2020 Volume 1

24, May 2020

ALP Program Report

2020 Spring - Cycle 41

Robert O. Miller, PhD, Colorado State University. Fort Collins, CO
Christopher Czyryca, Collaborative Testing, Inc, Sterling, VA

ALP Overview

The Agriculture Laboratory Proficiency (ALP) Program fall 2020 Round Cycle 41 was completed May 24, 2020, with results from one-hundred seven labs enrolled from the US, Canada, South Africa, Italy, Ukraine, 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 fifteen 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.

Special points of interest:

- Soil homogeneity assessment indicate ALP reference soil materials were highly uniform for Cycle 41.
- Fifty-four Laboratories provided soil pH (1:1) H_2O results and medians ranged from 5.19 - 7.95.
- Cycle 41 soil M3-P ICP ranged from 4.4 to 109 mg kg^{-1} with MAD values ranging 0.8 - 8.6 mg kg^{-1} across the five soils.
- NH4oAc-3 K values ranged from 79 - 462 mg kg^{-1} for the five ALP soils of PT Cycle 41.
- Sikora Buffer pH values showed high consistency across 24 of 28 testing labs for PT Cycle 41.
- Botanical K, ranged from 2.12 - 8.78% with four of forty-one labs noted for inconsistency.
- Botanical B results showed high consistency across the thirty of thirty-three labs for PT Cycle 41.
- Water Na content showed very high consistency by sixteen of nineteen labs across all PT samples.

Proficiency Materials

Standard Reference Soils (SRS) materials utilized for Cycle 41 were: SRS-2001 a Seaton silt loam, collected Trempaleau Cty, WI; SRS-2002 Cibola silty clay loam collected Riverside Cty; SRS-2003 a Lloyd clay loam collected Rowan Cty, NC; SRS-2004 is a loam collected near Lethbridge, Alberta, Canada; and SRS-2005 a Paxton fine sandy loam collected Kennebec county, ME. Chemical properties of the SRS materials ranges: pH (1:1) H_2O 5.19 - 7.95; NO_3-N 11.2 - 130 mg kg^{-1}; Bray P1 (1:10) 4.2 - 69.3 mg kg^{-1}; M3-K 77 - 467 mg kg^{-1}; SO_4-S 5.8 - 106 mg kg^{-1}; DTPA-Zn 0.50 - 1.26 mg kg^{-1}; SOM-LOI 1.30 – 6.29%; CEC 10.1 - 38.1 cmol kg^{-1}; clay 12.1 - 38.4% and soil available H_2O 9.8 - 16.3 %.

Standard Reference Botanical (SRB) materials for Cycle 41 were: SRB-2001 a sudan grass composite from Riverside, Cty, CA; SRB-2002 leaf composite from NE; SRB-2003 grape leaf composite from WA; and SRB-2004 jalapeno pepper composite from IA. SRB median analytes ranged: NO_3-N 208 - 16200 mg kg^{-1}; Dumas N 0.931 - 4.08%; total P 0.20 - 0.41%; total K 2.12 - 8.78%; total Mg 0.18 - 0.63%; total S 0.11 - 0.21 %, total Cu 7.6 - 30.0 mg kg^{-1}; and total Pb 0.06 - 0.33 mg kg^{-1}.

Standard Reference Water (SRW) samples represent an agriculture water samples collected: SRW-2001 a water sample collected from a domestic well Gretna, NE; SRW-2002 was collected from a Colorado River canal near Blythe, CA; and SRW-2003 from river in central WA. SRW median concentrations ranged: pH 7.31 - 8.27; EC 0.03 - 0.99 dSm^{-1}; SAR 0.14 - 2.4; Ca 0.21 - 4.19 mmolc L^{-1}; Na 0.06 - 4.27 mmolc L^{-1}; HCO_3 0.30 - 4.0 mmolc L^{-1}; and NO_3 0.01 - 1.89 mmolc L^{-1}.

Inside this issue:

Soil Homogeneity Evaluation

SRS material homogeneity was evaluated based on soil test codes pH (1:1) H_2O, pH Adams Evans, EC (1:1), P Olsen, K Olsen, NO_3-N and SOM-WB on analysis of six jars, each in analyzed in triplicate by an independent laboratory. Homogeneity results were within acceptable limits for all soils, with the lowest noted for pH H_2O. Homogeneity was also evaluated on SRB and SRW matrix samples.

"..soil pH, EC and Olsen P analysis Stdev values for Cycle 41 met homogeneity standards."

Table 1. ALP soils homogeneity evaluation Cycle 41, 2020.

