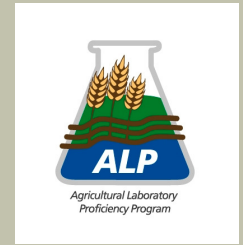


ALP Program Report



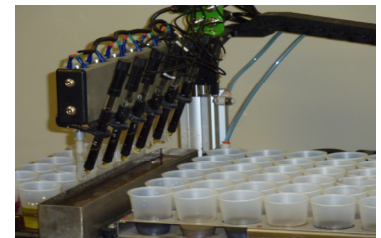
Robert O. Miller, PhD, ALP Technical Director, Windsor, CO
Christopher Czuryca, Collaborative Testing Services, Inc, Sterling, VA

ALP Overview

Special points of interest:

- An assessment homogeneity of all ALP proficiency materials indicate were highly uniform for Cycle 54.
- Sixty-four laboratories provided soil pH (1:1) H₂O results, medians ranged from 5.77 - 7.95.
- Soil M3-P ICP for Cycle 54 ranged from 21 to 387 mg kg⁻¹ with intra-lab stdev MAD values ranging 1.0 - 11 mg kg⁻¹ across the five soils.
- Soil M3-K values ranged from 41 - 500 mg kg⁻¹ for the five ALP soils of PT Cycle 54.
- Results for soil NO₃-N indicate a wide range in concentrations with SRS-2409, having a median concentration of 0.6 mg kg⁻¹
- Botanical N by combustion was reported by 41 labs, with five labs showing high bias across all PT materials for Cycle 54.

The Agriculture Laboratory Proficiency (ALP) Program spring 2024 Round Cycle 54 was completed May 20, 2024, with results from one-hundred sixteen labs enrolled from the US, Canada, South Africa, Italy, Guatemala and Philippines. Proficiency samples consisted of five soils, four botanical and three water samples. Analytical methods are base on those published by AOAC, regional soil work groups, the Soil Plant Analysis Council and Forestry Canada. ALP has completed nineteen years of service to Ag laboratory industry.



Data was compiled for each method (test code) and proficiency material. Data analysis of each material include: the number results; grand median value; median absolute deviation (MAD), 95% Confidence Interval; method intra-lab standard deviation (s); lab mean, and standard deviation. Additional information on methods and statistical protocols can be found at the program web site.

Proficiency Materials

Standard Reference Soils (SRS) materials utilized for Cycle 54 were: SRS-2406 Gazwell mucky clay, collected Solano Cty, CA; SRS-2407 collected near Magrath, AB Canada; SRS-2408 a Plainfield sand collected from Wood Cty, WI; SRS-2409 is Nolin silt loam collected Caldwell Cty, KY; and SRS-2410 a Hinckley loamy sand collected Hampshire MA. Chemical properties of the SRS materials ranges: pH (1:1) H₂O 5.77 - 7.95; SMP Buf 6.21 - 7.45 mg kg⁻¹; Bray P1 (1:10) 15.0 - 303 mg kg⁻¹; SO4-S 2.7 - 59.4 mg kg⁻¹; M3-K 41 - 499 mg kg⁻¹; M3-Ca 826 - 8366 mg kg⁻¹; DTPA-Zn 0.55 - 3.30 mg kg⁻¹; SOM-LOI 2.37 - 18.9%; CEC 6.1 - 55.5 cmol kg⁻¹; clay 5.0 - 46.2% and NO₃-N 0.65 - 116 mg kg⁻¹.

Standard Reference Botanical (SRB) materials for Cycle 54 were: SRB-2405 tomato leaf composite from CA; SRB-2406 potato petiole composite from WA; SRB-2407 forage mix composite from IA and SRB-2408 citrus leaf composite from CA. SRB median analytes concentrations: NO₃-N 182 - 18,100 mg kg⁻¹; Dumas N 2.32 - 4.18 %; wet digestion total P 0.150 - 0.383 %; total K 1.26 - 9.63 %; total Ca 0.39 - 3.64 %; total S 0.22 - 0.83 %, total B 4.3 - 74.1 mg kg⁻¹; and Zn 24.1 - 56.9 mg kg⁻¹.

Standard Reference Water (SRW) samples represent an agriculture water samples collected: SRW-2404 a water sample collected from an irrigation canal Neligh, NE; SRW-2405 collected irrigation supply canal western Colorado, and SRW-2406 from a domestic well in, southern Indiana. SRW median concentrations: pH 7.95 - 8.15; EC 0.29 - 1.47 dSm⁻¹; SAR 0.42 - 1.77; Ca 1.95 - 10.2 mmolc L⁻¹; Na 0.46 - 2.27 mmolc L⁻¹; HCO₃ 2.15 - 6.62 mmolc L⁻¹; and NO₃ 0.008 - 0.11 mmolc L⁻¹.

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Homogeneity Evaluations



“..soil pH, Buf pH A&E, Olsen P and SOM-WB analysis Stdev values for Cycle 54 met homogeneity standards.”

