

QAM-I-102

Operation and Calibration
of the Autoanalyzer

Revision 15

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1. Applicability and Purpose

- a. This procedure applies to the operation and calibration of the LaChat Quikchem 8000 Automated Ion Analyzer. An equivalent system may be used with proper revision of this procedure and Demonstration of Performance. Also, the procedure establishes daily, weekly, monthly, and troubleshooting guidelines for the care and maintenance of the instrument.

2. Definitions

- a. Wash Solution (DI) - the instrument start-up solution used for establishing a smooth baseline, before adding other reagents.
- b. Carrier Solution - the solution used to rinse between samples. Also aids flow of reagents in manifold.
- c. Manifold – Reaction block that contains valves and tubing needed to perform analysis
- d. Cycle time- the wash time plus the sample time for the autosampler. Also, the Sample load time plus Inject time on the manifold injection valve.
- e. Method – Analyte Table and Report options for each analysis.
- f. Tray – Calibration, Sample table, and DQM (data quality management) for each analysis.
- g. Analyte Table – Contains Calibration table, Peak width, any Threshold values.
- h. Data Quality Management (DQM) – file containing the quality control information and scheduling for each analysis.
- i. Refer to QAM-Q-101, “Laboratory Quality Control” for standard definitions.

3. Equipment, Reagents, and Standards

- a. Equipment
 - i. ASX 500 Autosampler (XYZ type)
 - ii. RP-100 Reagent Pump
 - iii. Sample processing module which includes the spectrophotometer
 - iv. Data collected is managed using a computer
 - v. Analysis manifolds for each analyte tested. Each includes reagent lines, mixing chambers, sample injection loop, and spectrophotometer filter. (Analyte dependent for wavelength, number, etc.)

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- vi. Reagents: Refer to individual procedure for specific reagents pertaining to each analyte being tested. Record all reagent preparation in the Reagent Logbook.
- vii. Omnion FIA Data System Software, version 2.0
- b. Standards: Refer to individual procedures for the calibration standards. Record all standard preparation in the Standard Logbook.

4. Procedure

a. Operation

i. Instrument start-up.

1. Turn on power to all unit modules at power strip switch located behind main unit.
2. Connect the manifold solution to the reagent lines and insure reagent lines are positioned properly in line tensioners.
3. Check condition of each reagent line. Replace lines that look worn and note in the maintenance logbook. Raise lever to apply tension to pump lines.
4. Snap line tensioners into place across the pump platen. Set final tension for each line being used. Turn the hydraulic pump on by pushing the red Manual Run/Stop button located on the top of the pump. Set the pump speed at 35 ml/min. If there will be a long delay before analysis begins, the gray MIN button may be used to conserve reagent feed.
5. The flow through the reagent lines should go through the reaction block toward the detectors. Pump the manifold solution through the system for 30 minutes, before adding reagents.

ii. Computer start-up.

1. With the instrument on, turn on the computer tower using the gray button in its center.
2. Enter Username and Password in the displayed box. Either click the OK button or press ENTER.
3. Double click the **Omnion FIA** Icon using the mouse pointer. An Omnion Box appears. Click the **OK** button.
4. The Autosampler initialization box appears, and then the Data System logon is shown. Enter an appropriate Username and Password to enter the system. Default entries are not used for security purposes.
5. The Data System selection box appears. Click on the Quikchem FIA icon.

