2020 Volume 2

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ALP Program Report

2020 Summer - Cycle 42

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ALP Overview

Special points of interest:

- Soil homogeneity assessment indicate ALP reference soil materials were highly uniform for cycle 42.
- Fifty-nine Laboratories provided soil pH (1:1) H_2O results and medians ranged from 4.98 - 7.90.
- Soil M3-P ICP for cycle 42 ranged from 18.5 to 92.1 mg kg^{-1} with MAD values ranging 1.0 - 6.4 mg kg^{-1} across the five soils.
- Soil NH_4OAc-3 K values ranged from 72 - 545 mg kg^{-1} for the five ALP soils of PT cycle 42.
- Botanical N by combustion was reported by 34 labs, with nine labs showing high bias values on the two materials with > 6% N for cycle 42.
- Botanical K, ranged from 1.30 - 7.07% with four of forty labs noted for inconsistency.
- Botanical Zn results showed high consistency across thirty-four of thirty-nine labs for PT cycle 42.
- Water Na content showed very high consistency by eleven of thirteen labs across all PT samples.

Inside this issue:

The Agriculture Laboratory Proficiency (ALP) Program fall 2020 Round Cycle 42 was completed August 27, 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.

https://www.gazettenet.com/getattachment/d4b1bb82-77b8-4212-9842-c4f7b8697824/24home-soiltest-hg-112417-ph1

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 42 were: SRS-2006 a Marshall silty clay loam, collected in Brown Cty, KS; SRS-2007 silty clay loam collected near Three Bridges, Ontario, Canada; SRS-2008 an Adrian muck collected in Porter Cty, IN; SRS-2009 is a Pulaski fine sandy loam collected in Payne Cty Oklahoma; and SRS-2010 a Newdale silt loam collected in Power Cty, ID. Chemical properties of the SRS materials ranges: pH (1:1) H_2O 4.98 - 7.90; NO_3-N 15.4 - 110 mg kg^{-1}; Bray P1 (1:10) 13.9 - 60.3 mg kg^{-1}; M3-K 66 - 500 mg kg^{-1}; SO_4-S 4.6 - 12.1 mg kg^{-1}; DTPA-Mn 0.98 - 62.0 mg kg^{-1}; SOM-LOI 0.98 – 21.1%; CEC 3.4 - 50.5 cmol kg^{-1}; clay 7.7 - 30.9% and saturated paste moisture 29.3 - 70.0 %.

Standard Reference Botanical (SRB) materials for Cycle 42 were: SRB-2005 arugula Leaf composite from AZ; SRB-2006 walnut Leaf composite from CA; SRB-2007 soybean leaves from AR; and SRB-2008 grass hay from NEA. SRB median analytes ranged: NO_3-N 52 - 9870 mg kg^{-1}; Dumas N 1.7 - 6.35%; total P 0.15 - 0.67%; total K 1.30 - 7.07%; total Ca 0.49 - 2.56%; total S 0.13 - 1.20 %, total B 10.3 - 47.9 mg kg^{-1}; and total Sr 4.9 - 302 mg kg^{-1}.

Standard Reference Water (SRW) samples represent an agriculture water samples collected: SRW-2004 a water sample collected from a stream near Royal, IA: SRW-2005 was collected from a well near Ellsworth, MN; and SRW-2006 from an irrigation canal Weld Cty, CO. SRW median concentrations ranged: pH 7.31 - 8.17; EC 0.10 - 0.82 dSm^{-1}; SAR 0.28 - 1.73; Ca 0.55 - 5.60 mmolc L^{-1}; Na 0.17 - 1.49 mmolc L^{-1}; HCO_3 0.48 - 5.67 mmolc L^{-1}; and NO_3 0.006 - 1.01 mmolc L^{-1}.

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 five 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 H_2O. Homogeneity was also evaluated on SRB and SRW matrix samples.

"..soil pH, Buf pH A&E, Olsen P and SOM-WB analysis Stdev values for Cycle 42 met homogeneity standards."

Table 1. ALP soils homogeneity evaluation 2020, Cycle 42.