SRS material homogeneity was evaluated based on soil test codes pH (1:1) H₂O, buffer pH Adams Evans, EC (1:1), P Olsen, K Olsen, NO₃-N, SOM-WB and DTPA-Zn on analysis of six jars of each PT soil, each in analyzed in triplicate by an independent laboratory. Homogeneity results were within acceptable limits for all soils, with the lowest noted for pH H₂O, Table 1. Proficiency soil antecedent moisture contents were: 13.2 ± 0.2%, 8.1 ± 0.1%, 2.1 ± 0.1%, 3.6 ± 0.1% and 1.5 ± 0.1%, respectively.

Homogeneity was also evaluated on SRB and SRW matrix samples. Botanical results indicate all samples were well homogenized for N, P, Zn and C, with RSD values less than 0.5% of N and C on three of botanical samples, Table 2. Water results for EC, Ca, NO₃ and NH₄ were well homogenized with RSD values for EC less than 1% for all three of the PT samples, Table 3.

Table 1. ALP soils homogeneity evaluation Cycle 54, 2024.

Sample	pH (1:1) H ₂ O		EC (dS m ⁻¹)		P Olsen (mg kg ⁻¹)		SOM-WB (%)	
	Mean ¹	Std	Mean	Std	Mean	Std	Mean	Std
SRS-2406	6.05	0.01	0.75	0.012	21.9	1.0	21.03	0.48
SRS-2407	8.04	0.02	0.45	0.007	19.7	1.1	4.03	0.23
SRS-2408	5.69	0.03	0.04	0.002	6.7	0.55	3.04	0.10
SRS-2409	7.16	0.01	0.24	0.006	6.0	0.48	3.33	0.08
SRS-2410	6.39	0.02	0.96	0.020	39.3	1.4	2.35	0.12

¹ Statistics based on five randomly selected soil replicates, each analyzed in triplicate.

Table 2. ALP botanical homogeneity evaluation Cycle 54, 2024.

Sample	N (%)		P (%)		Zn (mg kg ⁻¹)		C (%)	
	Mean ¹	Std	Mean	Std	Mean	Std	Mean	Std
SRB-2405	4.25	0.012	0.33	0.01	29.3	0.9	38.58	0.09
SRB-2406	3.95	0.010	0.35	0.01	32.7	1.2	32.32	0.04
SRB-2407	3.53	0.026	0.37	0.01	23.8	0.3	42.30	0.03
SRB-2408	2.41	0.037	0.14	0.01	52.6	1.1	42.73	0.16

¹ Statistics based on three randomly selected botanical replicates analyzed.

Table 3. ALP water homogeneity evaluation Cycle 54, 2024.

Sample	EC (dS m ⁻¹)		Ca (meq L ⁻¹)		NO ₃ (meq L ⁻¹)		NH ₄ (meq L ⁻¹)	
	Mean ¹	Std	Mean	Std	Mean	Std	Mean	Std
SRW-2404	0.261	0.003	40.0	0.75	133.6	9.3	0.67	0.51
SRW-2405	1.022	0.009	149.7	2.12	127.3	19.4	3.70	0.67
SRW-2406	0.521	0.003	45.4	0.85	107.7	26.5	0.49	0.69

¹ Statistics based on three randomly selected soil replicates, each analyzed in triplicate.

SRS - pH (1:1)_{H2O}

Sixty-five laboratories provided ALP results for soil pH (1:1) H₂O (test code 116). Soils ranged from acid to alkaline, median range 5.77 - 7.95. Lab results were ranked low to high based on sample SRS-2408 (see Figure 1) with median pH designated by horizontal lines for each soil. Generally across labs all soils showed good consistency across labs. Labs #3 through #5 showed low bias across all five soils. Labs #9, #42, #55 and #65 were inconsistent across the five soils for cycle 54. Source of bias is likely associated with ISE performance and/or method compliance. Inconsistency could be result of extract carry-over.

pH precision across the five ALP soils indicates very high precision, with median intra-lab standard deviation (*s*) values ranging from 0.017 to 0.030 pH units, the lowest noted for SRS-2306. Eight labs had poor precisions, with standard deviations exceeding consensus median intra-lab *s*. Specifically *s* for labs #3, #5, #14, #38, #60, and #62 exceeded 0.08 pH units for SRS-2406. Soil SRS-2408 was the least variable with respect to intra-lab variance.

