QuikChem® Method 12-120-02-1-B

DETERMINATION OF CALCIUM IN SOILS BY FLOW INJECTION ANALYSIS

Lachat Applications Group

Revision Date:

16 May 2012

LACHAT INSTRUMENTS
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QuikChem® Method 12-120-02-1-B

Calcium in 1M Ammonium Acetate Extracts of Soils

0.25 to 50.0 mg Ca/L

10-1000 mg Ca/L

– Principle –

Soil extracts are injected into a 1M Ammonium acetate carrier and mixed with an acidic solution of o-cresolphthalein complexone and 8-hydroxyquinoline. Calcium is complexed by the o-cresolphthalein and magnesium interference is eliminated by adding 8-hydroxyquinoline as a magnesium chelating agent. The pH is then raised to about 10 and the purple colored complex produced is proportional to the calcium concentration.

– Interferences –

1. Magnesium concentrations of approximately 10 mg/L would be required to produce a 0.30 mg Ca/L positive error in the determination of calcium.
2. Soil extracts may have color that interferes. Activated charcoal can be used to remove this interference.
3. Particulates must be filtered prior to analysis of soil extracts.

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QuikChem® Method 12-120-02-1-B

DETERMINATION OF CALCIUM IN SOIL EXTRACTS BY FLOW INJECTION ANALYSIS

1. SCOPE AND APPLICATION

1.1. This method covers the determination of calcium in 1.0M Ammonium Acetate extracts of soils

1.2. The applicable ranges are 0.25 to 50.0 mg Ca/L and 10-1000 mg Ca/L, respectively. The calculated method detection limits are 0.05 mg Ca/L for the low range; and reporting 0.7 mg Ca/L due to y-intercept for the high range. The method throughput is 51 injections per hour.

2. SUMMARY OF METHOD

2.1. Extracted soil samples are injected into a deionized water carrier and mixed with an acidic solution of o-cresolphthalein complexone and 8-hydroxyquinoline. Calcium is complexed by the o-cresolphthalein and magnesium interference is eliminated by adding 8-hydroxyquinoline as a magnesium chelating agent. The pH is then raised to about 10 and the purple colored complex produced is proportional to the calcium concentration.

3. DEFINITIONS

The definitions and purposes below are specific to this method, but have been conformed to common usage as much as possible.

3.1. CALIBRATION BLANK (CB) -- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

3.2. CALIBRATION STANDARD (CAL) -- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

3.3. INSTRUMENT PERFORMANCE CHECK SOLUTION (ICP) -- A solution of one or more method analytes used to evaluate the performance of the instrument system with respect to a defined set of criteria.

3.4. LABORATORY SPIKED BLANK (LSB) -- An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The LSB is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

3.5. LABORATORY SPIKED SAMPLE MATRIX (LSM) -- An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LSM is analyzed exactly like sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.

3.6. LABORATORY REAGENT BLANK (LRB) -- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the if analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

3.7. LINEAR CALIBRATION RANGE (LCR) -- The concentration range over which the instrument response is linear.

3.8. MATERIAL SAFETY DATA SHEET (MSDS) -- Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

3.9. METHOD DETECTION LIMIT (MDL) -- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

3.10. PRACTICAL QUANTITATION LIMIT (PQL) -- The lower level where measurements become quantitatively useful is called the PQL. The PQL is defined as PQL = 10 x s, where s = the standard deviation of 21 replicates of a standard 2.5 – 5 times the MDL.

3.11. QUALITY CONTROL SAMPLE (QCS) -- A solution of method analytes of known concentrations that is used to spike an aliquot of LRB or sample matrix. The QCS is obtained for a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

3.12. STOCK STANDARD SOLUTION (SSS) -- A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

3.13. USEPA -- United States Environmental Protection Agency.

4. INTERFERENCES

4.1. Magnesium concentrations of approximately 10 mg/L would be required to produce a 0.30 mg Ca/L positive error in the determination of calcium.

4.2. Soil extracts may have color that interferes. Activated carbon can be used to remove this interference.

4.3. Particulate must be filtered prior to analysis of soil extracts.

5. SAFETY

5.1. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials.

5.2. Each laboratory is responsible for maintaining a current awareness file of the Occupational Health and Safety Act (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data sheets (MSDS) should be made available to all personnel involved in the chemical analysis. The preparation of a formal safety plan is also advisable.

5.3. The following chemicals have the potential to be highly toxic or hazardous, for detailed explanation consult the MSDS.

5.3.1. Hydrochloric Acid

5.3.2. O-Cresolphthalein complexone

5.3.3. 8-hydroxyquinoline

6. EQUIPMENT AND SUPPLIES

6.1. Balance -- analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2. Glassware -- Class A volumetric flasks and pipettes or plastic containers as required. Samples may be stored in plastic or glass.

6.3. Flow injection analysis equipment designed to deliver and react sample and reagents in the required order and ratios.

6.3.1. Sampler

6.3.2. Multichannel proportioning pump

6.3.3. Reaction unit or manifold

6.3.4. Colorimetric detector

6.3.5. Data system

7. REAGENTS AND STANDARDS

7.1. PREPARATION OF REAGENTS

Use deionized water (10 megohm) for all solutions.

Degassing with helium:

To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in^2) through a helium degassing tube (Lachat Part No. 50100.) Bubble He through the solution for one minute.

