RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 1 of 16

## Analysis of Available Phosphate and Soluble Potash in Fertilizer by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES)

#### 1. Scope:

This document provides a procedure for analysis of available phosphorus (P) and soluble potassium (K) in fertilizers using ICP-OES. It is not suitable for the determination of total phosphorus and potassium.

#### 2. Principle:

An ICP-OES utilizes a plasma energy source to excite electrons to higher energy orbitals, which return to ground state releasing characteristic UV or visible wavelengths that can be quantified.

Solid and liquid samples are stored at room temperature in the sealed glass jars or plastic pouches. Samples that may spoil or ferment shall be stored in a refrigerator. Refer to RA-SP-SMPL-PREP for sample preparation, storage, and disposal.

#### 3. Safety:

All laboratory safety rules for sample preparation and analysis shall be followed. Read the SDS for all materials before use.

Gloves, eye protection, and a lab coat shall be worn when handling hazardous materials and reagents.

Ammonium hydroxide, disodium EDTA and dibasic ammonium citrate can cause skin irritation. Use in a fume hood and avoid contact with skin.

Nitric acid and hydrochloric acid are very toxic and extremely corrosive and shall be used under fume hood. Avoid contact with skin and breathing vapors.

#### 4. Definitions:

- ICV Initial Calibration Verification. A mid-level standard that is analyzed after the calibration standards that is obtained from a different vendor than calibration standards.
- CCV Continuing Calibration Verification. A mid-level standard used to demonstrate the instrument remains in calibration and is analyzed before the first sample, after every 10-12 samples, and at the end of the analytical sequence.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 2 of 16

- CB Calibration Blank. Solution used to prepare calibration standards and is analyzed after the ICV and CCV standards to evaluate carryover.
- ICB Initial Calibration Blank. A 40% ammonium citrate-ETDA solution used to demonstrate no carryover from ICV or instrument.
- CCB Continuing Calibration Blank. A 40% ammonium citrate-ETDA solution used to demonstrate no carryover from CCV, instrument, or sample.
- IS Internal Standard. Scandium and ionization buffer (Cesium) that is mixed with the sample during sample introduction to account for matrix variations.
- MB Method Blank. A reagent blank that is subjected to the entire extraction process to demonstrate all aspects of the extraction are free from interferences.
- QC Quality Control Sample. A NIST or Magruder sample that is prepared and analyzed with each set to demonstrate the accuracy of the test.
- MDL Method Detection Limit. The minimum concentration of an analyte that can be reported and measured with 99% confidence that the analyte concentration is greater than zero and is determined in a given matrix using this method.
- RL Reporting Limit. The lowest level that can be reported accounting for variances in matrices with a reasonable degree of accuracy and precision. The RL is generally 1-5 times higher than the MDL.

#### 5. Interferences:

- 5.1. Spectral interferences are characterized by an overlap (either partial or direct) of the analyte of interest by an interfering element. Also, the background signal for determination of an analyte signal can interfere. These interferences can lead to suppression or enhancement of signals.
- 5.2. Physical interferences are characterized as a difference in matrix between samples and calibration standards which affect the sample transport or nebulization.
- 5.3. Chemical interferences occur when there is a difference in the way that the sample and the calibration standards react in the plasma such as ionization, molecule formation and plasma loading. A special case of chemical interferences emerges when the sample contains a high concentration of easily ionized elements (alkali group elements).

#### 6. Equipment and Supplies (equivalents are acceptable):

- 6.1. PerkinElmer (PE) ICP-OES AVIO 5000
- 6.2. Hot water bath-shaker unit (Serial # 14188-1)

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 3 of 16

- 6.3. Analytical balance capable of weighing 0.0001g
- 6.4. Class A 250 mL and 50mL volumetric flasks
- 6.5. 15mL Polypropylene sample tubes with caps (VWR Cat# 10026-076)
- 6.6. 50mL Graduated polypropylene beakers (VWR Cat# 25384-150)
- 6.7. Class A 100mL glass graduated cylinder (VWR Cat# 470228-968)
- 6.8. Whatman No. 2 filter paper (Sigma Aldrich Cat# 1002-110)