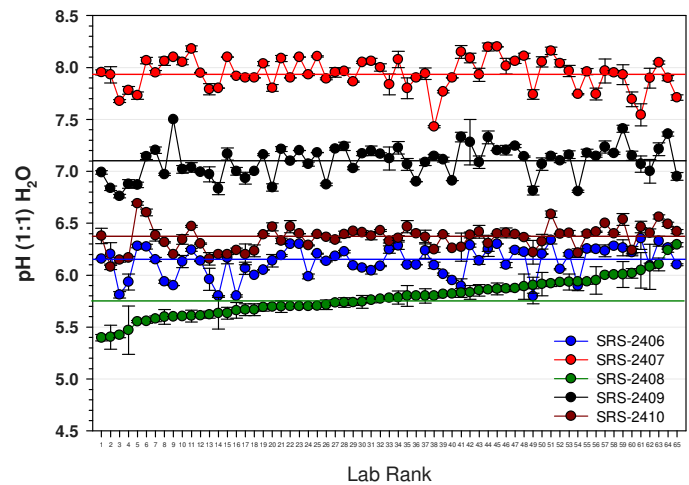


Figure 1. pH (1:1) H₂O distribution plots for SRS materials, ALP 2024 Cycle 54.

SRS - Phosphorus: Bray P1, M3-P, Olson P

Bray P1 results were reported by twenty-eight labs. Olsen-P was reported by 49 labs and M3-P ICP was reported by 46 labs. Median soil Bray P1 values ranged from 15 - 302 mg kg⁻¹ PO₄-P; Olsen P 6.5 to 37 mg kg⁻¹ P; Bray P2 ranged from 28 to 443 mg kg⁻¹ P; and M1-P from 3.4 to 242 mg kg⁻¹ P, across the five soils. Ranking lab results based on sample SRS-2408, median Bray P1 concentrations are shown in indicated in Figure 2. Soil SRS-2406, moderate in concentration was variable between labs. Soils SRS-2406 and SRS-2407 had near identical concentrations of approximately 30 mg kg⁻¹ P. Lab #6, #20 and #24 had in consistent results across all five soils. Lab #28 had Bray P1 high bias for all five soils.

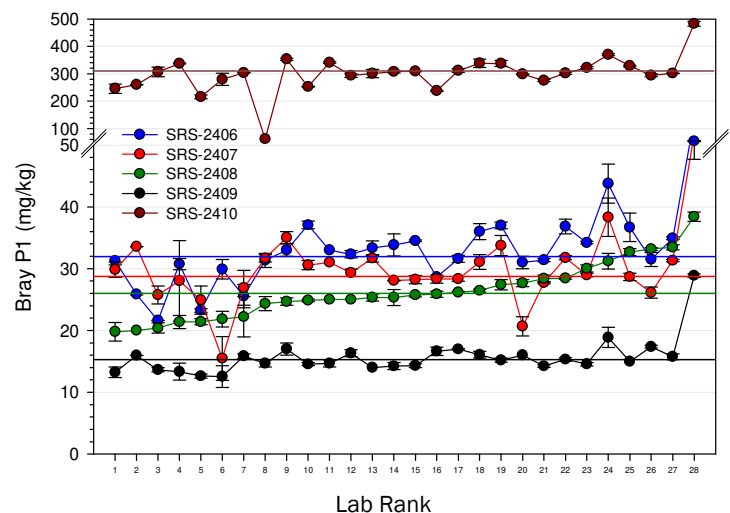


Figure 2. Bray P1 distribution plots for SRS materials, ALP 2024 Cycle 54.

Nine labs reported M3-P Spec median concentrations ranging 17.6 - 344 mg kg⁻¹ P. Thirteen laboratories reported Bray P2 with medians ranging from 29.7 - 443 mg kg⁻¹ PO₄-P. Six labs reported M1-P results and one results for Modified Morgan P, with medians ranging from 1.9 - 20.5 mg kg⁻¹ PO₄-P. Modified Kelowna was reported by two laboratories ranging from 11.3 - 193 mg kg⁻¹ P and total P (US-EPA 513) ranged 243 - 913 mg kg⁻¹ P with the highest concentration noted for SRS-2410.

SRS - Potassium

Forty-four laboratories provided ALP results for soil M-3 K (test code 159) results. Results were ranked low to high based on sample SRS-2408 (see Figure 3). Soil SRS-2406 and SRS-2409 were the most inconsistent across labs. The source of the variability is for 2406 is likely associated with high SOM. Lab #1 showed low bias on all soils. Across all soils labs #14, #27 and #42 were inconsistent across the five soils for M3-K. Source of inconsistency is likely related to extraction, analysis instrument and/or method compliance.

M3-K intra-lab *s* values were lowest for soil SRS-2408, with a median intra-lab value of 3.0 mg kg⁻¹ K and highest for SRS-2407 with a value of 12.6 mg kg⁻¹. M3-K within-lab precision across the ALP soil materials indicates very good precision, generally, for soils with less than 200 mg kg⁻¹ K. Precision was poor (based on intra-lab *s*) for three labs which exceeded 10 mg kg⁻¹ K on SRS-2406. Across the five soils for Cycle 54 eight labs were flagged for poor precision. Poor precision is attributed to extraction and/or analysis instrument operation.