Sample	pH (1:1) H_2O		EC (1:1) (dSm^{-1})		Olsen P ($mg\ kg^{-1}$)		SOM-WB (%)	
	Mean [1]	*Std*	Mean	*Std*	Mean	*Std*	Mean	*Std*
SRS-2001	5.14	*0.02*	1.02	*0.02*	7.4	*0.4*	2.19	*0.07*
SRS-2002	7.87	*0.03*	1.65	*0.03*	19.2	*1.4*	1.21	*0.05*
SRS-2003	6.01	*0.02*	0.24	*0.02*	3.3	*1.0*	1.76	*0.03*
SRS-2004	7.90	*0.02*	0.42	*0.03*	5.4	*0.7*	4.95	*0.13*
SRS-2005	6.53	*0.02*	0.85	*0.03*	18.3	*1.0*	5.40	*0.13*

[1] Statistics based on six soil replicates, each analyzed in triplicate ALP Cycle 41.

2020 Cycle 41 Observations

Results for soil pH (1:1) H_2O (test code 115) analysis MAD values for Cycle 41 averaged 0.07 pH units across the soils. Median within lab pH standard deviation was 0.048 pH units. Soil displacement CEC ranged 10.1 to 19.1 cmol kg^{-1} across the five soils. Sample SRS-2002 had a large discrepancy in soil CEC values: displacement 12.3 cmol kg^{-1} and estimated CEC of 32.5 cmol kg^{-1}. SRS-2001 had an abnormally low extractable Cl of 3.6 mg kg^{-1}, likely associated with Seaton silt loam soil series. Soil ammonium acetate K (Test code 140) MAD values ranged 4.9 - 35.9 mg kg^{-1} and ammonium acetate Mg MAD values ranged 23.1 to 37.2 mg kg^{-1} for the five soils. These results for K and Mg were consistent with past cycles in 2020 and are attributed to: (1) improved lab consistency; (2) soils generally higher in potassium; and (3) ICP operation.

Across the four botanical samples Dumas combustion N MAD values averaged 0.061% nitrogen with intra-lab s of 0.036%, 0.031%, 0.017% and 0.034%, respectively. Botanical sample SRB-2004 had a very low median Mn with a concentration of 16.3 ppm and with a MAD of 1.2 ppm. Generally the jalapeno composite sample SRB-2004 had lower median concentrations of NO3-N, Cl, Ca, Mg, Al, Mn, Fe and Cu relative to the other three botanical samples. One observation on Cycle 41, intra-lab relative variability was lowest for N other macro elements for all four botanical samples.

Water EC results showed high consistency across samples. Across the three water samples EC Median values ranged from 0.746, 1.00 and 0.034 dSm^{-1}, respectively. Cl values ranged from 0.048 - 2.64 molc L^{-1} across the three water samples with MAD values ranging 0.029 to 0.101 molc L^{-1}. Sample SRW-2002 had and SAR of 2.45 with a MAD of 0.03.

SRS - pH (1:1)H2O

Fifty-five laboratories provided ALP results for soil pH (1:1) H_2O (test code 115). Soils ranged from acid to alkaline, median range 5.19 - 7.95. Lab results were ranked low to high based on sample SRS-2001 (see Figure 1) with median pH designated by horizontal lines for each soil. Generally soils SRS-2004, and SRS-2005 showed good consistency across labs. Labs #1, #19, #40. #54 and #55 were inconsistent across soils. Source of bias is likely associated with ISE performance and/or method compliance. Inconsistency could be result of extract carry-over.

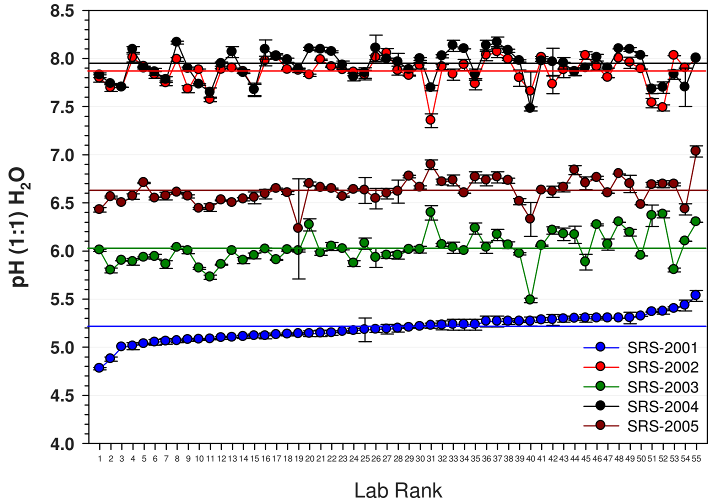


Figure 1. pH (1:1) H_2O distribution plots for SRS materials, ALP 2020 Cycle 41.