Sample	pH (1:1) H_2O		pH A&E Buffer		Olsen P (mg kg^{-1})		SOM-WB (%)	
	Mean [1]	*Std*	Mean	*Std*	Mean	*Std*	Mean	*Std*
SRS-2006	5.41	*0.01*	7.27	*0.01*	5.9	*0.5*	3.52	*0.108*
SRS-2007	7.09	*0.01*	7.70	*0.01*	8.5	*1.0*	8.46	*0.41*
SRS-2008	5.89	*0.01*	6.89	*0.02*	10.9	*0.6*	24.94	*2.11*
SRS-2009	4.88	*0.01*	7.73	*0.01*	8.1	*0.5*	0.92	*0.05*
SRS-2010	7.88	*0.02*	7.85	*0.004*	23.0	*1.5*	1.76	*0.11*

[1] Statistics based on five randomly selected soil replicates, each analyzed in triplicate ALP Cycle 42.

2020 Cycle 42 Observations

Results for soil pH (1:1) H_2O (test code 115) analysis MAD values for Cycle 42 averaged 0.07 pH units across the soils. Median within lab pH standard deviation was 0.055 pH units. Soil displacement CEC ranged 3.4 to 50.5 cmol kg^{-1} across the five soils. Sample SRS-2008 had a large discrepancy in soil CEC values: displacement 50.5 cmol kg^{-1} and estimated CEC of 33.7 cmol kg^{-1}. SRS-2001 had an abnormally low extractable Cl of 2.4 mg kg^{-1}, likely associated with Marshall clay loam soil series. Soil ammonium acetate Ca (Test code 140) MAD values ranged 45 - 489 mg kg^{-1} and ammonium acetate Mg MAD values ranged 4.8 to 75 mg kg^{-1} for the five soils. These results for Ca 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 0.098% nitrogen with intra-lab median *s* of 0.025%, 0.013%, 0.029% and 0.019%, respectively. Botanical sample SRB-2008 had a very low median Mn with a concentration of 8.7 ppm and with a MAD of 0.5 ppm. Generally the walnut leaf composite sample SRB-2006 had lower median concentrations of NO_3-N, N, Cl, P, K, S, Na, and Mo and relative to the other three botanical samples. One observation on Cycle 42, intra-lab relative variability was lowest for N than 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 0.338, 0.817 and 0.10 dSm^{-1}, respectively. Cl values ranged from 0.051 - 0.610 molc L^{-1} across the three water samples with MAD values ranging 0.033 to 0.041 molc L^{-1}. Sample SRW-2004 had and SAR of 1.74 with a MAD of 0.04.

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

Fifty-nine laboratories provided ALP results for soil pH (1:1) H_2O (test code 115). Soils ranged from acid to alkaline, median range 4.98 - 7.90. Lab results were ranked low to high based on sample SRS-2009 (see Figure 1) with median pH designated by horizontal lines for each soil. Generally soils SRS-2006, 2008 and SRS-2009 showed good consistency across labs. Labs #10-#4 showed consistent low bias across all five soils. Labs #43, #56, #57, #58 and #59 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.

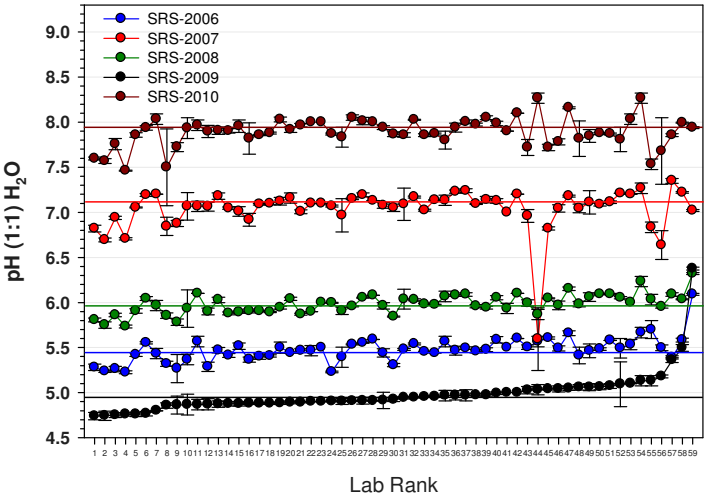


Figure 1. pH (1:1) H_2O distribution plots for SRS materials, ALP 2020 Cycle 42.