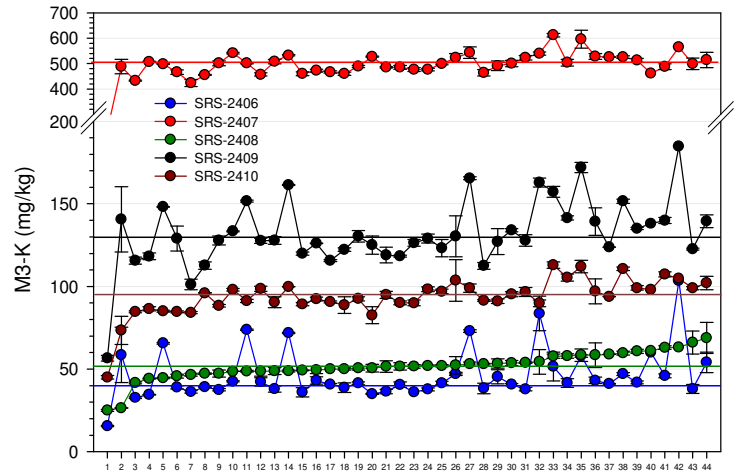


Figure 3. Extractable M3-K distribution plots for SRS materials, ALP 2024 Cycle 54.

SRS - SOM-LOI

Forty-seven laboratories provided ALP results for soil SOM-LOI (test code 183). Soil Median SOM-LOI values ranged from 2.36 to 18.86%. Results were ranked based on sample SRS-2408 (see Figure 4) and had high consistency. Lab #26 had consistent low bias across all soils. Labs #1, #16, and #18 were inconsistent across the five soils. Source of bias is likely related to muffle furnace operation and/or method compliance.

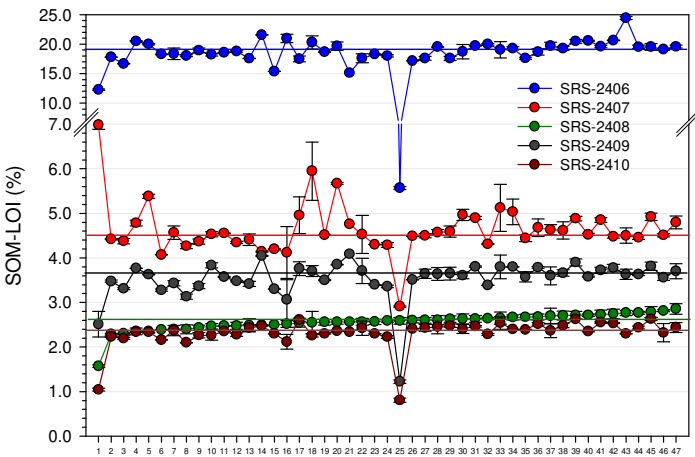


Figure 4. SOM-LOI distribution plots for SRS materials, ALP 2024 Cycle 54.

SOM-LOI precision across the five soils indicates high median intra-lab precision *s* values ranging from 0.074 to 0.47% SOM-LOI, highest for SRS-2406. Across labs, *s* values for SRS-2408 ranged from 0.002 - 0.25%. Across soils low precision was noted for several laboratories. Specifically *s* for labs #13, #18, #30, #37, #42, #45 and #47 exceeded 0.10% SOM-LOI for SRS-2408. Lab #16 had poor precision on multiple samples. Poor precision may be associated with the muffle furnace and heating time.

ALP Soil NO₃-N

Fifty-five laboratories provided ALP results for soil extractable NO₃-N (test code 127) results. Results were ranked low to high based on sample SRS-2407 (see Figure 5). Soil SRS-2409 was the lowest in concentration, lowest of all samples tested in the ALP program since 2006. Soil SRS-2405 was the most inconsistent across labs. Laboratory #1 showed low bias on all soils. Across all soils labs #40, #53 and #54 were inconsistent across three of five soils and the source likely related to extraction, analysis instrument and/or method compliance.

Soil NO₃-N intra-lab *s* values were lowest for soil SRS-2409, with a median intra-lab value of 0.3 mg kg⁻¹ and highest for SRS-2410 with a value of 5.6 mg kg⁻¹. Soil NO₃-N within-lab precision across the ALP soil materials indicates very good precision, generally, for soils with less than 30 mg kg⁻¹. Precision was poor (based on intra-lab *s*) for three labs which exceeded 10 mg kg⁻¹ NO₃-N on SRS-2406. Across the five soils for Cycle 54 fifteen labs were flagged for poor precision.