Reagent 1. -cresolphthalein Complexone Solution

By Volume: In a **1L** volumetric flask, dissolve **0.5 g o-cresolphthalein complexone, sodium salt** (Fisher-Acros 40575, or equivalent) in a solution of **100 mL DI water** and **50.0 mL concentrated hydrochloric acid** (HCl). Dissolve **2.0 g 8-hydroxyquinoline** (8-quinolinol, C_9H_7NO) in the solution, dilute to the mark with **DI water** and invert three times.

By Weight: To a tared **1L** container add **943 g DI water, 60 g of concentrated hydrochloric acid** (HCl) and **0.25 g o-cresolphthalein complexone, sodium salt** (Fisher-Acros 40575, or equivalent). Shake or stir until dissolved. Add **2.0 g 8-hydroxyquinoline** (8-quinolinol, C_9H_7NO) to the solution and shake or stir until dissolved.

Reagent 2. AMP

By Volume: In a **1L** volumetric flask, dissolve **175.0 g 2-amino-2-methyl-1-propanol** (AMP) in approximately **500 mL DI water**. If the AMP has solidified, warm in hot water before weighing. Dilute to the mark with **DI water** and invert three times.

By Weight: To a **1L** tared container, add **819 g DI water** and **175 g 2-amino-2-methyl-1-propanol** (AMP). If the AMP has solidified, warm in hot water before weighing. Stir or shake until dissolved.

Reagent 3: 1M Neutral Ammonium Acetate (pH~7.0)

By Volume: In a **1L** volumetric flask, dissolve **77.08 g** of **ammonium acetate** (CH_3COONH_4) in approximately **500 mL DI water**. Dilute to the mark with **DI water** and stir to dissolve. This solution is used as the diluent for standards, carrier solution for the method and is also used in extractions. Scale up according to need.

Reagent 4: Washed Activated Charcoal: (For decolorization of soil extracts)

Soak charcoal in extraction solution (**Reagent 3**) overnight. Suction off liquid. Rinse 3-4 times with DI water. Dry in oven, and crush with clean plastic spoon. Approximately 1 scoop (~0.04g) is needed for each sample.

7.2. PREPARATION OF STANDARDS

To prepare the stock and working standards, the following containers will be required:

By Volume: Two 1 L volumetric flasks and six 100 mL volumetric flasks.

By Weight: Two 1L containers and six 125 mL containers.

Standard 1. Working Stock Standard 1000 mg Ca/L in 1M Ammonium Acetate

By Volume: In a **1L** volumetric flask, dissolve **2.77 g calcium chloride** ($CaCl_2$) in a solution of **600 mL DI water** and **77.08 g** of **ammonium acetate** (CH_3COONH_4). Dilute to the mark with **DI water** and invert to mix.

Standard 2. Working Stock Standard 50.0 mg Ca/L

By Volume: In a **1 L** volumetric flask add **100 mL of Standard 1** (1000 mg Ca/L). Dilute to the mark with **Reagent 3 (1M Ammonium Acetate)** and invert to mix. Prepare fresh weekly.

High Range Standards

Working Standards (Prepare Daily) Concentration mg Ca/L	**A** 1000.0	**B** 500.0	**C** 250.0	**D** 100.00	**E** 50.00	**F** 25.00	**G** 10	**H** 0

By Volume

Volume (mL) of **Stock standard 1** diluted to 100 mL with **Reagent 3**	100	50	25	10	5	2.5	1	0.0

By Weight

Weight (g) of **Stock standard 1** diluted to final weight (~100 g) divided by **factor** below with **Reagent 3**	100	50	25	10	5	2.5	1	0.0
Division Factor Divide exact weight of the standard by this **factor** to give the final weight	1	0.5	0.25	0.1	0.05	0.025	0.01	0.0

Low Range Standards

Working Standards (Prepare Daily) Concentration mg Ca/L	**A** 50.0	**B** 25.0	**C** 10.0	**D** 5.00	**E** 2.5	**E** 1.00	**F** 0.5	**G** 0.25	**H** 0.00

By Volume

Volume (mL) of **Stock standard 2** diluted to 100 mL with **Reagent 3**	100	50	20	10	5	2.5	1	0.5	0.0

By Weight

Weight (g) of **Stock standard 2** diluted to final weight (~100 g) divided by **factor** below with **Reagent 3**	100	50	20	10	5	2.5	1	0.5	0.0
Division Factor Divide exact weight of the standard by this **factor** to give the final weight	1	0.5	0.25	0.1	0.05	0.025	0.01	0.005	0.0

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Dried, sieved samples are extracted in 1M ammonium acetate. Soil volume should be chosen based upon expected concentration and measuring range.

8.2. Samples must be filtered prior to analysis

8.3. Samples with interfering color can be treated with washed, activated carbon prior to the analysis. Filter through washed #40 ashless Whatman filter paper or equivalent

9. QUALITY CONTROL

9.1. Each laboratory using this method is required to operate a formal quality control (QC) program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated. An analytical batch shall be defined as environmental samples that are analyzed together with the same method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

9.1.1. Analyses of matrix spike and matrix spike duplicate samples are required to demonstrate method accuracy and precision and to monitor matrix interferences (interferences caused by the sample matrix). The procedure and QC criteria for spiking are described in section 9.3.