pH precision across the five ALP soils indicates very high precision, with median intra-lab standard deviation (s) values ranging from 0.036 to 0.087 pH units, the lowest noted for SRS-2001. For specific labs poor precision was noted for seven laboratories, exceeding by three times that noted for consensus median intra-lab s. Specifically s for labs #19, #25, #26, #28 and #40 exceeded 0.10 pH units for SRS-2005. Soil SRS-2001 was the least variable with respect to intra-lab variance for Cycle 41.

SRS - Phosphorus: Bray P1, Bray P2, Olsen, Modified Morgan, M1, and M3

Bray P1 results were reported by thirty-one labs. M3-P ICP was reported by 35 labs. Median soil Bray P1 values ranged from 3.7 - 70.3 mg kg^{-1} PO_4-P; Olsen P 3.5 to 20.4 mg kg^{-1} P and Bray P2 ranged from 6.9 to 266 mg kg^{-1} P, across the five soils. Ranking lab results based on sample SRS-2004, median M3-P ICP concentrations are shown in indicated in Figure 2. A saw tooth trend was noted for soils SRS-2001 and SRS-2004 associated with the moderate P concentrations. Soil SRS-2003, lowest in concentration, showed low intra-lab variability. Lab #1 showed low bias on all three of five samples. Labs #4, #19, #34 and #35 were inconsistent. Inconsistency is likely related to extraction, analysis instrument and/or method compliance.

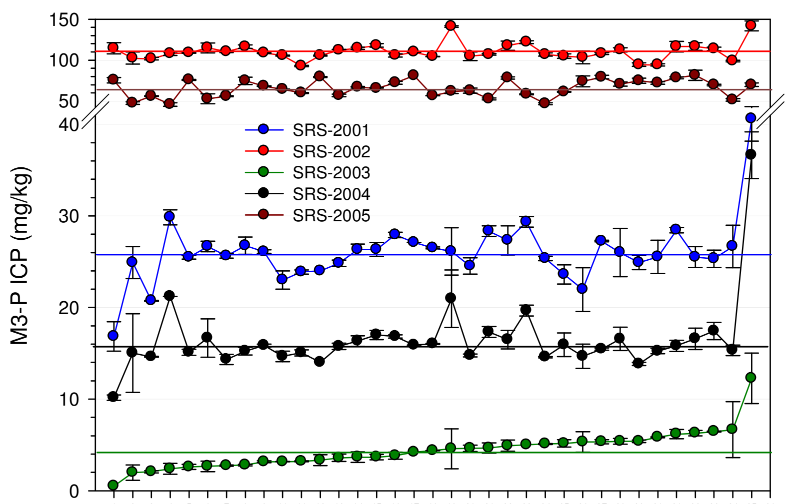


Figure 2. M3-P ICP distribution plots for SRS materials, ALP 2020 Cycle 41.

Four laboratories provided ALP results for Mehlich 1 P, with medians ranging from 2.6 to 87.6 mg kg^{-1} PO_4-P. M3-P Spec median concentrations were 4.5 - 100.1 mg kg^{-1} P reported by eight labs. Modified Kewolna was reported by two laboratories ranging from 2.9 - 45.8 mg kg^{-1} P and total P (US-EPA503 ranged 326 - 900 mg kg^{-1} P with the highest concentration noted for SRS-2005.

SRS - Potassium

Fifty laboratories provided ALP results for soil K (test code 141) results. Results were ranked low to high based on sample SRS-2001 (see Figure 3). Soils SRS-2002 and SRS-2005 were the most inconsistent across labs. Lab #1 had low bias on four of five soils. Labs #3, #7, #8, #14, #27, #31, #44 and #49 were inconsistent across the five soils for K. It is worth noting twenty-four labs found near identical K concentrations for SRS-2001 and SRS-2005. Source of inconsistency is likely related to sample extraction, analysis instrument and/or method compliance.

Potassium intra-lab *s* values were lowest for soil SRS-2001, with a median intra-lab value of 3.5 mg kg^{-1} Kg and highest for SRS-2004 with a value of 12.4 mg kg^{-1} Kg. Potassium 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 seven labs which exceeded 12 mg kg^{-1} K on SRS-2002. Poor precision is attributed to extraction and/or analysis instrument operation.