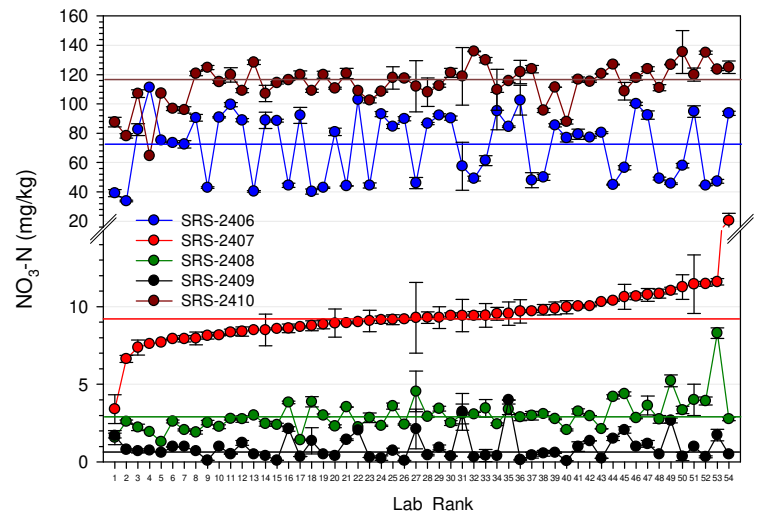


Figure 5. Soil NO₃-N distribution plots for SRS materials, ALP 2024 Cycle 54.

SRB - NO₃-N

Twenty-nine laboratories provided ALP results for NO₃-N by cadmium reduction and ISE (test codes 202, 203 and 204). Median values are designated by horizontal lines for each of the four botanical materials labs based on sample SRB-2408 (see Figure 6). Lab #1 had low bias results for all four materials. The data plot shows labs #2, #5, #11 and #29 were inconsistent on two of four samples.

Botanical NO₃-N (test code 202) results for Cycle 54 indicate very high precision, with intra-lab median standard deviation (*s*) values ranging from 31 to 1080 mg kg⁻¹ across the four samples. NO₃-N (test code 202) intra-lab *s* values for SRB-2405 ranged from 5 - 270 mg kg⁻¹; SRB-2406 ranged from 25 - 4200 mg kg⁻¹, SRB-2407 ranged from 2 - 554 mg kg⁻¹ and SRB-2408 ranged from 2 - 122 mg kg⁻¹. Labs #4, #11, and #12 had consistently high standard deviations for samples SRB-2405 and SRB-2406 highest NO₃-N content. Four of 28 labs were flagged for poor precision across the four samples.

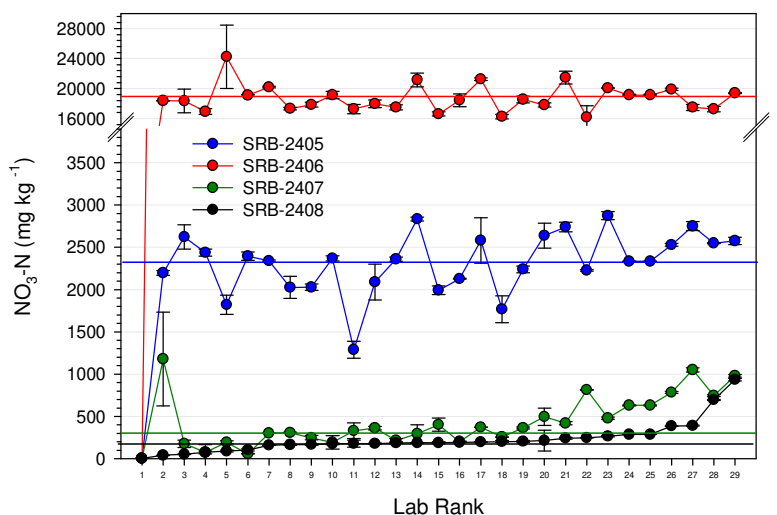


Figure 6. Nitrate distribution plots for SRB materials, ALP 2024, Cycle 54.

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SRB - Dumas Nitrogen and TKN

Forty-one laboratories provided ALP results for botanical Dumas (Combustion) Nitrogen (test code 210) and ten labs for TKN (Test code 209) for Cycle 54. Median values are designated by horizontal lines for each material and labs results ranked low to high based on sample SRB-2408 (see Figure 7). Lab #1 had consistently low bias and labs #37 - #41 had consistent high bias for Dumas N. TKN results were very inconsistent across all four samples.

Dumas N results indicate very high intra-lab precision across all labs for all samples. Intra-lab median N lab *s* values were 0.086% N for SRB-2405; 0.073 % N for SRB-2406; 0.071% for SRB-2407; and 0.067% for SRB-2408. Lab #27, #28, and #41 had consistently high standard deviations on three of four PT samples. TKN median intra-lab *s* values for SRB-2405 were 0.051%, SRB-2406 0.061%, SRB-2407 0.010% and SRB-2408 0.115% TKN nitrogen, respectively.