9.1.2. Analyses of laboratory blanks are required to demonstrate freedom from contamination.

9.1.3. The laboratory shall, on an ongoing basis, demonstrate through calibration verification and analysis of the ongoing precision and recovery sample that the analysis system is in control.

9.1.4. The laboratory should maintain records to define the quality of data that is generated.

9.2. INITIAL DEMONSTRATION OF PERFORMANCE

9.2.1. Method Detection Limit (MDL) –To establish the ability to detect the analyte, the analyst shall determine the MDL per the procedure in 40 CFR 136, Appendix B using the apparatus, reagents, and standards, that will be used in the practice of this method. An MDL less than or equal to the MDL in section 1.2 must be achieved prior to the practice of this method.

9.2.2. Initial Precision and Recovery – To establish the ability to generate acceptable precision results, the operator shall perform 10 replicates of a mid-range standard, according to the procedure beginning in Section 11.

9.2.2.1. Using the results of the replicates compute the average percent recovery (X) and the standard deviation (s) for the analyte. Use the following equation for the calculation of the standard deviation.

$$s = \sqrt{\frac{\sum x^2 - \frac{\left(\sum x\right)^2}{n}}{n-1}}$$

Where, n = Number of samples, x = concentration in each sample

9.2.2.2. Compare s and x results with the corresponding data in Section 17. If the results meet the acceptance criteria, system performance is acceptable and analysis of samples may begin. If however, s and x do not match the data in Section 17, system performance is unacceptable. In this event correct the problem, and repeat the test.

9.3. Matrix spikes- The laboratory must spike, in duplicate, a minimum of 5 percent of all samples (one sample in each batch of no more than twenty samples) from a given sampling site or if for compliance monitoring, from a given discharge. The two sample aliquots shall be spiked with the stock standard (section 7.2).

9.3.1. The concentration of the spike in the sample shall be determined as follows:

9.3.1.1. If, as in compliance monitoring, the concentration of the analyte in the sample is being checked against a regulatory concentration limit, the spiking level shall be at that limit or at 1 to 5 times higher than the background concentration of the sample (determined in Section 9.3.2), which ever is higher.

9.3.1.2. If the concentration of the analyte in a sample is not being checked against a limit, the spike shall be at the concentration of the precision and recovery standard used in Section 9.2.5 or at 1 to 5 times higher than the background concentration, whichever concentration is higher.

9.3.2. Analyze one sample aliquot out of each set of no more than twenty samples from each site or discharge according to the procedure beginning in Section 11 to determine the background concentration of (B) of the analyte.

9.3.2.1. If necessary, prepare a standard solution appropriate to produce a level in the sample at the regulatory compliance limit or at 1 to 5 times the background concentration (per Section 9.3.1).

9.3.2.2. Spike two additional sample aliquots with the spiking solution and analyze these aliquots to determine the concentration after spiking (A)

9.3.3. Calculate the percent recovery (P) of the analyte in each aliquot using the following equation.

$$P = \frac{(A - B)100}{T}$$

Where, A = Measured concentration of analyte after spiking, B = measured background concentration of analyte, T = True concentration of the spike

9.3.4. The percent recovery of the analyte should meet current laboratory acceptance criteria.

9.3.4.1. If the results of the spike fail the acceptance criteria, and the recovery of the QC standard in the ongoing precision and recovery test of the analytical batch is within the current laboratory acceptance criteria, an interference is present. In this case, the results may not be reported for regulatory compliance purposes and the analyst must assess the potential cause for the interference. If the interference is attributable to sampling, the site or discharge should be resampled. If the interference is attributable to a method deficiency, the analyst must modify the method, repeat the test required in Section 9.1.2 and repeat the analysis of the sample and the matrix spike.

9.3.4.2. If the results of both the spike and ongoing precision and recovery test fail the acceptance criteria, the analytical system is judged to be out of control, and the problem shall be identified and corrected, and the sample reanalyzed.

9.3.5. Compute the relative percent difference (RPD) between two sample results using the following equation:

$$RPD = \frac{(D_1 - D_2)}{(D_1 + D_2)/2} \text{ x } 100$$

Where, D1 = Concentration of analyte in the sample, D2 = Concentration of analyte in the second (duplicate) sample.

9.3.6. The RPD for duplicates shall meet the current laboratory acceptance criteria. If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected and the analytical batch reanalyzed.

9.4 Laboratory blanks – Laboratory reagent water blanks are analyzed to demonstrate freedom from contamination.

9.4.1. Analyze a laboratory reagent water blank initially (with the test in Section 9.2) and with each analytical batch of no more than twenty samples. The blank must be subjected to the same procedural steps as a sample.

9.4.2. If analyte is detected in the blank at a concentration greater than the Minimum Level (Section 1.6), analysis of the samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination. All samples must be associated with an uncontaminated method blank before the results may be reported for regulatory compliance purposes.

9.5. Calibration Verification – Verify calibration using the procedure described in Section 10

9.6. On-going Precision and Recovery (OPR) – With every analytical batch of no more than twenty samples, a midrange standard must be prepared using the procedure described in Section 11.

9.6.1. Compare the results with the current laboratory acceptance criteria. If the criteria are not met, the analytical system is judged to be out of control, and the problem must be immediately identified and corrected and the analytical batch reanalyzed.

9.7. Quality Control Samples (QCS) – It is suggested that the laboratory obtain and/or prepare a quality control sample using a source different from the source routinely used in section 7.2. The QCS is used to verify the concentrations of the calibration standards.