## 7. Reagents (equivalents are acceptable):

- 7.1. Nitric acid, 67-70%, Trace Metal Grade, CAS # 7697-37-2 (Fisher Scientific Cat# A509-P212)
- 7.2. EDTA (ethylenediamine tetra acetic acid), disodium salt, dihydrate, CAS #6381-92-6 (Fisher Scientific Cat# S311-500)
- 7.3. Ammonium citrate, dibasic, CAS # 3012-65-5 (Fisher Scientific Cat# A663-500)
- 7.4. Ammonium hydroxide 28-30% as NH<sub>3</sub>, CAS # 1336-21-6 (VWR Cat# 470300-204)
- 7.5. Triton X-100 Octyl phenol ethoxylate (CS 282-4)
- 7.6. Cesium chloride, CAS # 7647-17-8 (VWR Cat# BDH4124-1KG)
- 7.7. Scandium, 1000µg/mL in 2% nitric acid (SPEX CertiPrep Cat# PLSC2-2T)
- 7.8. Manganese, 1000µg/mL in 2% nitric acid (SPEX CertiPrep Cat# PLMN2-2T)
- 7.9. Phosphorus, 2000µg/mL, in water (Exaxol Cat# SP6170-1)
- 7.10. Potassium, 3000µg/mL, in water (Exaxol Cat# SP6202-1)
- 7.11. Phosphorus, 1000µg/mL in 2% nitric acid (SPEX CertiPrep Cat# PLP9-2X)
- 7.12. Potassium, 1000µg/mL in 2% nitric acid (SPEX CertiPrep Cat# PLK1-2X)

## 8. Standard and Extraction Solution Preparation:

Stock standards are purchased as custom standards from ISO 17034 accredited source accompanied by a certificate of analysis. Stock solution standards are stored at room temperature in polypropylene bottles with labels listing the expiration date.

- 8.1. Ammonium citrate-EDTA extraction solution is prepared by adding ~1.5L of DI water to a 2L volumetric flask. Add 25g of disodium EDTA and 50g dibasic ammonium citrate and mix to dissolve. In a fume hood, add 30mL of 1:1 NH<sub>4</sub>OH-H<sub>2</sub>O (v/v) and mix well. Adjust pH to 7.00 with additional 1:1 NH<sub>4</sub>OH-H<sub>2</sub>O dropwise using pH paper. This solution is susceptible to microbial activity and should be made frequently. The shelf life of this solution is approximately two weeks.
- 8.2. A 40% ammonium citrate-ETDA solution is made fresh for each set by mixing 100mL of the above ammonium citrate-EDTA solution with 150mL DI water for the MB. In addition, this solution is used for the ICB, CCB, and sample dilution.
- 8.3. Calibration standards are made by using ammonium citrate-EDTA solution and DI water. If standards are stored in a dark place, they have a shelf life of up to 3 weeks. Calibration standard preparation details and concentration levels are shown in Table 1.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 4 of 16

- 8.4. Custom standards that are prepared in a water and preserved with a biocide are strongly recommended as standards preserved in acid do not work due to the changes in the matrix and pH.
- 8.5. P standards are ranked from the lowest to highest concentration, while K standards are in the inverse order. This is intended because fertilizers usually do not contain high levels of both P and K (e.g. 52% P<sub>2</sub>O<sub>5</sub> and 60% K<sub>2</sub>O) and because high K impacts ionization, which can adversely impact P and K recoveries so standards containing both high P and high K should be avoided.
- 8.6. A 0 to 500  $\mu$ g/mL P and 0 to 1050  $\mu$ g/mL K calibration range is required to cover the range of 0.08%-57.3% for P<sub>2</sub>O<sub>5</sub> and 0.08%-63.2% for K<sub>2</sub>O in fertilizer.
- 8.7. Standard #5 is used for CCV and the same concentration from second source for ICV (see Table 1).
- 8.8. 40% Citrate EDTA extraction solution for the MB sample.
- 8.9. A Magruder reference sample is used for the QC sample.
- 8.10. 0.5% Triton X solution is prepared by diluting 1mL of Triton X-100 to 200mL with DI water.
- 8.11. Autosampler wash solution mix (2%/0.5% HNO<sub>3</sub>/HCI) is prepared by diluting 20mL of HNO<sub>3</sub> and 5 mL of HCI to 1000mL with DI water.
- 8.12. The internal standard solution (IS) (10μg/mL scandium and 0.018M cesium chloride in 4% HNO<sub>3</sub>) is prepared by dissolving 3g cesium chloride in DI water then mixing with 10mL of 1000μg/mL scandium stock solution, 40mL of concentrated HNO<sub>3</sub>, and 1mL of 0.5% Triton-X in a 1L volumetric flask and filling to mark with DI water.
- 8.13. The alignment solutions are 1mg/L manganese solution for axial and 10mg/L for radial prepared by diluting 0.1mL and 1mL of manganese stock solution with DI water to 100mL.