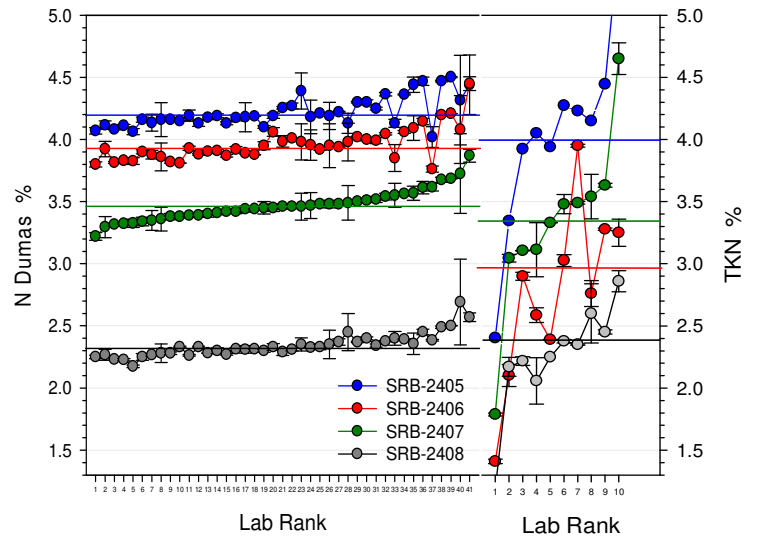


Figure 7. N distribution lab plots for SRB materials, ALP 2024 Cycle 54.

SRB - Phosphorus

Fifty laboratories provided ALP results for Cycle 54 phosphorus (P) (test code 212). Botanical results median values are designated by horizontal lines for each botanical material and labs results are ranked low to high based on sample SRB-2408 (see Figure 8). Labs #2, #45, #48, and #49 showed inconsistency. Source of inconsistency is likely related to sample digestion, analysis instrument and/or test code method compliance.

Botanical P results indicate very high precision, with median intra-lab standard deviation (*s*) values ranged 0.005 to 0.013 % P for test code 212 across the four botanical samples. Individual lab intra-lab *s* values for SRB-2405; ranged from 0.001 - 0.034% P; SRB-2406 ranged from 0.001 - 0.067 % P and SRB-2407 0.001 - 0.153 % P; and SRB-2408 0.001 - 0.044 % P. Eight labs were flagged for poor precision for botanical P for Cycle 54.

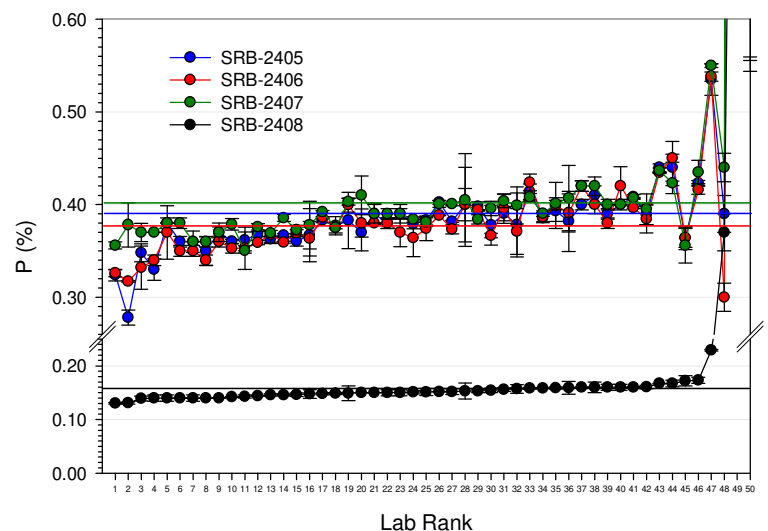


Figure 8. Phosphorus distribution lab plot for SRB materials, ALP 2024 Cycle 54.

SRB - Potassium

Fifty laboratories provided ALP results for potassium (K) (test code 213). Median values are designated by horizontal lines for each botanical material and labs results are ranked low to high based on sample SRB-2405 (see Figure 9). Labs #1, #9, #13 and #27 were inconsistent. Source of bias is related sample digestion, analysis instrument and/or method compliance.

Botanical K results indicate very high precision, with intra-lab median standard deviation (*s*) values ranging from 0.088 to 0.403 % K for test code 213 across the four samples. Individual lab intra-lab *s* values were: SRB-2405, ranged from 0.004 - 0.333 % K; SRB-2406, 0.010 - 1.49 % K; SRB-2407, 0.006 - 1.06 % K; and SRS-2408, 0.001 - 0.613 % K. Lab #50 had high standard deviations exceeding 0.20 % K on two of four samples. Across samples five labs were flagged for poor K precision for Cycle 54.