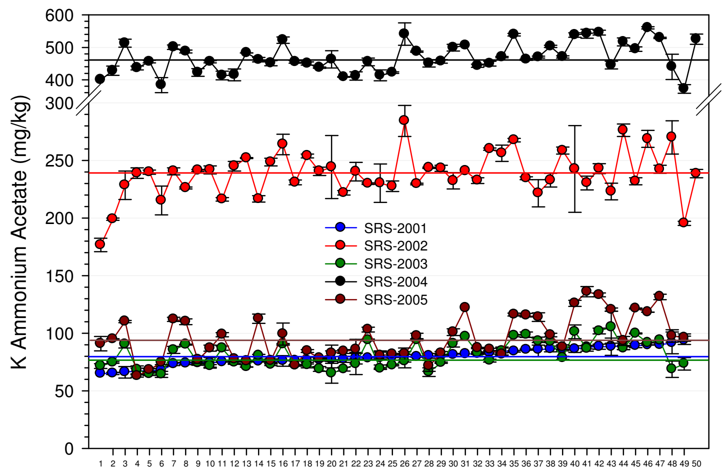


Figure 3. Extractable K distribution plots for SRS materials, ALP 2020 Cycle 41.

SRS - SOM-LOI

Forty-six laboratories provided ALP results for soil SOM-LOI (test code 182). Soil Median SOM-LOI values ranged from 1.30 to 6.28%. Results were ranked based on sample SRS-2001 (see Figure 4). Lab #1 had low bias and #46 high bias. Labs #4, #12 and #44 had inconsistency three of five soils. Sample SRS-2001 had high consistency associated with moderate SOM-LOI content. Bias was noted in three lab results. Source of bias is likely related to muffle furnace operation and/or method compliance.

SOM-LOI precision across the five soils indicates high intra-lab precision, with median *s* values ranging from 0.081 to 0.16% SOM-LOI, highest for SRS-2005. Across labs, *s* values for SRS-2002 ranged from 0.001 - 0.235%. Across soils low precision was noted for several laboratories. Specifically *s* for labs #6, #11, #15, #21, #30, #36, #42 and #46, exceeded 0.15% SOM-LOI for SRS-2003. Poor precision may be associated with muffle furnace crucible position and furnace heating time.

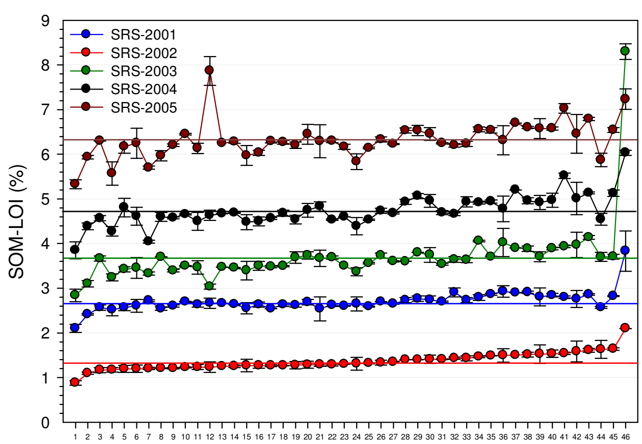


Figure 4. SOM-LOI distribution plots for SRS materials, ALP 2020 Cycle 41.

SRS - Sikora Buffer pH

Twenty-eight laboratories provided ALP results for Sikora Buffer pH (test code 123). Results were ranked low to high based on sample SRS-2001 (see Figure 5). Soil SRS-2005 was the most inconsistent in buffer pH across labs. Lab #1 had consistent low bias for five soils. Across soils, labs #2, #4, #13 and #27 were inconsistent across soils. Source of this inconsistency is likely related to instrument calibration or method compliance.

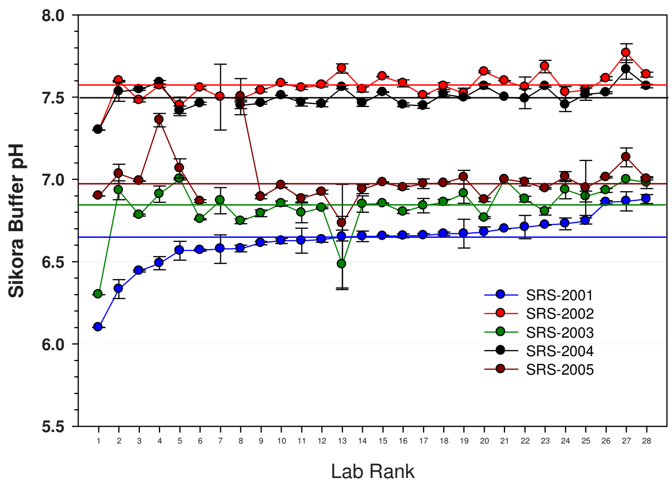


Figure 5. Soil Sikora Buffer pH distribution plot, ALP 2020 Cycle 41.

