regulate the operation of flow injection analyzer for nitrate/ nitrite.

2. Scope and application

2.1 This SOP applies to all personnel using and managing QuikChem 8500 for nitrate/ nitrite.

2.2 Samples include surface waters and industrial waste waters.

2.3 The application range is:

High range - 0.2 to 20 mg/L as NO_3^- or NO_2^-

Low range – 0.01 to 2 mg/L as NO_3^- or NO_2^-

3. Principle

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N- (1-naphthyl) ethylene diamine dihydrochloride. The resulting water soluble dye has a magenta color which is read at 520 nm. Nitrite alone also can be determined by removing the cadmium column.

4. Procedure

4.1. Reagents and standards

Use deionized water (DI) for all procedure.

4.1.1 Reagents

Reagent1. 15N Sodium Hydroxide

Add 150g NaOH very slowly to 250 g DI water.

Caution: the solution will get very hot! Swirl until dissolved. Cool and store in a plastic bottle.

Reagent2. Ammonium chloride buffer, pH 8.5

To a tared 1L container, add 85.0 g NH_4Cl, 1.0 g $Na_2EDTA{\cdot}2H_2O$ and 938 g DI water. Shake or stir until dissolved. Then adjust the PH to 8.5 with 15 N sodium hydroxide solution.

Reagent3. Sulfanilamide color reagent

To a 1L dark, tared container, add 876 g DI water, 170 g 85% H_3PO_4, 40.0 g sulfanilamide and 1.0 g N-(1-naphthyl) ethylene diamine dihydrochloride (NED). Shake until wet and stir with stir bar for 30 min until dissolved. **(Monthly)**

4.1.2 Standards

We recommend the use of NO_3^- standards when running the method for $NO_3^- + NO_2^-$.

Standard1. Stock nitrate standard 200 mg N/L as NO_3^-

In a 1L volumetric flask, dissolve 1.44g potassium nitrate (KNO_3) in about 600 mL DI water. Dilute to the mark and invert to mix. **(Six months)**

Standard2. Stock nitrite standard, 200 mg N/L as NO_2^-

In a 1L volumetric flask, dissolve 0.986 g sodium nitrite ($NaNO_2$) or 1.214g potassium nitrite (KNO_2) in about 800 mL DI water. Dilute to the mark and invert to mix**.** **(Refrigerate, 3-5 days.)**

Dilute to working concentration or use auto dilutor.

4.2 Sample preservation and storage

4.2.1 Drinking water:

- For nitrate: chill the sample to 4°C and analyze within 48 hours, unless the sample is chlorinated. If the sample is chlorinated, chill the sample to 4°C and analyze within 14 days.
- For nitrite: chill the sample to 4°C and analyze within 48 hours.
- For nitrate – nitrite (combined): acidify to pH <2 with concentrated H_2SO_4 at the time of collection, and analyze within 28 days.

4.2.2 Wastewater samples:

Requirements are nearly identical except that:

- Nitrite samples must be analyzed with 48 hours whether or not chlorine is present.
- Nitrate – nitrite samples must also be chilled at 4°C.

Caution: Samples must not be preserved with mercuric chloride or thiosulfate because this will degrade the cadmium column.

4.3 Instrument setup

4.3.1 Check if the manifold **(10-107-04-1-A Nitrate/ nitrite)** is installed on system unit. Contact Yili Cheng if not.

4.3.2 Check the connection according to the manifold diagram (fig.1). Port 3 from the diluter (fig.2) is connected to sample tube from manifold.