## 9. Sample Extraction:

- 9.1. For samples with P and/or K guarantees > 1% but < 40%, weigh ~0.5g (± 0.01g) of solid sample on a weigh paper and completely transfer to a 250mL class A volumetric flask. Record the exact sample weight (for liquids or slurries, mix well to evenly distribute any solid matter, then immediately transfer to tared 250mL volumetric flask with a disposable pipette.)</p>
- 9.2. Weigh 1-3g for samples with guarantees < 1% P and/or K. Weigh 0.4g for samples with guarantees > 40% P and/or K.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 5 of 16

- 9.3. Turn on the hot water bath-shaker and set the temperature to 65°C. Preheat the citrate-EDTA solution to 65±5°C, add 100mL to each flask, then insert a rubber stopper.
- 9.4. Shake the samples in the heated shaking unit gently at 65±5°C for one hour. The shaking rate (200rpm) should be set so that solution and/or solids do not build up in the neck of volumetric flask.
- 9.5. After the one-hour shaking is complete, remove samples from the heating unit. Allow samples to cool to room temperature (20 to 25°C), then dilute to volume with deionized water, stopper and mix well.
- 9.6. Filter each sample using a Whatman No. 2 filter paper and make dilutions If any visible debris is present in the extract. The sample will require filtration prior to ICP analysis.
- 9.7. Transfer ~10mL prepared sample to a 15mL centrifuge tube for ICP analysis. Samples may be stored at room temperature, however should be analyzed as soon as possible.

#### **10. Instrument Calibration:**

- 10.1. Refer to Table 2 for recommended P and K wavelengths and Table 3 for ICP-OES conditions and settings.
- 10.2. Verify the outlet gas pressures are 80-120 psi for argon and 80-100 psi for nitrogen.
- 10.3. Verify the chiller is at ~15°C and ~50 psi.
- 10.4. Inspect the ICP torch and injector for cleanliness. If either shows signs of build-up or staining, remove, and follow the cleaning procedure outlined in Section 17.1.
- 10.5. Inspect the nebulizer for clogs and the spray chamber for any evidence of build-up. If either is dirty, remove and follow the cleaning procedure outlined in Section 17.2.
- 10.6. Inspect the sample pump and peristaltic pump tubing and fittings to ensure they are clean and in good condition (see section 17.4).
- 10.7. In the instrument software on the Instrument tab, click Plasma Control and perform the following:
  - 10.7.1. Activate peristaltic pump and check the solution flow through the sample introduction system.
  - 10.7.2. Click on the pump icon to activate the peristaltic pump and allow solution to flow until it has filled rinse station.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 6 of 16