Sikora Buffer pH median intra-lab *s* values were lowest for ALP soil SRS-2002 and SRS-2004, averaging 0.025 and highest for SRS-2005 with a value of 0.23. Individual lab precision across the ALP soil materials indicates very high precision, generally, with the exception of soil SRS-2005 by six labs. Intra-lab precision was poor for labs #2, #5, #7, #13, #18 and #25 on three of five soils. Poor precision maybe associated with extraction and/or ICP-OES instrument operation. Three labs were flagged for poor precision.

SRB - NO_3-N

Twenty-four laboratories provided ALP results for NO_3-N by cadmium reduction, ISE and other (test codes 202, 203 and 204). Median values are designated by horizontal lines for each botanical material and labs results are ranked low to high based on sample SRB-2001 (see Figure 6). The data plot shows labs #1 - #4 had low bias on three of four samples. Labs #21 - #24 were inconsistent.

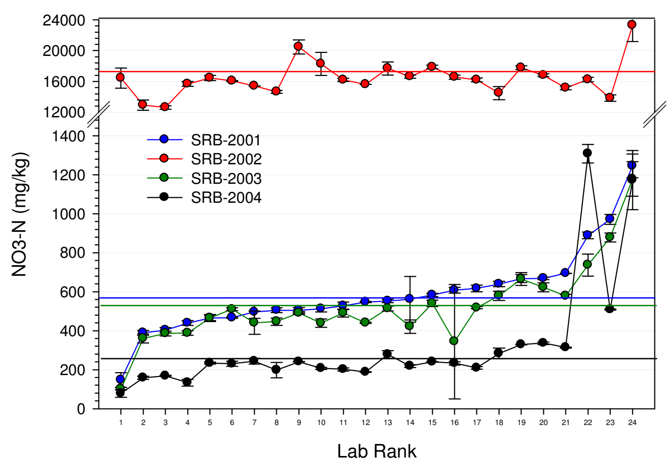


Figure 6. Nitrate distribution plots for SRB materials, ALP 2020, Cycle 41.

Botanical NO_3-N (test code 202) results for Cycle 41 indicate very high precision, with intra-lab median standard deviation (*s*) values ranging from 13.9 to 619 mg kg^{-1} for the four samples. Individual lab NO_3-N by cadmium reduction (test code 202) intra-lab *s* values for SRB-2001 ranged from 2.4 – 117 mg kg^{-1}; SRB-2002 ranged from 12 - 1510 mg kg^{-1}, SRB-2003 ranged from 2.5 – 293 mg kg^{-1} and SRB-2004 ranged from 1.2 - 39 mg kg^{-1} Lab #16 had consistently high standard deviations for three of four samples. Four labs were flagged for poor precision.

SRB - Dumas Nitrogen and TKN

Thirty-two laboratories provided ALP results for botanical Dumas (Combustion) Nitrogen (test code 210) and nine labs for TKN (Test code 209) for Cycle 39. Median values are designated by horizontal lines for each material and labs results ranked low to high based on sample SRB-2001 (see Figure 7). It is note worthy that TKN was inconsistent and lower than Dumas for all four samples. Lab #2 showed inconsistency across the three of four botanical samples. Sample SRB-2002 was inconsistent for TKN.

Dumas N results indicate very high precision across all labs for all samples. Individual lab Dumas N lab s values for SRB-2001, ranged 0.001 to 0.166% N, SRB-2002 ranged from 0.001 to 1.75 % N, SRB-2003 ranged from 0.003 to 0.006 % N, and SRB–2004 from 0.002 to 0.104 % N. Lab #22 had consistently high standard deviations on two samples. Lab TKN s values for SRB-2001 ranged from 0.004 to 0.078%, SRB-2002 ranged from 0.006 to 0.478% TKN, SRB-2003 ranged from 0.004 to 0.075% TKN nitrogen and SRB-2004 ranged from 0.006 to 0.135% TKN nitrogen.