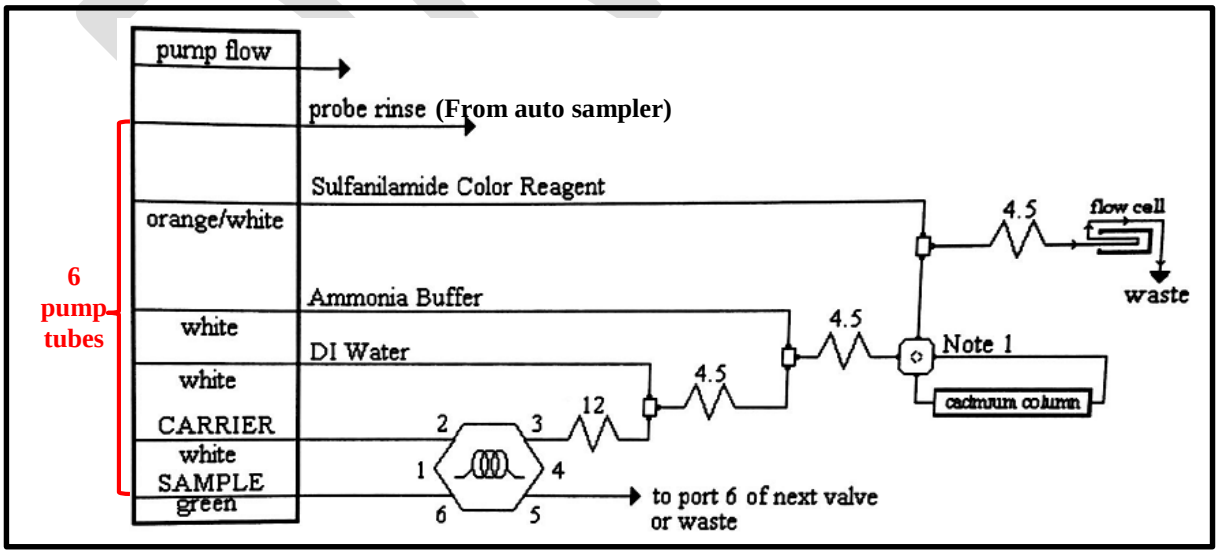


Fig.1 Manifold diagram

4.3.3 Check the state of cadmium column is **offline** (Fig.3).

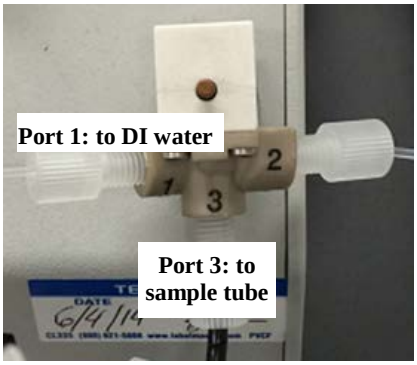


Fig.2 Ports on diluter **Fig.3 Cadmium column**

4.3.4 Make sure the two waste containers (under table) are well labelled and not close to being full. The left one is only samples and carrier (water); it could go into sewer if your sample is clean. The right one must be collected and disposed according to school safety policy.

4.3.5 Put 3 waste lines (fig. 4) into waste containers properly (waste 1 and 2 go into left container, waste 3 go into right container).

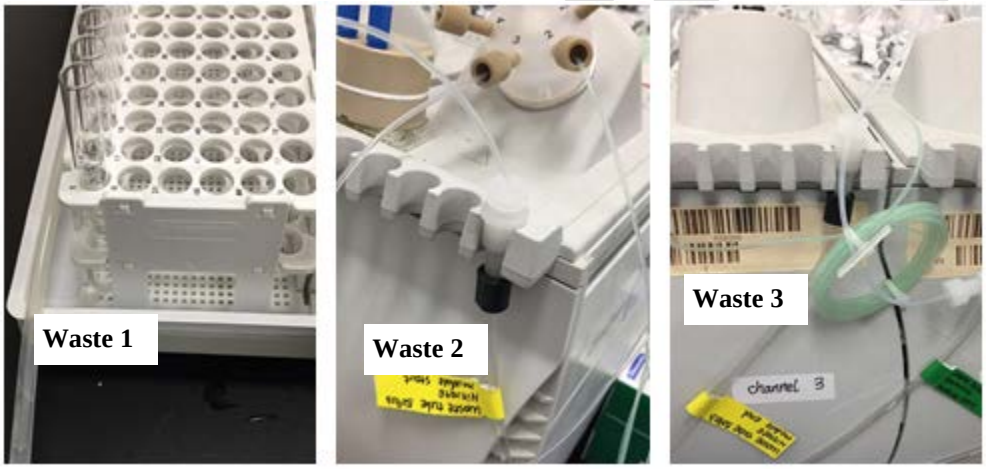


Fig.4 Waste lines

4.3.6 Install tubes in pump (fig.5).

- Mount pump tube into cartridges according to the direction flow. Install the pump cartridges; you can hear “Ka” sound at both sides.
- Adjust tension lever to twelve O’clock direction.
- Install all 6 tubes (show in manifold diagram) and make sure the arrows of cartridges point to the right direction.