9.8. Depending on the specific program requirements, field replicates and field spikes of the analytes of interest into samples may be required to assess the precision and accuracy of the sampling and sample transporting techniques.

10. CALIBRATION AND STANDARDIZATION

10.1. Prepare a series of standards, covering the desired range, and a blank by diluting suitable volumes of standard solution (suggested range in Section 7.2.).

10.2. Calibrate the instrument as described in Section 11.

10.3. Prepare standard curve by plotting instrument response against concentration values. A calibration curve may be fitted to the calibration solution concentration/response data using the computer. Acceptance or control limits should be established using the difference between the measured value of the calibration solution and the "true value" concentration.

10.4. After the calibration has established, it must be verified by the analysis of a suitable quality control sample (QCS). If measurements exceed ± 10% of the established QCS value, the analysis should be terminated and the instrument recalibrated. The new calibration must be verified before continuing analysis. Periodic reanalysis of the QCS is recommended as a continuing calibration check.

11. PROCEDURE

11.1. CALIBRATION PROCEDURE

11.1.1. Prepare reagent and standards as described in Section 7.

11.1.2. Set up manifold as shown in Section 17.

11.1.3. Input data system parameters as shown in Section 17.

11.1.4. Pump DI water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow the system to equilibrate until a stable baseline is achieved.

11.1.5. Place samples and/or standards in the sampler. Input the information required by the data system, such as concentration, replicates and QuikChem scheme (See Section 17).

11.1.6. Calibrate the instrument by injecting the standards. The data system will then associate the concentrations with the instrument responses for each standard.

11.2. SYSTEM NOTES

11.2.1. For information on system maintenance and troubleshooting refer to the Troubleshooting Guide in the System Operation Manual. This guide is also available on request from Lachat.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Calibration is done by injecting standards. The data system will then prepare a calibration curve by plotting response versus standard concentration. Sample concentration is calculated from the regression equation.

12.2. Report only those values that fall between the lowest and highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12.3. Report results in mg Ca/L.

13. METHOD PERFORMANCE

13.1. The method support data are presented in Section 17. This data was generated according to a Lachat Work Instruction during development of the method.

13.2. Although Lachat Instrument publishes method performance data, including MDL, precision, accuracy and carryover studies, we cannot guarantee that each laboratory will be capable of meeting such performance. Individual laboratory and instrument conditions, as well as laboratory technique play a major role in determining method performance. The support data serves as a guide of the potential method performance. Some labs may not be able to reach this level of performance for various reasons, while other labs may exceed it.

14. POLLUTION PREVENTION

14.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the United States Environmental Agency (USEPA) recommends recycling as the next best option.

14.2. The quantity of chemicals purchased should be based on expected usage during their shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

14.3. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N. W., Washington D. C. 20036, (202) 872-4477.

15. WASTE MANAGEMENT

15.1. It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operation. Compliance with all sewage discharge permits and regulations is also required.

15.2. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel", available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N. W., Washington D. C. 20036, (202) 872-4477.

16. REFERENCES

16.1. Hambleton, L.G., **Journal of the AOAC**, (1977) **60**, 845-852.

16.2. Van Staden, J.F., **Analyst** (1978) **10**, 296-299.

16.3. Lachat QuikChem Method number 10-120-02-1-B

17. TABLE, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

17.1 DATA SYSTEM PARAMETERS FOR QUIKCHEM 8000/8500

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample throughput:	70 samples/h, 70 s/sample
Pump Speed:	35
Cycle Period:	51

Analyte Data:

Concentration Units:	mg Ca/L
High Range	
Inject to Peak Start:	58 s
Peak Base Width:	67 s
Low Range	
Inject to Peak Start:	70 s
Peak Base Width:	62 s
Chemistry:	Direct/Bipolar

Calibration Data Low Range:

Level	1	2	3	4	5	6	7	8	9
Concentration mg Ca/L	50.0	25	10.0	5.0	2.5	1	0.5	0.25	0

Calibration Data High Range:

Level	1	2	3	4	5	6	7	8
Concentration mg Ca/L	1000	500	250	100	50	25	10	0

Calibration Fit Type:	3rd Order Polynomial
Weighting Method:	1/x
Force through zero:	No

Sampler Timing:

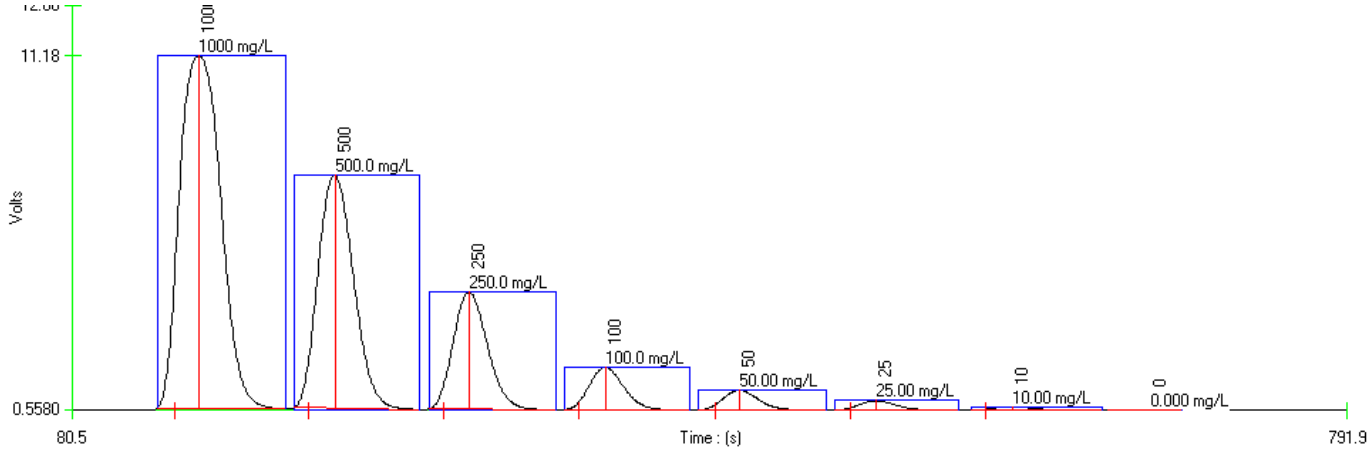
Min. Probe in Wash Period:	5 s
Probe in Sample Period:	30 s

Valve Timing:

Load Period:	20 s
Inject Period:	30 s

17.2. SUPPORT DATA FOR QUIKCHEM 8000/8500

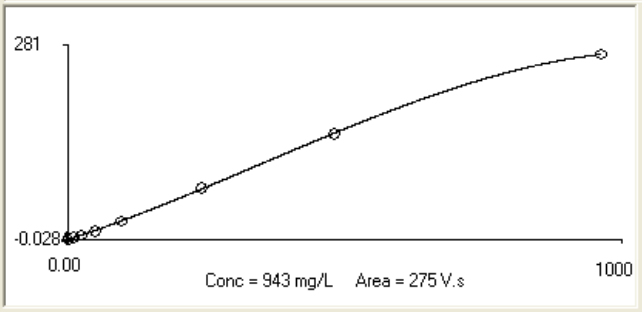
Calibration Data for Calcium; High Range



Calibration Graph and Statistics

Calcium

	Known Conc. (mg/L)	Rep.	Peak Area (V.s)	Peak Height (V)	% RSD	% Resid.	Unused	Det. Conc (mg/L)	Date	Time
1	1000	1	281.4	10.58	0.0	0.0	☐	1001	5/15/2012	11:46:46 AM
2	500.0	1	161.0	6.998	0.0	0.5	☐	497.6	5/15/2012	11:48:02 AM
3	250.0	1	77.78	3.510	0.0	-1.6	☐	253.2	5/15/2012	11:49:18 AM
4	100.0	1	28.19	1.273	0.0	0.3	☐	100.1	5/15/2012	11:50:33 AM
5	50.00	1	13.33	0.5893	0.0	1.9	☐	49.16	5/15/2012	11:51:49 AM
6	25.00	1	6.433	0.2799	0.0	2.3	☐	24.40	5/15/2012	11:53:05 AM
7	10.00	1	2.460	0.1058	0.0	1.8	☐	9.797	5/15/2012	11:54:21 AM
8	0.000	1	-0.02841	-0.001224			☐	0.5150	5/15/2012	11:55:37 AM

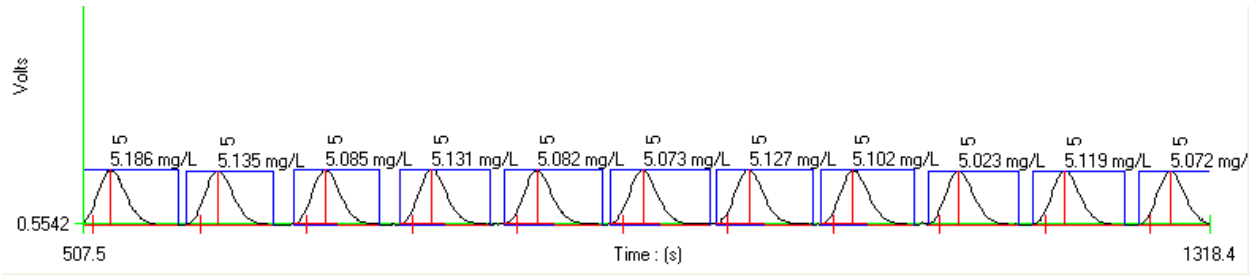


Area = - 2.043e-7 * Conc^3 + 2.214e-4 * Conc^2 + 0.2643 * Conc - 0.1592
Conc = 2.842e-5 * Area^3 - 0.008699 * Area^2 + 3.752 * Area + 0.6216
Correlation Coefficient (r) = 1.00000