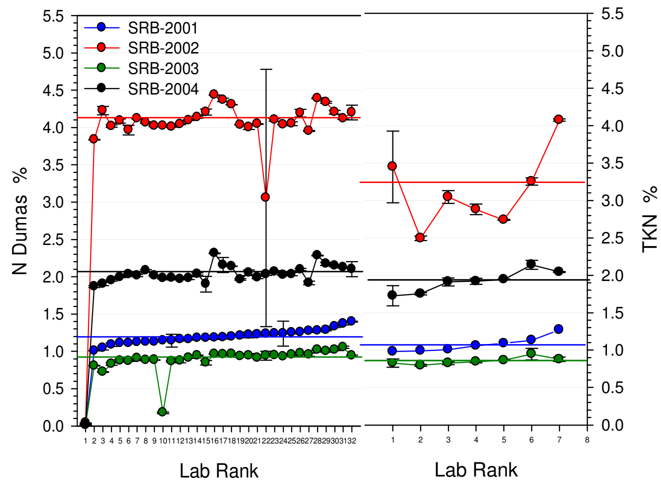


Figure 7. N distribution plots for SRB materials, ALP 2020 Cycle 41.

SRB - Potassium

Forty-one 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-2001 (see Figure 8). Labs #1, #8, #13, #25 and #25 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 0.011 to 0.068 %K for test code 213 across the four samples. Individual lab intra-lab s values were: SRB-2001, ranged from 0.006 to 0.324 % K; SRB-2002, 0.010 — 0.917 % K; SRB-2003, 0.003 - 0.229 % K; and SRS-2004, 0.010 to 0.177 % K. Four labs had high standard deviations exceeding 0.50 %K for SRB-2002 and two labs had standard deviations exceeding 0.20 %K for SRB-2001. Seven labs were flagged for poor K precision.

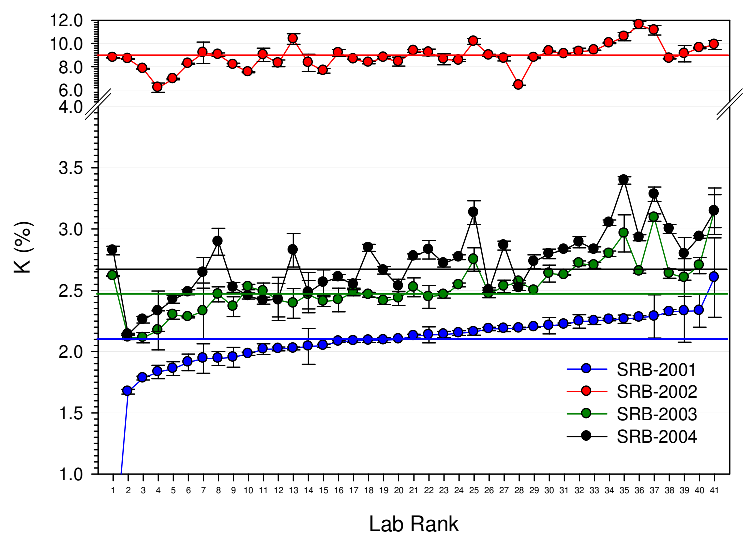


Figure 8. Potassium (code 213) plots for SRB materials, ALP 2020 Cycle 41.

SRB - Phosphorus

Forty-one laboratories provided ALP results for Cycle 41 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-2002 (see Figure 9). Consistent high bias was noted for labs #38 and #39. Labs #2, #7, #18, #26, and #34 showed inconsistency. Source of inconsistency is likely related to sample extraction, analysis instrument and/or method compliance.

Botanical P results indicate very high precision, with median intra-lab standard deviation (*s*) values ranged 0.010 to 0.018 % P for test code 212 across the four botanical samples. Individual lab intra-lab *s* values for SRB-2001; ranged from 0.001 - 0.035 % P; SRB-2002 ranged from 0.001 – 0.062 % P and SRB-2003 0.001 - 0.066 % P; and SRB-2004 0.001 - 0.057 % P. Labs #26 and #39 had a high standard deviation exceeding 0.05 % P on two of four botanical samples. Three labs was flagged for poor precision for botanical P.