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6. Each valve on the main unit will cycle twice.
 7. The last Method and Tray opened will be displayed immediately afterward.
- iii. Sample/Standard Table
1. Select "File" in the top menu.
 2. Click on "Open Method" in the drop-down menu. Highlight the analysis method to be performed in the selection box that becomes visible (nh3.met, no3no2.met, tptkn.met, or other appropriate method), and click OK.
 3. The analyte table for the method will be displayed. Minimize this table.
 4. Select "File" in the top menu. Click on "Open Tray" in the drop-down menu. Highlight the analysis tray to be used in the selection box that becomes visible (nh3g.tra, no3no2n2.tra, tptkn2.tra, or other appropriate tray), and click **OK**.
 5. Make any corrections to the standard table at the top of the tray.
 6. Enter the sample numbers, duplicates, method blanks, LCS/LCSD standards and spikes into the sample table section of the tray.
 7. If corrections are required, position cursor box where correction is located and click on "Tray" in the top menu.
 8. Select modification needed ("Insert Row" or "Delete Row"). When all changes are completed, highlight all rows in the Cup# column containing sample data and click on "Tray" in top menu. Then select "Renumber cups" from drop-down menu. Enter starting number for numbering cups (normally 1) and make sure that "Renumber samples" box is checked.
 9. Click on OK.
 10. Print the tray by selecting "Tray" from the top menu and then highlighting "Print Tray Worksheet" in drop-down menu. Click **Print** in the next box, and OK in the Print Setup box. Use this printout as a guide for loading the sample trays. Make sure to perform any indicated dilutions for the analysis.
 11. If desired, save the sample table by selecting "File" from the top menu, then "Save Table As" from the drop-down menu.
 12. Type a heading in the highlighted area and click OK.
 13. Type mmddyy (for the run identification) plus a letter to signify which analysis run this is (a for run 1, b for run 2).
 14. Finally, click OK.

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- iv. Output display adjustment
 1. Select "Method" from top menu.
 2. Select "Display Options" from drop-down menu presented.
 3. In the Display Options box, select the channel to be edited.
 4. Change the high and low mV values for the graph and click OK.
 5. Select "File" from the top menu, then "Save Method".
- v. Data Collection
 1. Place lines in analysis reagent bottles as stated in appropriate analysis method.
 2. Allow reagents to pump through system for 20-30 minutes.
 3. Click on the Run Tray icon at the top of the Tray Table screen.
 4. In the Tray Run box that appears type in the Data File name. Use the format **mmddy** plus a letter (**a** for run 1, **b** for run 2, etc).
 5. Click the **Run** button. The analysis should begin.
 6. To stop the analysis run before it completes, click on the **Stop** icon that replaced the **Run** icon when the analysis began.
 7. The calibration may be checked before the sample analysis run begins by clicking the **Review** icon at the top of the Tray screen. Select the analyte channel of interest to review the calibration curve, concentration calculation formula, and calibration correlation coefficient. If changes are required, follow the editing steps below. Print the accepted calibration table and affix to the Personal Logbook with the sample analysis.
 8. At the end of the analysis, the Run Time screen will display a message stating "Tray Run Complete". Also the **Run Tray** icon returns to the top of the Tray screen.
- b. Standard Curve Calculations
 - i. Check the standard curve of the analytical run by clicking the **Review** icon at the top of the Tray screen. If the curve fails, the run can be aborted immediately.
 - ii. Return to Tray screen by clicking "Exit" in the screen menu.
 - iii. Once the run has been aborted, restore the Analyte screen by clicking on it.
 - iv. Make the required changes to the analyte table (i.e. removing standards that indicate poor preparation), or remake the standard where feasible.