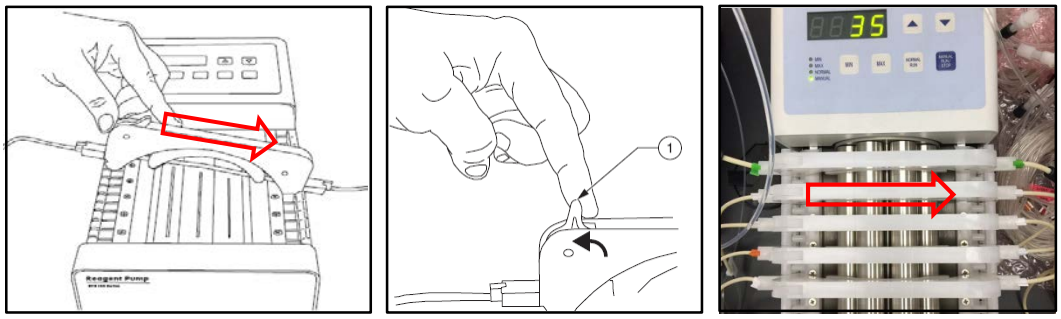


Fig.5 Install pump tubes

4.3.7 Open the six switches as the following order (fig.6).

Fig.6 instrument switch

4.4 System check and washing.

4.4.1 Insert 7 reagent lines into DI container (5 from pump, 2 from diluter, fig.7), and press the start button 

on pump for leak check. Check lines from beginning to the end.

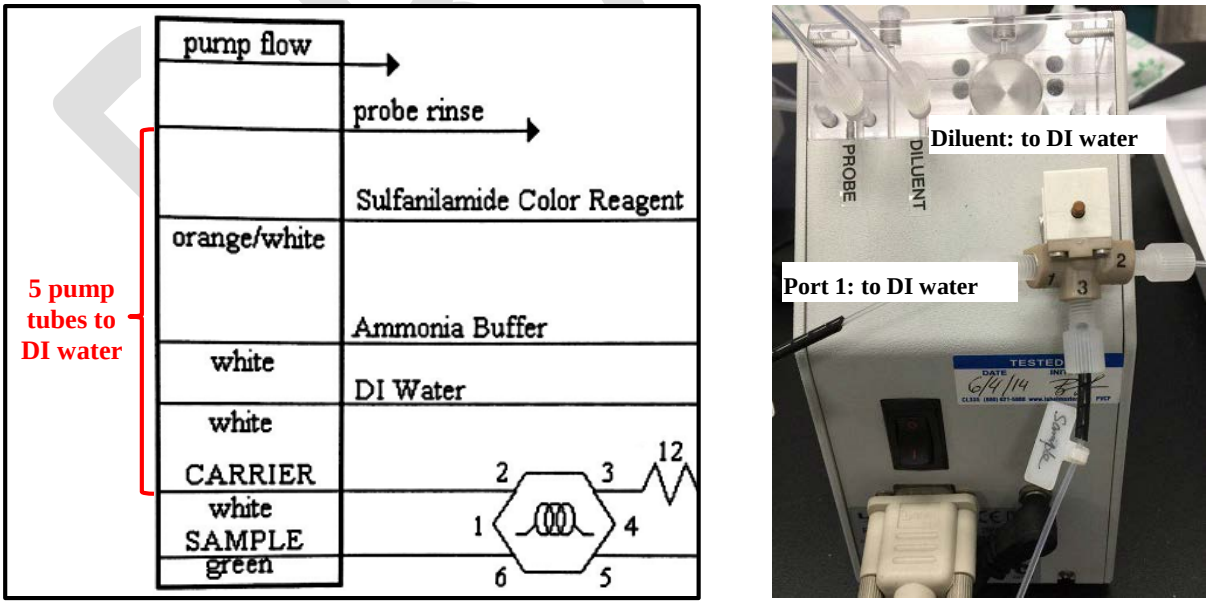


Fig.7 Insert 7 reagent lines to DI water

If there is any leak, taking lines out from DI water, and stop pump 

, and ask LFST to fix it. Wash for 5 min if no leak exits.

4.4.2 Log onto omnion 3.0  . Click 

to open method

4.4.3 Open a previous data file of the method to be run, save as your new one.

4.4.4 Click 

to view the baseline. Wait till it is constant.

4.4.5 Place sulfanilamide color reagent line and ammonia buffer reagent line into appropriate reagents. Pump for 5 minutes.

4.4.6 Switch the valve to place the cadmium in-line after pump reagents for 5 minutes if need to test nitrate. For nitrite, always keep cadmium off-line (Fig.3).

Caution: Never pump air or water through cadmium column.

4.5 Set up a method

This could be down while pump water and reagents.

4.5.1 Set channel and analytes

Run properties >Analytes,

Check the channel and turn off unused channel. Click on the channel/ analyte to edit analyte properties (fig.8).