pH precision across the five ALP soils indicates very high precision, with median intra-lab standard deviation (*s*) values ranging from 0.017 to 0.025 pH units, the lowest noted for SRS-2008. For specific labs poor precision was noted for five laboratories, exceeding by three times that noted for consensus median intra-lab *s*. Specifically *s* for labs #10, #25, #31, #44, #49 and #56 exceeded 0.10 pH units for SRS-2007. Soil SRS-2001 was the least variable with respect to intra-lab variance for Cycle 42.

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

Bray P1 results were reported by twenty-nine labs. M3-P ICP was reported by 40 labs. Median soil Bray P1 values ranged from 3.9 - 60.3 mg kg^{-1} PO_4-P; Olsen P 6.8 to 22.3 mg kg^{-1} P and Bray P2 ranged from 18.3 to 363 mg kg^{-1} P, across the five soils. Ranking lab results based on sample SRS-2006, median M3-P ICP concentrations are shown in indicated in Figure 2. A saw tooth trend was noted for soils SRS-2007, SRS-2008, and SRS-2009 associated with the moderate P concentrations. There was a lack of consistency for all three of samples for across all labs. Soil SRS-2003, lowest in concentration, showed low intra-lab variability. Labs #38, #39 and #40 showed consistent high on all three of five samples. Labs #4, #19, #34 and #35 were inconsistent.

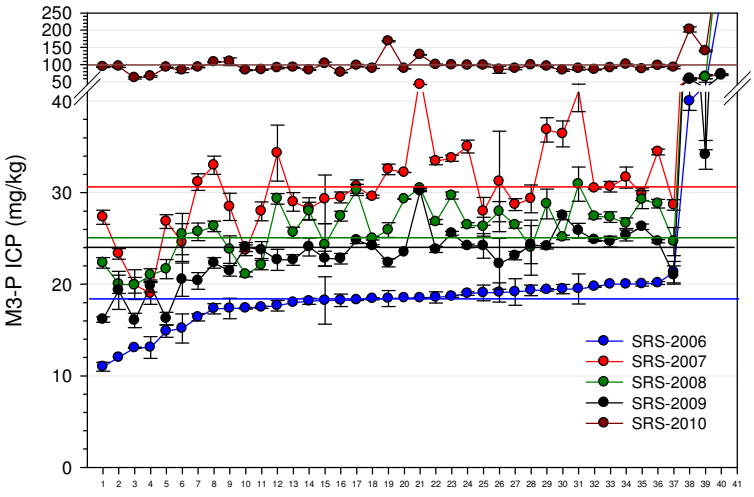


Figure 2. M3-P ICP distribution plots for SRS materials, ALP 2020 Cycle 42.

Four laboratories provided ALP results for Mehlich 1 P, with medians ranging from 4.7 to 259 mg kg^{-1} PO_4-P. M3-P Spec median concentrations were 14.1 - 81.9 mg kg^{-1} P reported by eight labs. Modified Kewolna was reported by two laboratories ranging from 9.0 - 47.2 mg kg^{-1} P and total P (US-EPA 503 ranged 185 - 800 mg kg^{-1} P with the highest concentration noted for SRS-2010.

SRS - Potassium

Forty-five laboratories provided ALP results for soil K (test code 142) results. Results were ranked low to high based on sample SRS-2007 (see Figure 3). Soils SRS-2008 were the most inconsistent across labs likely associated with the high SOM content. Lab #44 had high bias on all five soils. Labs #11, #20, #36, and #41 were inconsistent across the five soils for K. It is worth noting twenty-four labs found near identical K concentrations for SRS-2007 and SRS-2009. 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-2007, with a median intra-lab value of 1.5 mg kg^{-1} Kg and highest for SRS-2008 with a value of 8.5 mg kg^{-1} Kg. Potassium within-lab precision across the ALP soil materials indicates very good precision, generally, for soils with less than 300 mg kg^{-1} K. Precision was poor (based on intra-lab *s*) for five labs which exceeded 20 mg kg^{-1} K on SRS-2008. Poor precision is attributed to extraction and/or analysis instrument operation.