Weighting : 1/x

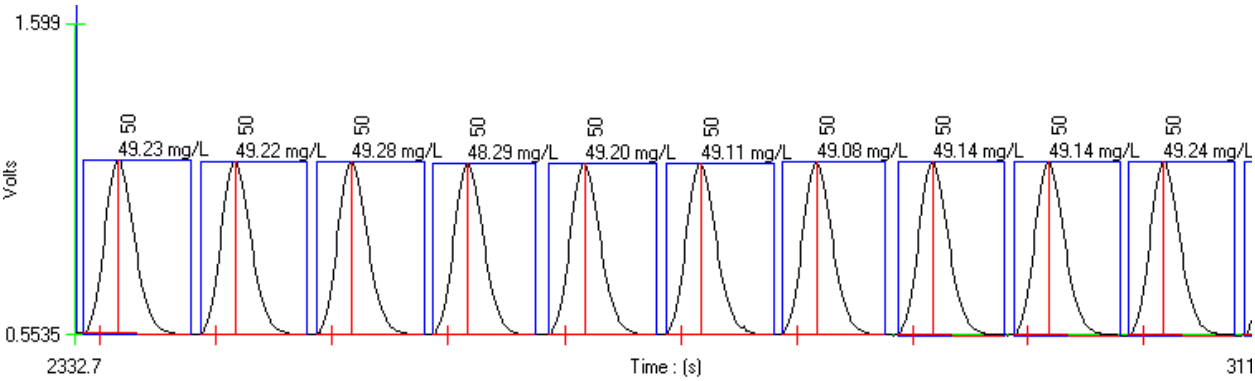
Settings

File Name 10 to 1000 mg cal.omn
Acq. Date: 15 may 2012



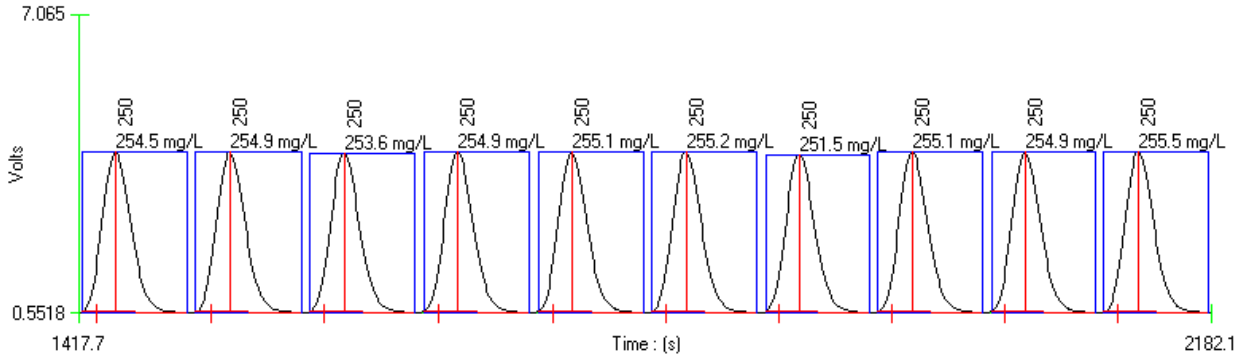
Method Detection Limit for Calcium using 5.0 mg Ca/L

MDL= 0.124 mg Ca/L; Reporting 0.7 mg Ca/L due to carry over.
Standard Deviation (s) = 0.0044 mg Ca/L, Mean (x) = 5.106 mg Ca/L, Known value = 5.0 mg Ca/L
File Name: 10 to 1000 mg support.omn
Acq. Date: 15 may 2012



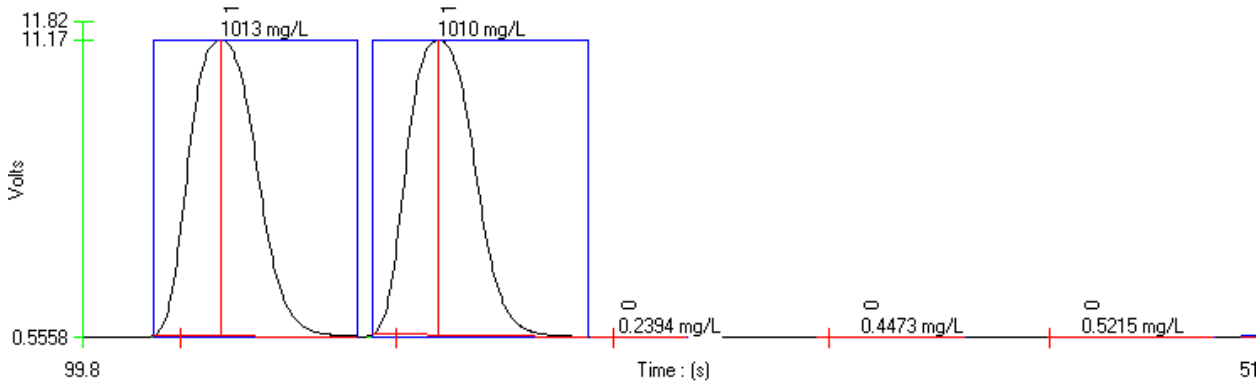
Precision data for Calcium using 50.0 mg Ca/L standard

% RSD = 0.15
Standard Deviation (s) = 0.022mg Ca/L, Mean (x) = 49.2 mg Ca/L, Known value = 50.0 mg Ca/L
File Name:10 to 1000 mg support.omn
Acq. Date: 15 may 2012



Precision data for Calcium using 250.0 mg Ca/L standard

% RSD = 0.46
Standard Deviation (s) = 1.18 mg Ca/L, Mean (x) = 254.52 mg Ca/L, Known value = 250.0 mg Ca/L
File Name:10 to 1000 mg support.omn
Acq. Date: 15 may 2012