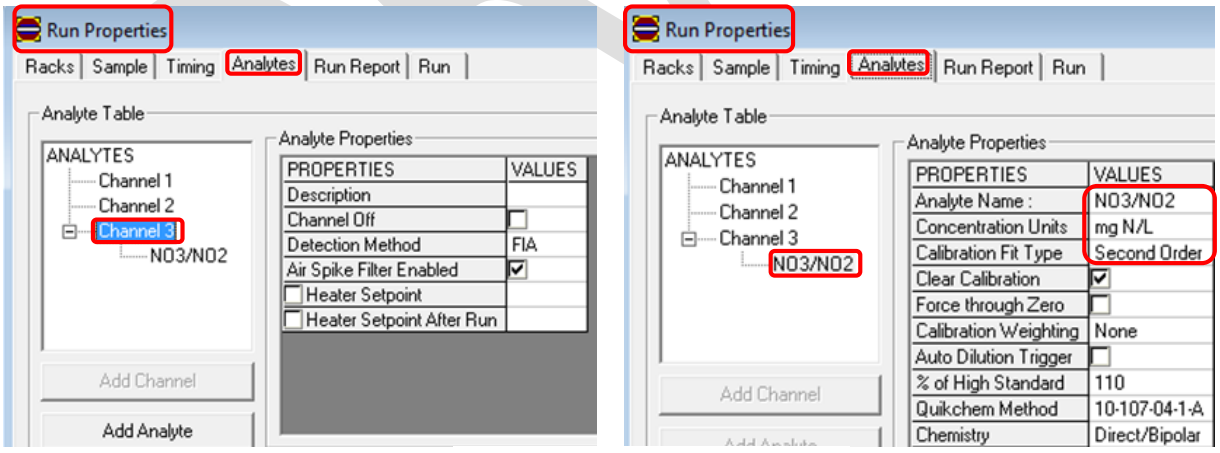
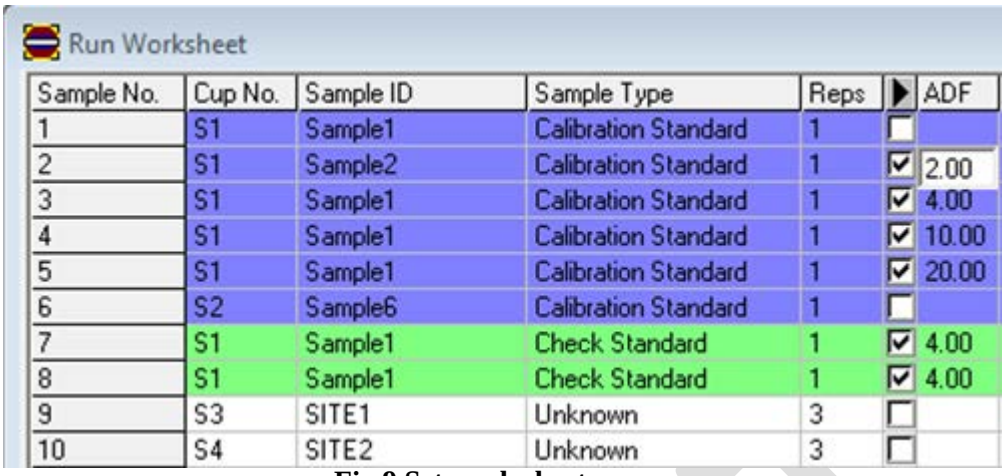


Fig.8 Set channel and analyte

4.5.2 Modify the worksheet with the standards, controls and samples to be run. A typical worksheet shows as follow (fig.9). (ADF: auto dilute factor).



Sample No.	Cup No.	Sample ID	Sample Type	Reps		ADF
1	S1	Sample1	Calibration Standard	1	☐	
2	S1	Sample2	Calibration Standard	1	☑	2.00
3	S1	Sample1	Calibration Standard	1	☑	4.00
4	S1	Sample1	Calibration Standard	1	☑	10.00
5	S1	Sample1	Calibration Standard	1	☑	20.00
6	S2	Sample6	Calibration Standard	1	☐	
7	S1	Sample1	Check Standard	1	☑	4.00
8	S1	Sample1	Check Standard	1	☑	4.00
9	S3	SITE1	Unknown	3	☐	
10	S4	SITE2	Unknown	3	☐	