- 10.8. To ignite plasma, click Plasma On/Off tab. If the plasma does not ignite, then perform the following:
  - 10.8.1. Check the sample introduction system to ensure all connections are made and are not leaking.
  - 10.8.2. Ensure there is enough Argon and the pressure is 80-120 psi.
  - 10.8.3. If the torch, injector, or nebulizer were removed prior to ignition, check that all O-rings are in good condition and that all parts have been seated correctly.
- 10.9. Once the plasma is lit, allow the ICP to stabilize for at least 1 hour before calibration.
- 10.10. Optimization is only needed if the torch was removed or replaced. In Instrument tab, click Spectrometer Control and follow the steps to realign the X-Y position of the torch:
  - 10.10.1. Re-align the axial spectrometer
    - 10.10.1.1. Click the axial icon in the Spectrometer Control window.
    - 10.10.1.2. Place autosampler probe in 1mg/L Mn solution and aspirate.
    - 10.10.1.3. Click on the align view and ensure the wavelength is set for Mn 257.610 and the delay time is set to 30 seconds.
  - 10.10.2. Re-align the radial spectrometer window by
    - 10.10.2.1. Click the radial icon in the Spectrometer Control window.
    - 10.10.2.2. Place auto sampler probe in 10mg/L Mn solution and aspirate.
    - 10.10.2.3. Click on the radial view. Ensure the wavelength is set for Mn
      - 257.610 and the delay time is set to 30 seconds.
  - 10.10.3. Once alignment is complete, send autosampler probe to wash position by clicking "Go to Wash" in Instrument tab to start at proper position.
- 10.11. The ICP-OES is calibrated each day of operation using a calibration blank and nine levels of calibration standards. Whenever possible, linear regression is preferred. Because of the dynamic range in fertilizer K concentration, non-linear curve fit may be necessary. Analyze the calibration standard solutions from Table 1.
  - 10.11.1. Use a linear regression of the calibration data for all P and one K (766 Radial) wavelengths passed through zero. The correlation coefficient (r) shall be ≥ 0.995. If any element fails, the instrument shall be recalibrated, and any samples analyzed using the failed curve shall be reanalyzed.
  - 10.11.2. Use a non-linear (calculated intercept) regression of the calibration data for K404 Axial and K766 Axial. If the correlation coefficient (r) is at 0.995 or greater, the system is calibrated, and the analysis of the samples may proceed.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 7 of 16

10.11.3. Elements that do not meet this criterion are not valid and recalibration is required.

#### 11. Analysis:

- 11.1. After the initial calibration is completed and the ICV and CB results are within the acceptance criteria, samples may be run. The acceptance criteria are discussed in Section 12. Report the average of at least three replicate measurements for all QC and samples.
- 11.2. Flush the system with instrument rinse solution (2% HNO<sub>3</sub> and 0.5% HCl) between each sample and standards to prevent carryover.
- 11.3. Dilute and reanalyze samples that are out of the calibration range or if it is needed.

#### 12. QA/QC:

12.1. Initial Calibration Verification (ICV):

An ICV standard is analyzed immediately after the initial calibration. The calibration curve is verified by the analysis of an ICV standard from the second source at or near the mid-range.

Acceptance Criteria: The ICV percent recovery is  $\pm 10\%$  of the true value. The % RSD shall be  $\leq 10\%$ . The ICV can be reanalyzed but must be successful twice in succession or corrective action must be taken.

**Corrective Action**: ICV wavelengths that do not pass these criteria are not valid to report. Continue the analysis if you don't need those wavelengths for the final report. Prepare new ICV standard and recalibrate until results are acceptable if those wavelengths are required for report.

12.2. Calibration Blank (CB):

The ICB is analyzed immediately after ICV and the CCB is analyzed after all CCV analyses.

Acceptance Criteria: All the analytes in the CB shall be less than the reporting limit (RL) for each element. However, the result may be greater than the Minimum Detection Limit (MDL) if the result does not adversely impact data quality.

**Corrective Action:** If the CB exceeds the acceptance limits, instrument contamination and carry-over from solutions (Citrate-EDTA solution and DI water) should be the sources of contaminations. Run the instrument rinse solution for at

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 8 of 16

least 3-5 minutes. Reanalyze the blank. If unsuccessful, locate and resolve the contamination problem before continuing.

12.3. Method Blank (MB):

The MB is analyzed after ICB and before samples to evaluate that the system (extraction procedure) is clean and free of interferences and checks for carryover.