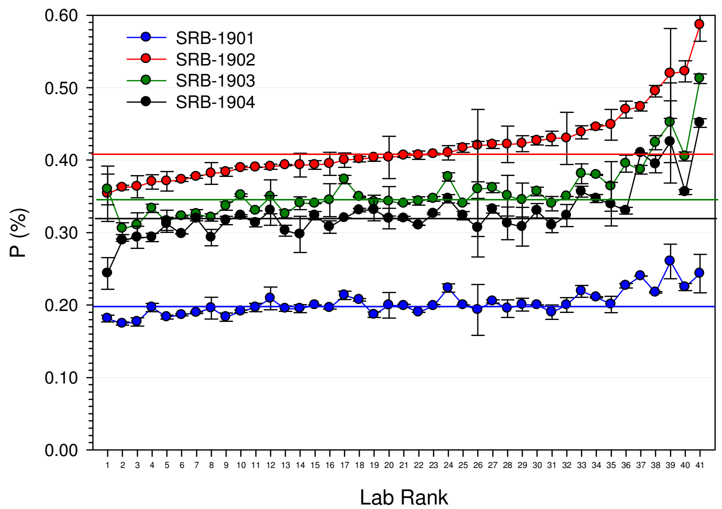


Figure 9. Phosphorus distribution plots for SRB materials, ALP 2020 Cycle 41.

SRB - Boron

Thirty-three laboratories provided ALP results for boron (B) (test code 219). 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-2001 (see Figure 10). Across samples labs #1 exhibited low bias. Labs #15, #25, #1 and #32 were inconsistent and data suggests that samples may have switched during analysis. Source of bias is likely related sample digestion, analysis instrument and/or method compliance.

Botanical B results indicate very high precision, with intra-lab standard deviation (*s*) values ranged from 0.64 to 2.8 mg kg^{-1} B for across the four botanical samples. Individual lab intra-lab *s* values for SRB-2001; ranged from 0.1 - 16.7 mg kg^{-1} B; SRB-2002 ranged from 0.05 – 2.6 mg kg^{-1} B; SRB-2003 0.08 - 3.1 mg kg^{-1} B; and SRB-2004 0.06 - 1.7 mg kg^{-1} B. Lab #33 had consistently high standard deviations for sample SRB-2001.

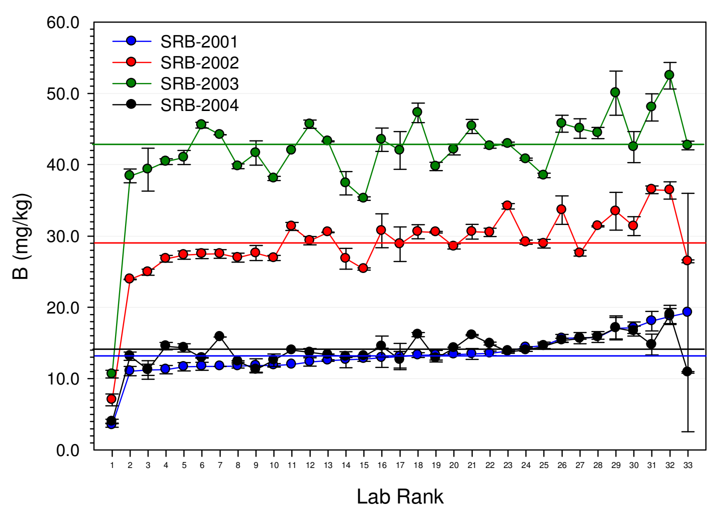
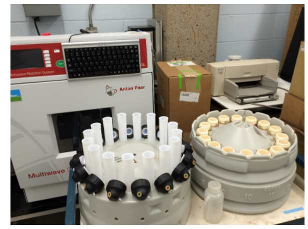


Figure 10. Boron distribution plots for SRB materials, ALP 2020 Cycle 41.

SRW - Water EC



Nineteen laboratories provided ALP results for water EC (test code 302). Lab result were ranked low to high based on sample SRW-2003 (see Figure 11). Sample SRW-2002 had the highest EC in Cycle 41. Lab #14 indicated consistent high bias on all samples. Lab #16 showed inconsistently across the three samples. Source of bias is likely associated with EC probe performance and/or calibration.

EC precision across the three water materials indicates good high precision, with intra-lab median Std values of 0.005, 0.007 and 0.001 dSm^{-1}, respectively. Precision for sample SRW-2003 was the most consistent across the ninteen participating laboratories. Intra-lab s values for lab #17 exceeded 0.07 dSm^{-1} on SRW-2002. Highest precision was noted for lab #5 with intra-lab s values of < than 0.004 dSm^{-1} on all three samples.