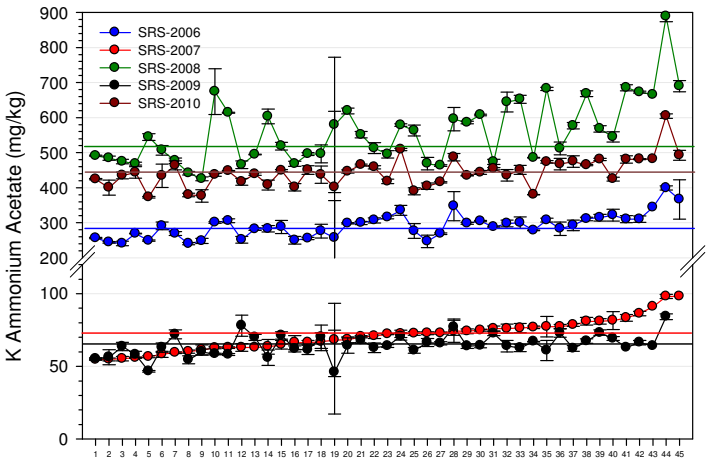


Figure 3. Extractable K distribution plots for SRS materials, ALP 2020 Cycle 42.

SRS - SOM-LOI

Forty-five laboratories provided ALP results for soil SOM-LOI (test code 182). Soil Median SOM-LOI values ranged from 0.98 to 21.1%. Results were ranked based on sample SRS-2009 (see Figure 4). Labs #4, #7, #24 and #45 had inconsistency three of five soils. Sample SRS-2009 had high consistency associated with the lowest SOM-LOI content. High bias was noted in one 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 0.017 to 0.20% SOM-LOI, highest for SRS-2008. Across labs, *s* values for SRS-2009 ranged from 0.005 - 0.301%. Across soils low precision was noted for several laboratories. Specifically *s* for labs #7, #9, #15, #20, #22, #34, #36 and #37, exceeded 0.12% SOM-LOI for SRS-2006. Poor precision may be associated with muffle furnace crucible position and furnace heating time.

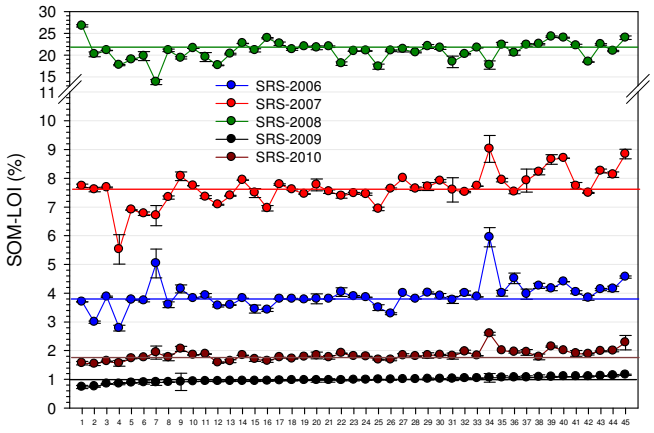


Figure 4. SOM-LOI distribution plots for SRS materials, ALP 2020 Cycle 42.

SRS - Saturated Paste Ca

Twenty-four laboratories provided ALP results for Saturated Paste Ca (test code 106). Results were ranked low to high based on sample SRS-2006 (see Figure 5). Soil SRS-2005 was the most consistent across labs. Lab #1 had consistent low bias for five soils. Across soils, labs #4, #14, #17 and #24 were inconsistent across soils. Source of this inconsistency is likely related to instrument calibration or method compliance.