Acceptance Criteria: All the analytes in the MB shall be less than the reporting limit (RL) for each element. However, the result may be greater than the Minimum Detection Limit (MDL) if the result does not adversely impact data quality.

**Corrective Action:** If the MB exceeds the acceptance limits, instrument contamination and carry-over from extraction procedure (glassware) are resources of this issue. Run the instrument rinse solution for at least 3-5 minutes. Reanalyze the blank. If unsuccessful, locate and resolve the contamination problem before continuing.

12.4. Continuing Calibration Verification (CCV):

The CCV is run after every 10-12 samples or less and at the end of the analytical sequence.

Acceptance Criteria: The CCV percent recovery is  $\pm$  15% of the true value. The % RSD shall be  $\leq$  10%. The CCV can be reanalyzed but must be successful twice in succession or corrective action must be taken.

**Corrective Action:** Wavelengths in CCV that do not pass these criteria are not valid to report. Continue the analysis if you don't need those wavelengths for the final report. Prepare new CCV standard and recalibrate until results are acceptable if those wavelengths are required to be used.

12.5. Quality Control Sample (QC):

A QC Sample shall be run with each set of samples. Each set includes 22 samples, one QC sample and one method blank. Both QC and method blank were processed through the entire analytical extraction method. QC should be a similar matrix as the samples and processed using the same extraction and analysis method.

Acceptable QC include reference material from NIST or a collaborative check sample such as Magruder reference sample with a known mean and standard deviation.

Acceptance Criteria: For NIST, result must be within the certified value. Magruder reference sample, results shall be within the reported mean of  $\pm 2$  standard deviations.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 9 of 16

**Corrective Action:** If recovery criteria are not met for one or two wavelengths, those wavelengths are not going to be used but the rest with acceptable criteria are valid to report. If all wavelengths fail, control samples must be re-extracted and re-analyzed.

12.6. Internal Standards (IS):

Scandium is used as an IS and its intensity is monitored during analysis.

Acceptance Criteria: The intensities of all internal standards are monitored in every sample and shall be within 80-120%.

**Corrective Action:** If the IS intensities are outside of this range, dilute and reanalyze the sample.

12.7. Method Detection Limit and Reporting Limit:

The method detection limit (MDL) is the lowest concentration of the analyte that a method can detect reliably, and reporting limit refers to a level at which reliable quantitative results may be obtained. The calculated MDL and RL for all wavelengths are shown in Appendix 1.

12.8. Blank Rinse:

Flush the system between standards and samples with the instrument rinse solution to prevent carryover from samples with high concentrations.

## 13. Discussion:

- 13.1. P213 and P214 in both view modes (Axial and Radial) are the preferred wavelengths because they are the strongest lines and cover wide range of concentrations from 0-500µg/mL. P177 and P178 are less sensitive lines which are relatively free from spectral interferences. However, these lines are weaker than P213 and P214 which could impact recovery of some of the lower P concentration. It is recommended to use P177 as a confirmation line due to its weak signal.
- 13.2. This method has a high bias for fertilizer products containing sources of phosphite or organic P compared to gravimetric or colorimetric methods that measure just orthophosphate. ICP-OES measures all ionizable forms of P found in the fertilizer extract. Therefore, it is recommended to use ICP-OES as a primary and gravimetric as a secondary method for confirmation in organic samples.
- 13.3. Both P213 and P214 lines have a potential spectral overlap and interference with Cu. The greatest probability for spectral overlap occurs when the fertilizer Cu concentration is high and the P concentration is low, which is unusual but possible. The situation can largely be corrected by using other wavelengths (P177 and P178).

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 10 of 16

- 13.4. K766 is the strongest line of K covering wide range of concentration from 0-1050µg/mL for Radial and 6-300µg/mL for Axial. K404 Axial is another wavelength which is slightly weaker than K766, and it can be used to cover 60-1050µg/mL K range.
- 13.5. Using multiple wavelengths is encouraged for the result consistency and confirmation, so that an unusual result can be identified and corrected.