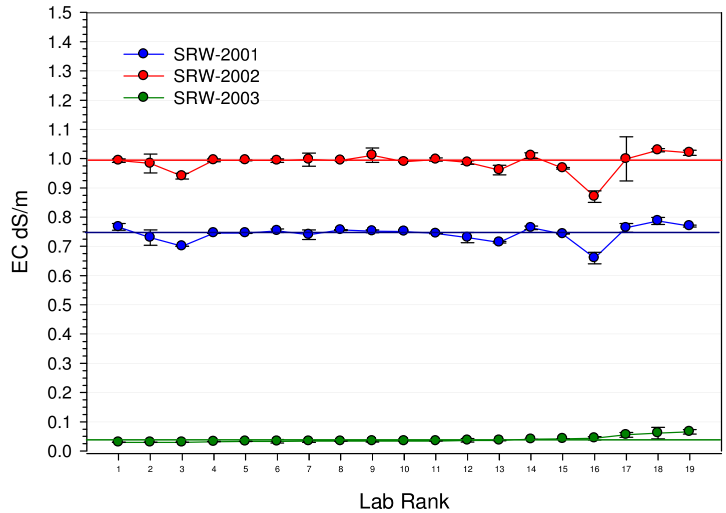


Figure 11 . Water EC distribution plots for SRW materials, ALP 2020 Cycle 41.

SRW - Na Results

Seventeen laboratories provided ALP results for water Na (test code 303). Lab results were ranked low to high based on sample SRW-2003 (see Figure 12) lowest in Na concentration. Median values are designated by horizontal lines. Labs #16 showed consistent high bias on all samples, and is likely a result of contamination or a reporting unit error.

Na precision across the three water solution matrices indicates excellent precision, with intra-lab s values of 0.106, 0.600, and 0.057 meq L^{-1} for SRW-2001, SRW-2002, and for SRW-2003, respectively. Water Na precision was excellent for all individual labs with only lab #14 exceeding 0.05 meq L^{-1} on two of the three samples. No labs were flagged for poor precision on ALP Cycle 41 for Na content.

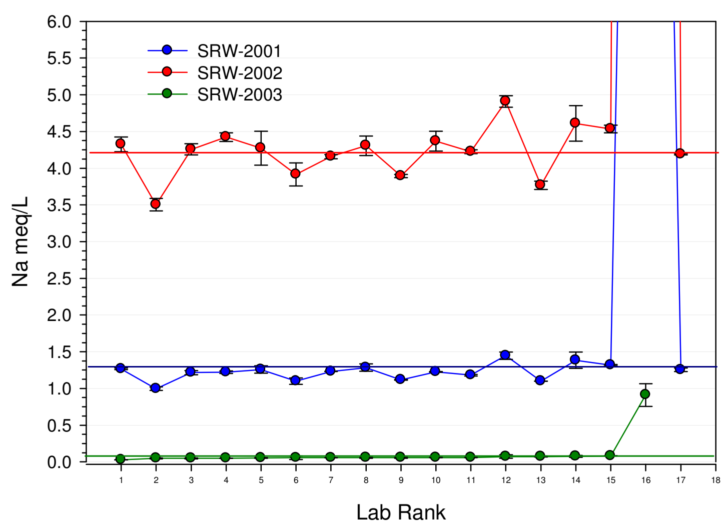


Figure 12. Water Na distribution plots for SRW materials, ALP 2020 Cycle 41.

Announcements

- The Illinois Soil Testing Association (ISTA) is now the Agricultural Laboratory Testing Association (ALTA) and has launched a new web site: www.ALTA.Ag. For more information contact the ALTA secretary, gfisher@unitedsoilsinc.com.

- The Soil and Plant Analysis Council (SPAC) and Agricultural Laboratory Testing Association (ALTA) have developed an international plant analysis certification program (PAC) for laboratories. Analyses include: N, P, K, S, Ca, Mg Zn, B, Mn, Fe, and Cu. The PAC program will be based exclusively on ALP proficiency testing data evaluated on a yearly basis. For more information can be found at ALTA.Ag.

- ALP has added new test methods to the soil proficiency program in 2020. Methods include Soil pH (1:1) 1.0 N KCL, Sikora 2 buffer pH. For more information on these methods contact the ALP Technical Director, Robert.Miller@cts-interlab.com.

- Cancellation. A summer lab analysis workshop that as scheduled in conjunction with the Soil Work Group meeting at Clemson University has been cancelled due to the COVID-19 pandemic.

- If there is a specific soil type, soil properties or botanical sample materials that you believe should be considered for the proficiency program please contact the ALP Program Technical Director.

Summary

ALP is celebrating twelve years of service with the completion of Cycle 41. Since 2005 ALP has completed the analysis of 205 soils, 128 plant samples and 116 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 41. 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 41 Ship

June 23, 2020

"Action springs not from thought, but from a readiness for responsibility."

— Dietrich Bonhoeffer

ALP Agricultural Laboratory Proficiency Program