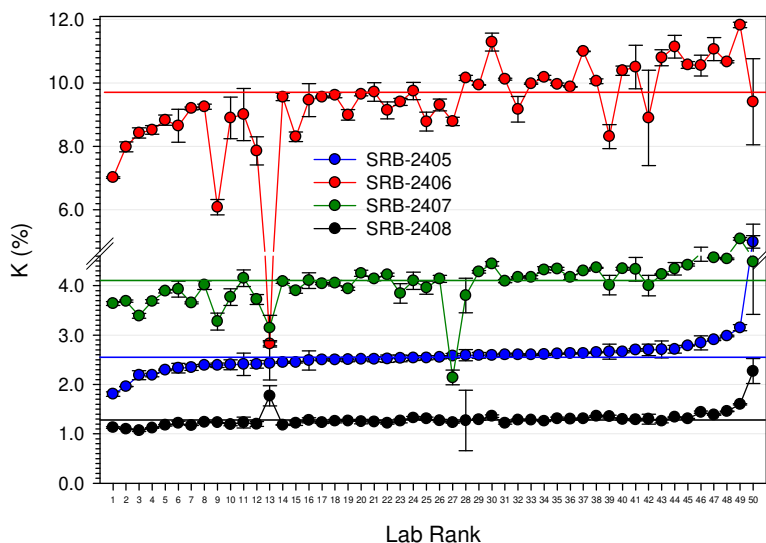


Figure 9. Potassium lab plot for SRB materials, ALP 2024 Cycle 54.

SRB - Magnesium

Forty-eight laboratories provided ALP results for Magnesium (Mg) (test code 215). Result median values are designated by horizontal lines for each botanical material and individual labs results are ranked low to high based on sample SRB-2407 (see Figure 10). Across samples labs #1 had low bias on all samples. Labs #10, and #44 were inconsistent. Source of bias is likely related sample digestion, analysis instrument and/or method compliance.

Botanical Mg results indicate very high precision, with median intra-lab standard deviation (*s*) values ranged from 0.006 to 0.029 % Mg for across the four botanical proficiency samples. Individual lab intra-lab *s* values for SRB-2405; ranged from 0.001 - 0.131 % Mg; SRB-2406 ranged from 0.002 - 0.077 % Mg; SRB-2407 0.001 - 0.016 % Mg; and SRB-2408 0.001 - 0.055 % Mg. Lab #10 had consistently high standard deviations for three samples.

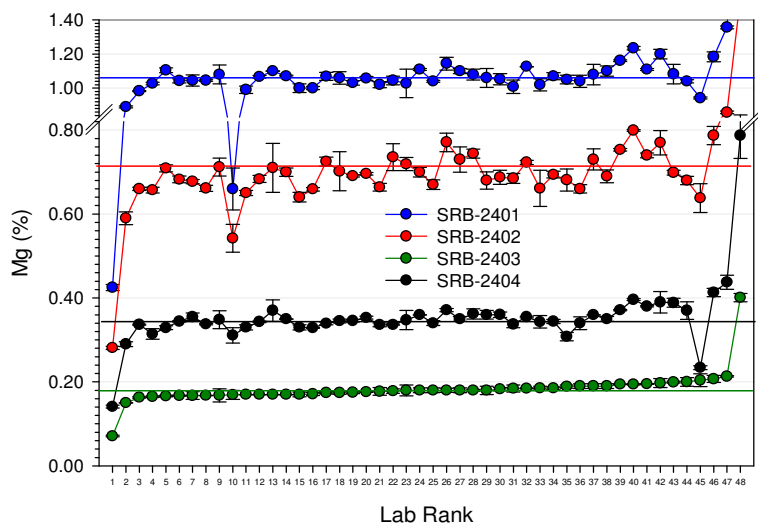
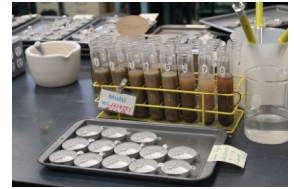


Figure 10. Magnesium (code 215) lab plots for SRB materials, ALP 2024 Cycle 54.

SRW - Water EC

Twenty-one laboratories provided ALP results for water pH (test code 302) and EC. Lab results were ranked low to high based on sample SRW-2404 (see Figure 11). Sample SRW-2404 had the highest EC in for Cycle 54. Labs #1 and #19 indicated inconsistency across samples. Source of bias is likely associated with EC probe performance and/or calibration.



EC precision across the three water materials indicates very high precision, with intra-lab median s values of 0.006, 0.020 and 0.009 dSm^{-1} , for the three water samples, respectively. Precision for sample SRW-2404 was the most consistent across the nineteen participating laboratories. Intra-lab s values for lab #4 exceeded 0.042 dSm^{-1} on SRW-2405. Highest precision was noted for lab #21 with intra-lab s values of < than 0.004 dSm^{-1} for all three samples. Two labs were flagged for precision for EC.