## 14. Calculations:

14.1. The calibration standards are prepared as  $\mu$ g/mL for P and K and the final fertilizer results are reported as percentage (%) for P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O. The ICP software automatically calculates for P<sub>2</sub>O<sub>5</sub> and K<sub>2</sub>O when all the sample parameters are entered. It requires the following calculation:

 $P_2O_5 \% = [P \times (250/W) \times 142/(31 \times 2)]/10000$ 

Where P = ICP-OES P reading in  $\mu$ g/mL, 250 = final volume in mL, W = test portion weight in g, 142 = MW of P<sub>2</sub>O<sub>5</sub>, 31 = MW of P, 2 = mole ratio of P<sub>2</sub>O<sub>5</sub>/P, and 10000 = conversion of  $\mu$ g/mL to %.

 $K_2O \% = [K \times (250/W) \times 94.2/(39.1 \times 2)]/10000$ 

Where K = ICP-OES K reading in  $\mu$ g/mL, 250 = final volume in mL, W = test portion weight in g, 94.2 = MW of K<sub>2</sub>O, 39.1 = MW of K, 2 = mole ratio of K<sub>2</sub>O/K, and 10000 = conversion of  $\mu$ g/mL to %.

14.2. In case of pre-drying samples, this calculation is required:

((100-pre-dry%)/100)\* ICP concentration

## **15. Maintenance and Troubleshooting:**

- 15.1. Torch and Injector:
  - 15.1.1. Examine the torch and injector build-up and clean periodically but no later than after 150 samples.
  - 15.1.2. To clean the torch and injector, soak in aqua regia (3:1 HCI: HNO<sub>3</sub>) for at least 4 hours or overnight in a fume hood to remove persistent deposits. Rinse with DI water and dry thoroughly using clean air or nitrogen. Never touch the torch (quartz) with bare hands.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 11 of 16

- 15.2. Nebulizer:
  - 15.2.1. The nebulizer should be replaced as needed, when back pressure rises dramatically, or background and/or IS intensities become suppressed.
  - 15.2.2. To clean the nebulizer, soak in a solution of 5% nitric acid or *aqua regia* for at least 4 hours. If the deposits persist, increase the acid concentration, up to 20%, rinse with DI water, and dry thoroughly using clean air or nitrogen. Do not use sonication or a wire to clean, as they may damage the nebulizer.
- 15.3. Spray Chamber:

The spray chamber contains O-rings which require periodic replacement. If the O-rings appear flattened, cracked or show signs of deposits, they should be replaced or cleaned using mild soap and water.

15.4. Tubing:

Inspect sample and peristaltic pump tubing for cleanliness and flat spots and replace if needed.

- Black/Black for internal standard, 0.76 mm I.D should last 10-16 hours
- Orange/White for sample, 0.64 mm I.D should last 10-16 hours
- Yellow/Yellow for drain, 1.42 mm I.D replace only if notable flat spots or reduction in rinse flow

#### 16. References:

- 16.1. Determination of Phosphorus and Potassium in Commercial Inorganic Fertilizers by Inductively Coupled Plasma-Optical Emission Spectrometry: Single Laboratory Validation. Bartos et al: Journal of AOAC International, Vol. 97, No 3, 2014.
- 16.2. Avio 599, Hardware and Manual Guide. Perkin Elmer, Inc., May 2017.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 12 of 16

Standard ID	Volume mL	Citrate mL	Stock 1 2000 Ρ μg/mL <sup>a</sup>	Stock 2 3000 Κ μg/mLb	Ρ Conc. µg/mL	P₂O₅ µg/mL	P₂O₅ Solution %	P₂O₅ Fertilizer %	K Conc μg/mL.	K2O μg/mL	K2O Solution %	K2O Fertilizer %
Blank	50	20	0	0	0	0	0	0	0	0	0	0
1	50	20	0.05	17.5	2	4.58	0.000458	0.229	1050	1264.2	0.12642	63.21
2	50	20	0.2	12.5	8	18.32	0.001832	0.916	750	903.0	0.09030	45.15
3	50	20	0.5	7.5	20	45.8	0.00458	2.29	450	541.8	0.05418	27.09
4	50	20	1.5	5	60	137.4	0.01374	6.87	300	361.2	0.03612	18.06
5	50	20	3	3	120	274.8	0.02748	13.74	180	216.7	0.02167	10.84
6	50	20	5	1	200	458	0.04580	22.90	60	72.2	0.00722	3.61
7	50	20	7.5	0.4	300	687	0.06870	34.35	24	28.9	0.00289	1.44
8	50	20	10	0.2	400	916	0.09160	45.80	12	14.4	0.00144	0.72
9	50	20	12.5	0.1	500	1145	0.11450	57.25	6	7.2	0.00072	0.36