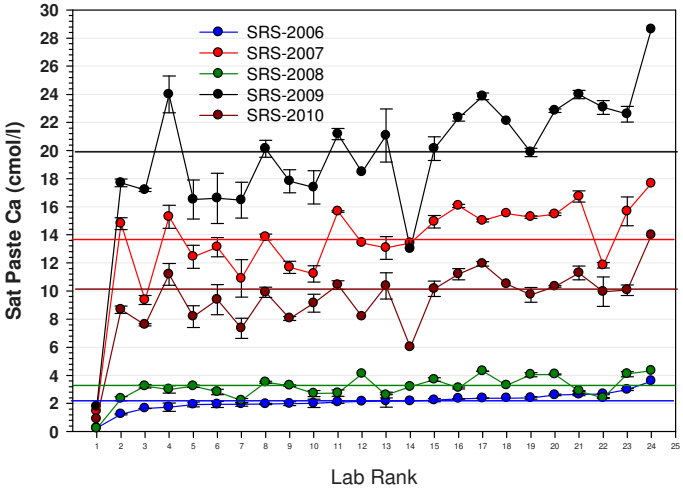


Figure 5. Saturated paste Ca distribution plot, ALP 2020 Cycle 42.

Saturated Paste Ca median intra-lab *s* values were lowest for ALP soil SRS-2006 and SRS-2008, averaging 0.06 cmol/l and highest for SRS-2010 with a value of 0.37 cmol/l. Individual lab precision across the ALP soil materials indicates very high precision, generally, with the exception of soil SRS-2009. Intra-lab precision was poor for labs #4 through #7 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-six 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-2006 (see Figure 6). The data plot shows labs #19 - #24 had high bias on three of four samples. Labs #2 and - #15 were inconsistent.

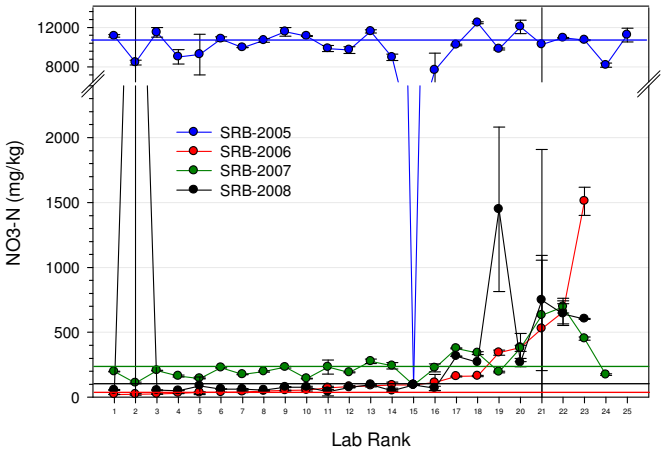


Figure 6. Nitrate distribution plots for SRB materials, ALP 2020, Cycle 42.

Botanical NO_3-N (test code 202) results for cycle 42 indicate very high precision, with intra-lab median standard deviation (*s*) values ranging from 6.7 to 208 mg kg^{-1} for the four samples. Individual lab NO_3-N by cadmium reduction (test code 202) intra-lab *s* values for SRB-2005 ranged from 2.4 – 2070 mg kg^{-1}; SRB-2006 ranged from 1 - 560 mg kg^{-1}, SRB-2007 ranged from 1.5 – 425 mg kg^{-1} and SRB-2008 ranged from 1.2 - 78 mg kg^{-1} Lab #21 had consistently high standard deviations for three of four samples. Two labs were flagged for poor precision.

SRB - Dumas Nitrogen and TKN

Thirty-four 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-2006 (see Figure 7). Labs #27 - #34 showed a consistent high bias trend on the two botancial samples with > 6.0% N. It is note worthy that TKN was inconsistent and lower than Dumas for two samples. Sample SRB-2006 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-2005, ranged 0.006 to 0.758% N, SRB-2006 ranged from 0.004 to 0.343 % N, SRB-2007 ranged from 0.001 to 0.627 % N, and SRB–2008 from 0.001 to 0.285 % N. Lab #1 had consistently high standard deviations on all samples. Lab TKN *s* values for SRB-2005 ranged from 0.007 to 0.73%, SRB-2006 ranged from 0.004 to 0.45% TKN, SRB-2007 ranged from 0.004 to 0.63% TKN nitrogen and SRB-2008 ranged from 0.005 to 0.123% TKN nitrogen.