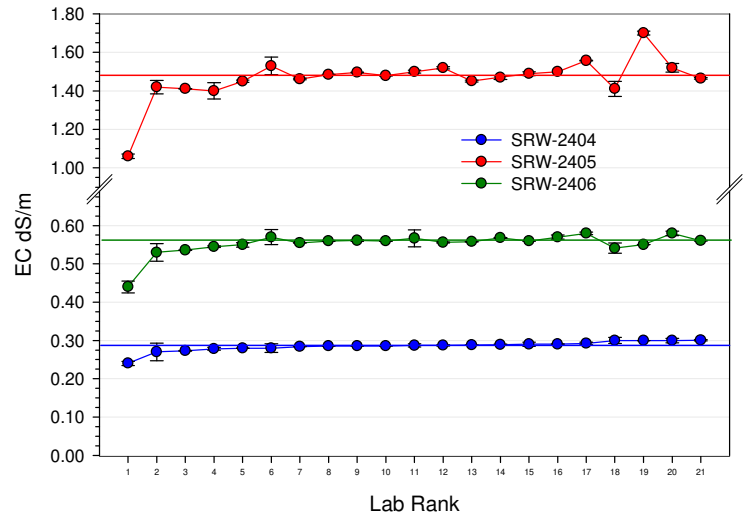


Figure 11. Water EC distribution plots for SRW materials, ALP 2024 Cycle 54.

SRW - Mg

Twenty laboratories provided ALP results for water Mg (test code 304). Lab results were ranked low to high based on sample SRW-2404 (see Figure 12). Median values are designated by horizontal lines. Lab #1 showed consistent low bias on all two samples, and is likely a result of a calibration error.

Magnesium precision across the three water solution matrices indicates excellent precision, with intra-lab s values of 0.024, 0.206, and 0.043 mmolc L^{-1} for SRW-2404, SRW-2405, and for SRW-2406, respectively. Water Mg precision was excellent for all individual labs with only labs #4, #11, and #15 exceeding 0.03 mmolc L^{-1} Mg on sample SRW-2406. One lab was flagged for poor precision on MG for ALP Cycle 54.

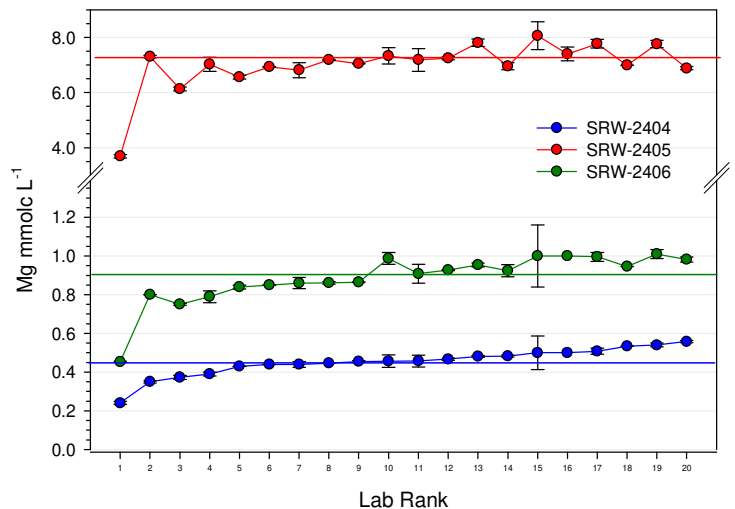


Figure 12. Water Mg distribution plots for SRW materials, ALP 2024 Cycle 54.

Announcements

- ▶ The ALP Program welcomes Kassidy Taylor, program operations specialist at Collaborative Testing Services in Sterling, Virginia. Kassidy, oversees data management, statistical analysis and proficiency reports.
- ▶ The ALP program soil carbon proficiency program will initiate in January 2025. Test parameter include, total Soil Carbon (SC), soil organic carbon (SOC), soil inorganic carbon (SIC), pH and moisture content. The program will consist three cycles per year each with four soils. The soil carbon proficiency program will be required for labs seeking ALTA-SAC certification.
- ▶ The 18th International Symposium on Soil and Plant Analyses (ISPPA) will be held in June 9-13, 2025 in Durham, North Carolina. Topics include soil, plant and water analysis, soil health, proximal sensors and plant nutrition. More information and enrollment can found at: <https://www.issposymposium.org/>
- ▶ ALTA has established a additional proficiency program, Enhanced Quality Assurance (EQA) program. EQA is based on double blind testing of lab participants using ALP standard reference soils. For mor information visit ALTA.Ag .
- ▶ ALP has standard reference soils and plant tissue samples available for purchase. For more information on these contact the ALP Technical Director, Robert.Miller@cts-interlab.com.

Summary

ALP is has provided nineteen years of service with the completion of Cycle 54. Since 2005 ALP has completed the analysis of 270 soils, 188 plant samples and 174 water samples providing comprehensive proficiency data on inter and intra laboratory performance across a range of analytical methods.

We thank all laboratories who participated in Cycle 54. As the coordinators of the program we appreciate your consideration and participation in the proficiency program. We continually seek feedback from laboratory participants to improve the service and function of the program. Please forward all comments to info@cts-interlab.com.

Cycle 55 Ship
September 20, 2024

**“Yesterday’s the past, tomorrows
the future, but today is a gift. ”**

– Bil Keane, 1984