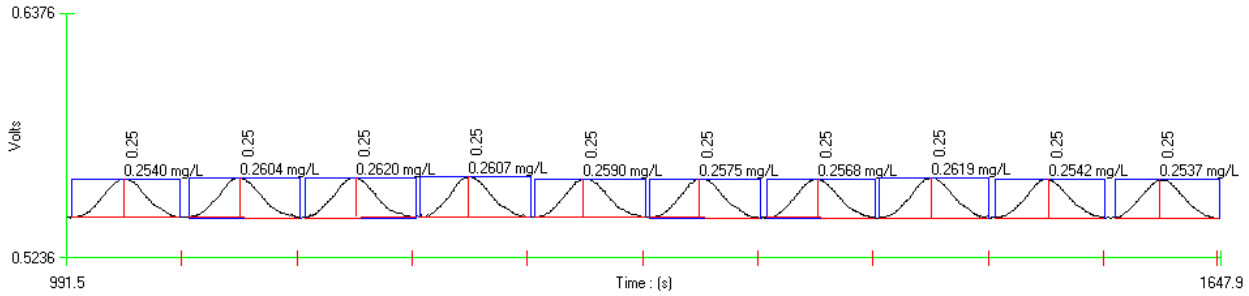
Carryover Study: 2 replicates of 1000.0 mg Ca/L standard followed by 3 blanks

Carryover Failed: Blank concentration after high standard is larger than the calculated MDL of 0.044 mg Ca/L.

Report MDL of 0.6 mg/L.

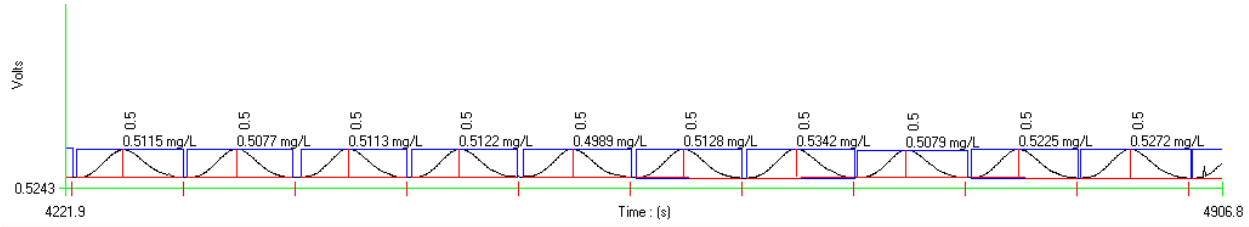
File Name:10 to 1000 mg support.omn

Acq. Date: 15 may 2012



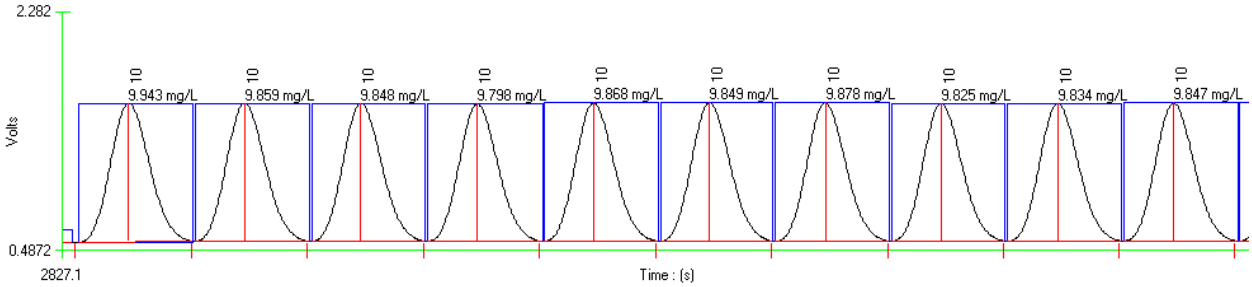
Method Detection Limit for Calcium using 0.25 mg Ca/L

MDL = 0.05 mg Ca/L;
Standard Deviation (s) = 0.0033mg Ca/L, Mean (x) = 0.258 mg Ca/L, Known value = 0.25 mg Ca/L
File Name: 0.25-50 supp.omn
Acq. Date: 15 May 2012



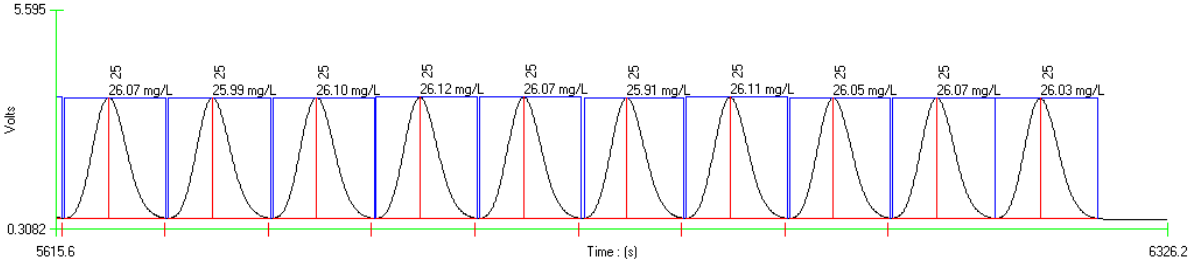
Precision for Calcium using 0.50 mg Ca/L

%RSD= 2.0
Standard Deviation (s) = 0.0104 mg Ca/L, Mean (x) = 0.515 mg Ca/L, Known value = 0.50 mg Ca/L
File Name: 0.25-50 supp.omn
Acq. Date: 15 May 2012