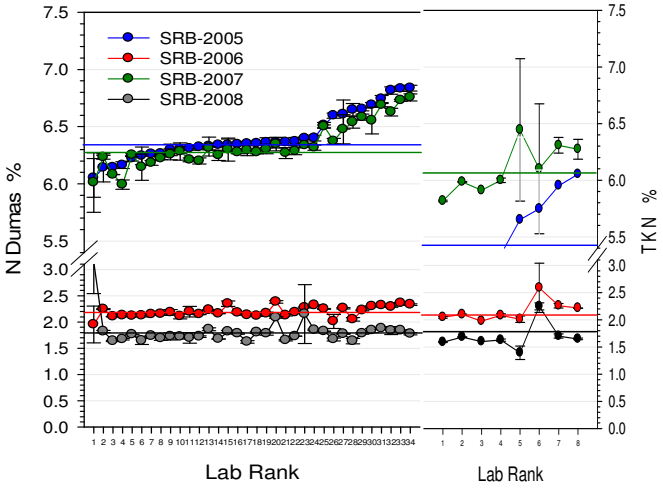


Figure 7. N distribution lab plos for SRB materials, ALP 2020 Cycle 42.

SRB - Phosphorus

Forty laboratories provided ALP results for Cycle 42 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-2006 (see Figure 8). Consistent high bias was noted for labs #37, #38, #39 and #40. Labs #5, #13, #22, and #36 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 0.006 to 0.010 % P for test code 212 across the four botanical samples. Individual lab intra-lab *s* values for SRB-2005; ranged from 0.001 - 0.110 % P; SRB-2006 ranged from 0.001 – 0.113 % P and SRB-2007 0.001 - 0.084 % P; and SRB-2008 0.001 - 0.085 % P. Lab #12 had a high standard deviation exceeding 0.05 % P on two of four botanical samples. Three labs were flagged for poor precision for botanical P for cycle 42.

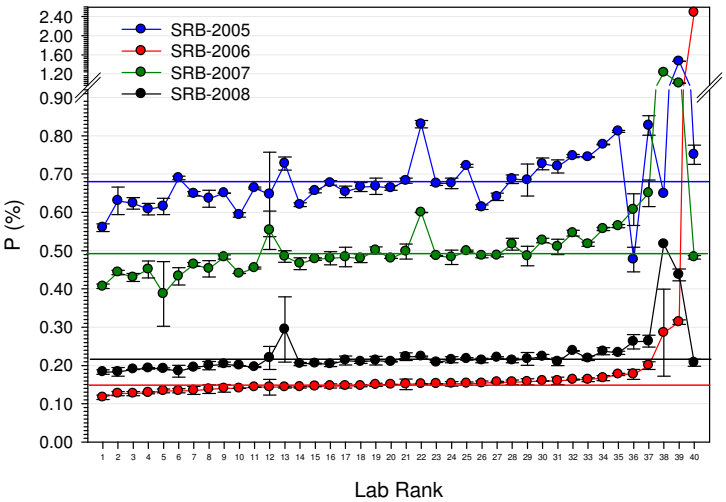


Figure 8. Phosphorus distribution lab plot for SRB materials, ALP 2020 Cycle

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-2006 (see Figure 9). Labs #5, #16, #33, and #37 were inconsistent. Laboratories #39 and #40 had consistent high bias across all four botanical materials evaluated. 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 0.022 to 0.099 %K for test code 213 across the four samples. Individual lab intra-lab *s* values were: SRB-2005, ranged from 0.010 - 1.06 % K; SRB-2006, 0.001 - 1.21 % K; SRB-2007, 0.006 - 0.477 % K; and SRS-2008, 0.003 - 3.16 % K. Lab #5 had high standard deviations exceeding 0.25 %K on three of four samples. Four labs were flagged for poor K precision for cycle 42.