Fig.9 Set work sheet

4.5.3 Set concentration for each standard and check standard in **run properties** (fig.10).

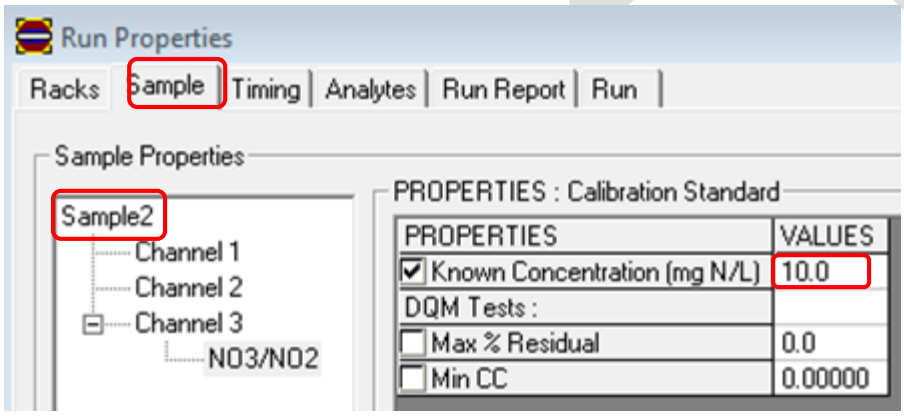


Fig.10 Set known concentrations

4.6 Analysis

4.6.1 Load standard, control and sample vials onto auto sampler according to the worksheet information.

4.6.2 Click on 

to stop preview and then click on 

to start run the worksheet.

4.6.3 After calibration of standards, Click on calibration results icon 
to see the calibration curve, correlation coefficient should be greater than or equal to 0.990. If not, contact Yili Cheng.

4.6.4 For check standard, compute the recovery as follow:

% recovery = D/ k*100. (D= determined value, K= actual value).

% recovery should be within 0.9 -1.1, contact Yili Cheng if not.

4.6.5 Test a midrange calibration standard to check the calibration every 20 samples.

4.6.6 Check the waste containers often during the analysis. Make sure no leak exist.

4.6.7 Click on 

(better choose stop after current sample) if tray needs to be paused (fig. 11).

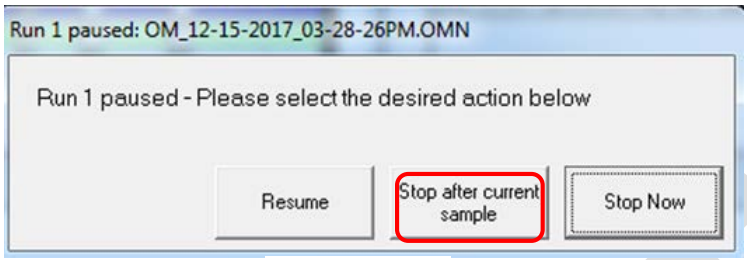


Fig.11 Pause

4.6.8 The run is complete when the start icon turns green.

4.6.9 Export data

Configuration > option > data export (fig.12)

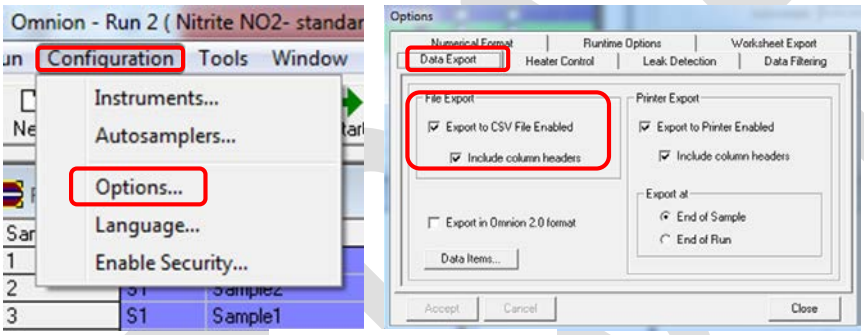


Fig.12 Data export

4.7 System shut down

4.7.1 Set the cadmium column to off-line (fig.3).

4.7.2 Place all reagent lines into DI water and rinse for 8min.

4.7.3 Remove all reagent lines from DI water and pump air for 5 minutes to remove the liquid from the manifold.

4.7.4 Power off pump, remove all pump tubing cartridges from the pump and release the pump tube, store pump tubes appropriately (fig.13).

Fig.13 Store pump tubes

4.7.5 Close the software. Exit windows. Turn off computer.

4.7.6 Power off auto sampler, system unit, diluter and power strip.

5. References:

Lachat applications group, QuikChem Method 10-107-04-1-A, determination of nitrate/ nitrite in surface and wastewaters by flow injection analysis. November 2007.

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