Table 1. ICP Calibration Standard Preparation

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 13 of 16

Element	Primary Wavelength (nm)	Calibration Range µg/mL	Curve Fit	Detection Mode	Spectral Deconvolution	
Р	177.434	0 to 500	Linear	Radial	None	
Р	177.434	0 to 400	Linear	Axial	None	
Р	178.221	0 to 500	Linear	Radial	None	
Р	178.221	0 to 400	Linear	Axial	None	
Р	213.617	0 to 500	Linear	Radial	Cu 213.598	
Р	213.617	0 to 400	Linear	Axial	Cu 213.598	
Р	214.914	0 to 500	Linear	Radial	Cu 214.898	
Р	214.914	0 to 400	Linear	Axial	Cu 214.898	
K	404.721	60 to 1050	Non-Linear	Axial	None	
К	766.490	6 to 300	Non-Linear	Axial	None	
К	766.490	0 to 1050	Linear	Radial	None	
Sc	361.383					

Table 2. Recommended Wavelengths and Calibration Conditions

# Table 3. PerkinElmer (PE) ICP-OES AVIO 5000 Conditions

Factor	Setting	Factor	Setting
Power	1500 Watts	Spray Chamber	Baffled Cyclonic
Plasma Flow	12 L/min	Nebulizer Type	SeaSpray
Auxiliary Flow	0.2 L/min	Sample Pump Tube	Orange/White
Nebulizer Flow	0.7 L/min	Internal Standard	Black/Black
Sample Flow Rate	1.0 mL/min	Waste Pump Tube	Yellow/Yellow
Source Equilibration Delay	15 sec	CsCl ionic Buffer	0.018M
Delay time	20 sec	Sc Internal Standard	10µg/mL

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 14 of 16

Views	K₂O % 766.49 Radial	P₂O₅ % 177.43 Radial	P₂O₅ % 178.22 Radial	P₂O₅ % 213.62 Radial	P₂O₅ % 214.91 Radial	K₂O % 766.49 Axial	K <sub>2</sub> O % 404.71 Axial	P <sub>2</sub> O <sub>5</sub> % 177.43 Axial	P₂O₅ % 178.22 Axial	P <sub>2</sub> O <sub>5</sub> % 213.62 Axial	P₂O₅ % 214.91 Axial
Mean	0.466	0.207	0.240	0.216	0.234	0.426	-	0.240	0.268	0.177	0.206
SD	0.026	0.021	0.0096	0.0115	0.0088	0.019	-	0.009	0.007	0.009	0.011
MDL	0.083	0.065	0.030	0.036	0.027	0.058	-	0.027	0.023	0.028	0.035
RL	0.083	0.194	0.091	0.108	0.082	0.360	3.62	0.082	0.068	0.084	0.106

Appendix 1. Calculated MDL and RL for all wavelengths in Radial and Axial views

See 13.3 and 13.4 for discussions on preferred wavelengths.

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 15 of 16

Approvals:

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Stacy Aylesworth Senior Environmental Scientist (Supervisor) \_<u>4/16/21</u>

Date

<u>Maryam Khosravífard</u>

Maryam Khosravifard Environmental Program Manager I 4/20/21

Date

\_Sarva Balachandra\_

Sarva Balachandra Quality Assurance Officer <u>5/3/21</u>

Date

RA-SP-P-K-ICP Revision: 0 Revision Date: Original Date: 4/16/21 Page 16 of 16

# Revision Log:

Date	What was Revised? Why?
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