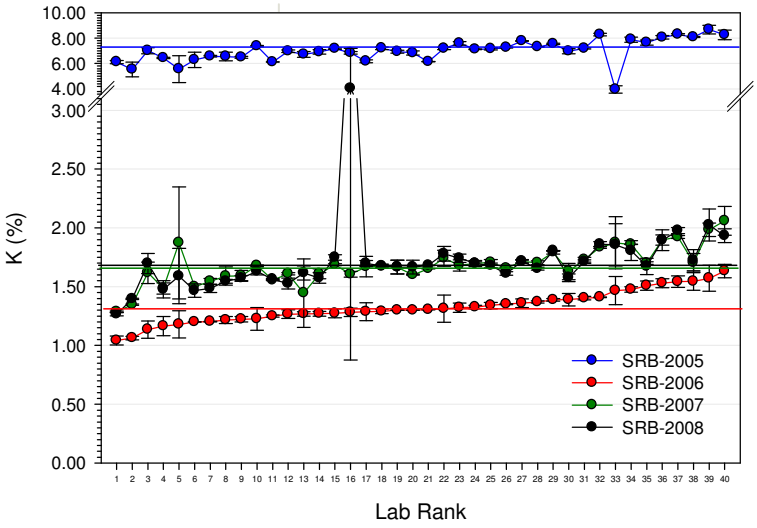


Figure 9. Potassium lab plot for SRB materials, ALP 2020 Cycle 42.

SRB - Zinc

Thirty-three laboratories provided ALP results for zinc (Zn) (test code 220). 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-2008 (see Figure 10). Across samples labs #1 and #2 exhibited low bias. Labs #4, #17, #35, #37 and #40 were inconsistent. Source of bias is likely related sample digestion, analysis instrument and/or method compliance.

Botanical Zn results indicate very high precision, with median intra-lab standard deviation (*s*) values ranged from 0.62 to 1.5 mg kg^{-1} Zn for across the four botanical samples. Individual lab intra-lab *s* values for SRB-2005; ranged from 0.14 - 7.5 mg kg^{-1} Zn; SRB-2006 ranged from 0.10 – 13.0 mg kg^{-1} Zn; SRB-2007 0.15 - 6.2 mg kg^{-1} Zn; and SRB-2008 0.10 - 28.8 mg kg^{-1} Zn. Lab #37 had consistently high standard deviations for sample SRB-2001. Five labs were flagged for poor K precision for cycle 42.

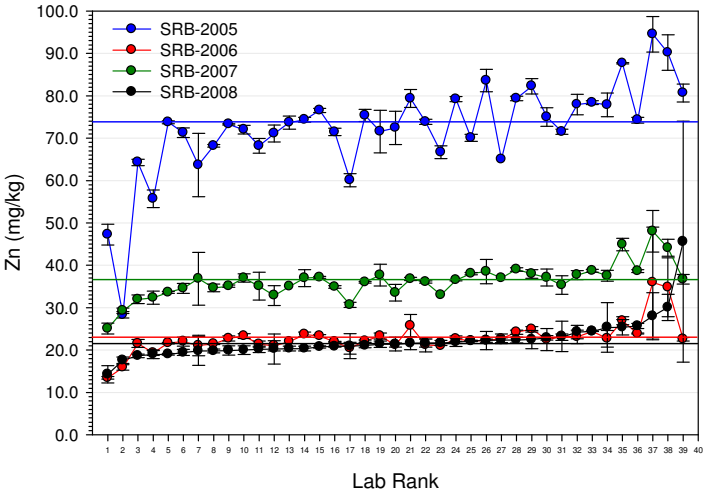
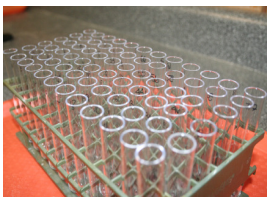


Figure 10. Zinc (code 220) lab plots for SRB materials, ALP 2020 Cycle 42.

SRW - Water EC



Thirteen laboratories provided ALP results for water EC (test code 302). Lab result were ranked low to high based on sample SRW-2006 (see Figure 11). Sample SRW-2005 had the highest EC in Cycle 42. 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 0.006, 0.008 and 0.0001 dSm^{-1}, respectively. Precision for sample SRW-2006 was the most consistent across the thirteen participating laboratories. Intra-lab *s* values for lab #13 exceeded 0.02 dSm^{-1} on SRW-2004. Highest precision was noted for lab #2 with intra-lab *s* values of < than 0.002 dSm^{-1} on all three samples.