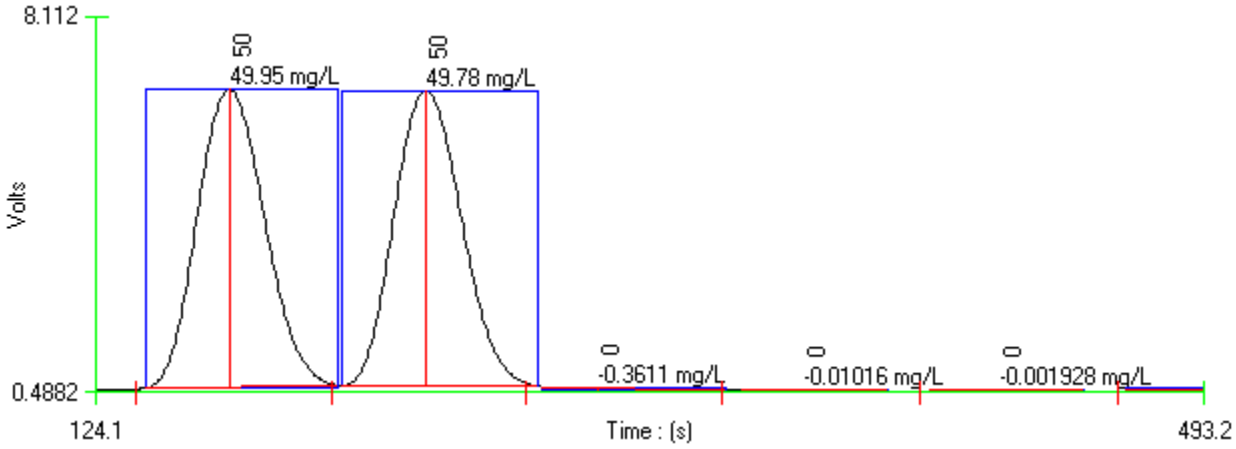
Precision data for calcium using 10.0 mg Ca/L standard

% RSD = 0.39
Standard Deviation (s) = 0.038 mg Ca/L, Mean (x) = 9.86 mg Ca/L, Known value = 10.0 mg Ca/L
File Name: 0.25-50 supp.omn
Acq. Date: 15 May 2012



Precision data for ammonia using 25.0 mg Ca/L standard

% RSD = 0.24
Standard Deviation (s) = 0.063 mg Ca/L, Mean (x) = 26.05 mg Ca/L, Known value = 25.0 mg Ca/L
File Name: 0.25-50 supp.omn
Acq. Date: 15 May 2012



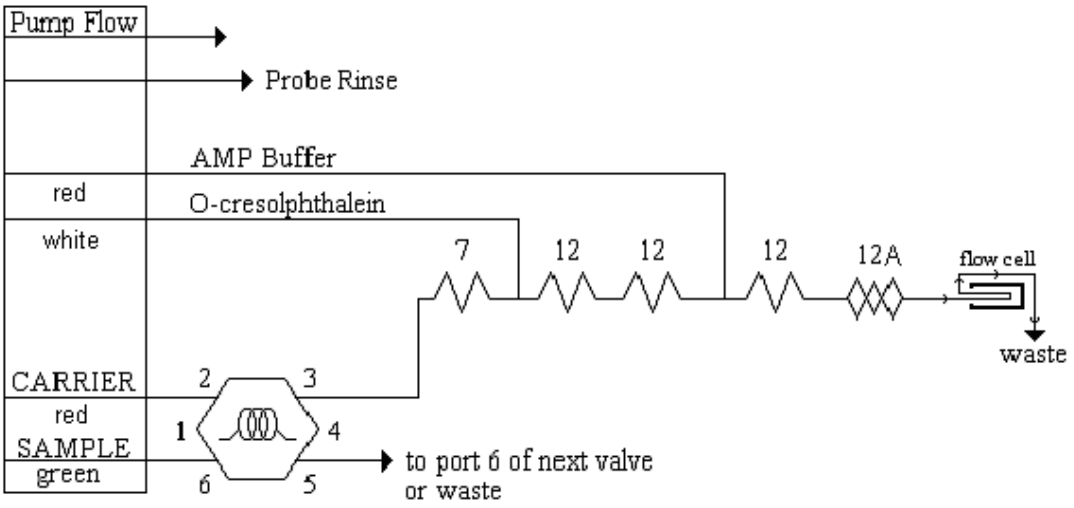
Carryover Study: 2 replicates of 50.0 mg Ca/L standard followed by 3 blanks

Carryover Passed

File Name: Ca Support MDL and Carryover.omn

Acq. Date: 15 May 2012

17.3. CALCIUM IN SOIL EXTRACTS MANIFOLD DIAGRAM



Carrier: **Reagent 3**
Manifold Tubing: **0.8 mm (0.032 in) i.d.** This is **5.2 µL/cm**.
QC8000/8500 Sample Loop,
Low range 120cm 0.8 mm i.d. (0.032 " i.d.)
High Range: 16 cm 0.5 mm i.d. (0.022" i.d.)
Interference Filter: 600 nm

Apparatus: An injection valve, a 10 mm path length flow cell, and a colorimetric detector module is required.

7: **135** cm of tubing on a 7 cm coil support
12: **255** cm of tubing on a 12 cm coil support
12A: **255** cm of tubing on a 12 cm alternating coil support

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