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- v. Removal of internal standards from the calibration curve is prohibited unless the point is removed and the calibration rerun.
 - vi. Restart the analysis run.
 - vii. If the highest standard is removed, the upper calibration limit is not used higher than the new high standard. Samples above the calibration limit should be diluted and rerun.
- c. Spreadsheet Preparation
- i. When the analysis run is complete, select "File" from the menu at the top of the screen.
 - ii. Select "Export Data" from the drop-down menu.
 - iii. The Export Data window appears. Ensure that the following items are highlighted: Sample ID, Man Dil Facto, Sample Date, Sample Time, & Results/Peak Area
 - iv. Channel Range area should contain the channels used for analysis. Correct as necessary.
 - v. Click the **OK** button.
 - vi. The "Save Export Data As" screen should be displayed.
 - vii. Change the target folder to **C:\My Documents**, a pen/flash drive, floppy disk, or other storage device, and ensure that the file to be exported is the current analysis file. Click **OK**.
 - viii. To view the data, open "Microsoft Excel."
 - ix. In Excel, Click on the **File Open** icon.
 - x. In the Open box that appears, change the file type to "All Files". Highlight the exported file from those presented and click on **Open**. Since the export file is not in a format that Excel can immediately open, an Import Wizard box is displayed.
 - xi. Make sure that "Delimited" is selected and click **Next**.
 - xii. Check "Comma" as the delimiter. Click **Next** again and then **Finish**. The data file should be displayed in a columnar form with column headings corresponding to the selections when exported.
 - xiii. Insert rows for the report heading, the curve printout, and result column heading(s). Format the final results to have numbers with 4 places to the right of the decimal.
 - xiv. Select all rows and columns of the spreadsheet when through.
 - xv. If more than one page of results is expected, click on Sheet in Page Setup. Click in "Rows to repeat at top" and select the heading rows by highlighting with the computer mouse. Click **Print**. In the Print box, check "Selection" and click **OK**. The file will be printed.
- d. Export of Files to disk or portable device

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- i. Place a portable storage device in the drive and select "Export File"
 - ii. The "Save Export Data As" screen is displayed. Change the target folder to storage device. Select the analyte file displayed and click **OK**. Answer "Yes" if you want to replace the current or older file.
 - iii. Do each analysis channel separately. TP and TKN can be combined into one report if done in the same analytical run.
- e. Troubleshooting
- i. Reagent flow should be regular and forward. Check flow by briefly removing each reagent line from the DI water at start up and watching the progress of the bubble in the line. If there is a problem, continue as follows:
 - ii. Pump 5 N sodium hydroxide and 3 N hydrochloric acid through the lines for 15 minutes each followed by 15 minutes of DI water.
 - iii. If blockage still occurs take apart the connections on the reagent block exhibiting poor or no flow and check the connection tips and the block openings.
 - iv. A needle should be used to clean the block openings if necessary. Alternately, replace /redo the line connection for a better fit. Also, you may try flushing the block with DI water using a syringe.
 - v. Change the lines on the manifold when they begin to feel flat to the touch or appear hazy in the section that is in contact with the rollers.
 - vi. A poor baseline during analysis as evidenced by erratic results may also indicate worn reagent lines. Change all lines at the same time.
 - vii. Refer to the manufacturer's instrument manual for other troubleshooting areas.
 - viii. All maintenance and troubleshooting repairs shall be recorded in the Autoanalyzer Maintenance Log (Elog), Attachment 1.

5. Quality Control and Safety Aspects

- a. All aspects of this procedure shall comply with QAM-Q-101, "Laboratory Quality Control" and QAM-S-101, "Laboratory Safety." The manifold solution and DI wash water shall be prepared fresh each day.
- b. The date of the maintenance and the initials of the analyst who performed it shall be recorded in the maintenance logbook.
- c. There shall be a minimum of four standards and a blank used for the standard curve for each of the analytes being tested.
- d. The baseline of the run shall be \pm the value equal to the LOQ for the analyte being tested.

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6. References

- a. "Flow Injection Analyzer Training Manual", QuikChem 8000 Automated Ion Analyzer Continuum Series, Zellweger Analytics, March, 1999.
- b. The National Environmental Laboratory Accreditation Conference Institute (TNI) standard, 2016.

7. Attachments

- a. Example Autoanalyzer Maintenance Log

Working Copy

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Example Autoanalyzer Maintenance Log

FIA System

Initial and date boxes

<i>Monthly</i>	<i>Monthly</i>	<i>As needed</i>	<i>As needed</i>	<i>As needed</i>	<i>As needed</i>	<i>As needed</i>	<i>As needed</i>
Clean wash reservoir	Wipe platens and rollers	Replace sampler lines	Replace pump lines	Clean sampler probe	Clean analyte manifold	Replace manifold lines	Other/ comments (describe)

Working Copy