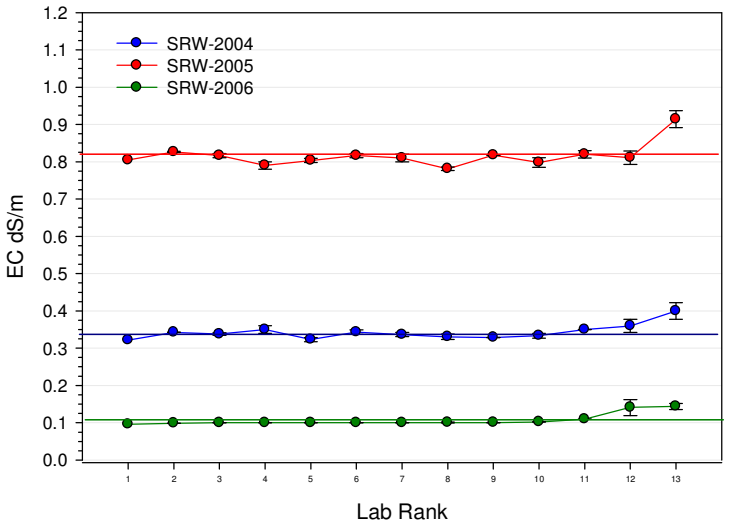


Figure 11 . Water EC distribution plots for SRW materials, ALP 2020 Cycle 42.

SRW - Mg Results

Fourteen laboratories provided ALP results for water Mg (test code 303). Lab results were ranked low to high based on sample SRW-2006 (see Figure 12) lowest in Na concentration. Median values are designated by horizontal lines. Labs #1 showed consistent low bias on tow of thee samples, and is likely a result of a calibration error.

Mg precision across the three water solution matrices indicates excellent precision, with intra-lab *s* values of 0.017, 0.051, and 0.007 meq L^{-1} for SRW-2004, SRW-2005, and for SRW-2006, respectively. Water Mg precision was excellent for all individual labs with only lab #7 exceeding 0.15 meq L^{-1} on two of the three samples. One lab was flagged for poor precision on ALP Cycle 42 for Mg content.

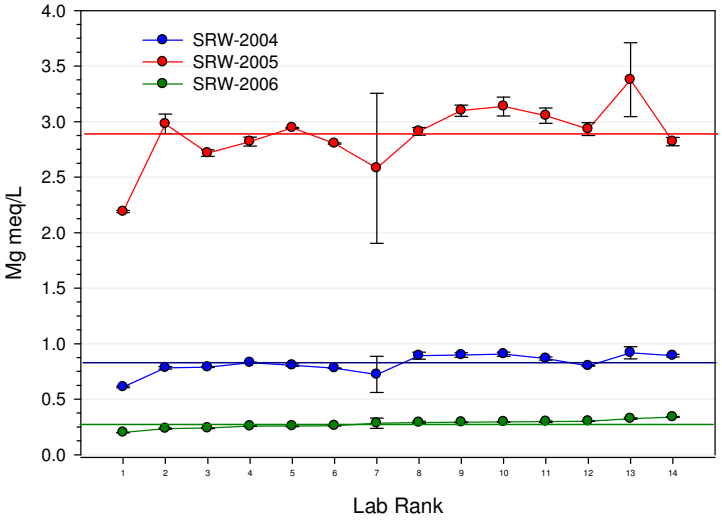


Figure 12. Water Mg distribution plots for SRW materials, ALP 2020 Cycle 42.

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.

- A new ALP web site for program participants will initiate September 8th. Generally information on the program will be available, and a special section on method specific topics and lab quality control /quality control will be offered. +

- 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.

- 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 has provided fifteen years of service with the completion of Cycle 42. Since 2005 ALP has completed the analysis of 210 soils, 132 plant samples and 119 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 42. 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 42 Ship

September 17, 2020

"The true laboratory is the mind, where behind illusions we uncover the laws of truth."

— Jagadish Chandra Bose, 1937

ALP
Agricultural Laboratory Proficiency Program