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USDA LTAR Common Experiment measurement: Concentration of phosphorus, potassium, and sulfur in aboveground biomass

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John L. Kovar[1], Ann Marie Fortuna[2]

[1]USDA Agricultural Research Service, National Laboratory for Agriculture and the Environment, Ames, IA;
[2]USDA Agricultural Research Service, Agroclimate and Hydraulics Research Unit, El Reno, OK

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Lori J Abendroth
USDA ARS

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Abstract

An adequate supply of plant nutrients is an important part of sustainable crop production and the overall health of a crop. Knowing the amount of each nutrient removed in the harvested biomass (grain, forage, etc.) is the first step in calculating plant nutrients required. When the removed nutrients are not replaced, soil fertility declines over time, leading to a loss of yield potential. Phosphorus (P) is a primary macronutrient, with total P concentration in crops generally varying from 0.1 to 0.5%. Phosphorus can be found in roots, stems, and leaves, but most P is in seeds and fruit. It is a component of phytin, a major storage form of P in seeds and tubers. Potassium (K) is a primary macronutrient stored in the stems and foliage of aboveground biomass. Seeds contain only small amounts of K. Potassium is mobile in plant tissues and can easily leach. Therefore, estimates of plant biomass removed and remaining in the field are valuable for determining soil K fertility. Sulfur (S) is a secondary macronutrient in seeds and plant tissues. Nodule formation in legume crops requires S. Research has shown that S fertilization may improve nitrogen (N)-use efficiency in many crops by increasing N uptake. Determine P, K, and S concentrations in the biomass by first digesting the material. The nutrient concentrations in the digestate can then be determined using inductively coupled plasma-optical emission spectrometry (ICP-OES).

Sample collection

1 In most cases, biomass samples will be collected at each harvest, potentially resulting in multiple samples per year.

2 For the common experiment, it is important to collect samples from BAU and ASP treatments when plants are at the same phenological (developmental) stage.

3 This process may require multiple samplings if crops significantly differ in developmental stage between or among the treatments.

Sample processing

3d

4 Dry the biomass samples in a forced-air oven at 60 °C until a constant weight is reached (48 to 72 hours).

5 Grind a subsample to pass a 2-mm screen, using a Wiley or similar mill.

6 Grind a 2-mm subsample to pass a 1-mm screen, using a Udy cyclone or similar mill.

7 If samples are stored for an extended period, redry before analysis.

Sample analysis

8 Send samples to a commercial laboratory or analyze them at an ARS or University laboratory if equipment and personnel are available.

8.1 When choosing a commercial lab, consider the following factors :
(1) whether the laboratory participates in a proficiency testing program, e.g., NAPT or ALP
(2) whether the laboratory returns samples for archiving purposes.

Note

The following protocol details methods for performing laboratory procedures in-house.

9 Before the concentration of nutrients in biomass can be determined, it is first necessary to destroy the organic matrix and solubilize the nutrients of interest.

10 Several methods are available for this purpose, the most common of which are :
i) addition of concentrated acid to the sample followed by heating
ii) addition of concentrated acid to the sample in a sealed vessel followed by microwave heating (Bryson and Mills, 2014; Havlin and Soltanpour, 1980).

(i) Nitric and hydrochloric acid digestion with peroxide, open vessel

11 This method involves the acid dissolution of the organic matrix by digestion via external heating (hot plate, digestion block, or automated digestion block system), with the subsequent quantitative determination of P, K, and S in the digestate.

12 Pre-digest a representative sub-sample containing up to 0.5 g of dry matter in nitric acid overnight.

13 Digest the sample in heated nitric acid and hydrogen peroxide.

14 After cooling, filter the vessel contents, centrifuge or allow to settle, dilute with nitric or hydrochloric acid to volume, and analyze by ICP-OES.

(ii) Microwave-assisted digestion, closed vessel

15 This method involves the dissolution of the organic matrix in a closed vessel by nitric acid digestion via microwave heating for the subsequent quantitative determination of P, K, and S.

16 Digest a representative sub-sample in concentrated nitric acid using a suitable laboratory microwave unit.

17 Cap and heat a fluorocarbon (PFA or TFM) microwave vessel containing the sample and acid in the microwave unit.

18 After cooling, filter the vessel contents, centrifuge or allow to settle, dilute to volume, and analyze by ICP-OES.

Calculations and sufficiency ranges

19 Report P, K, and S concentrations on a dry-matter basis.

20 Calculate the amount of P, K, or S leaving the field by multiplying the biomass concentration by the amount of biomass removed (g nutrient/kg dry matter × kg dry matter/ha).

21 When reviewing P, K, and S data, it may be helpful to review sufficiency ranges for row crops, fruits, and vegetables. The Bryson and Mills (2014) reference contains tables for macro- and micro-nutrients although these will vary by environment. Utilize local publications for greater accuracy.

Quality assessment and quality control

22 Recommend a standard operating procedure for your laboratory for measuring P, K, and S, with the following items included:

22.1 Perform instrument calibration and maintenance according to the manufacturer's instructions and log the results.

22.2 Prepare a standard calibration curve on the day of analysis with a minimum of six (6) concentrations, including a blank. Run calibration check standards throughout the assay every 20 samples to verify calibration accuracy and adjust for any drift. Values should be within 10% of the known value.

22.3 Run a certified reference standard (e.g., National Institute of Standards and Technology (NIST)) with each analytical sequence to verify calibration and method accuracy. Certified values should fall between 90% and 110% recovery. The results should not vary more than three standard deviations from the accepted mean value.

22.4 Determine method detection limits (MDL) based on the open-vessel nitric/hydrochloric acid or the closed-vessel nitric acid digestion matrices.

Illustrative information

23

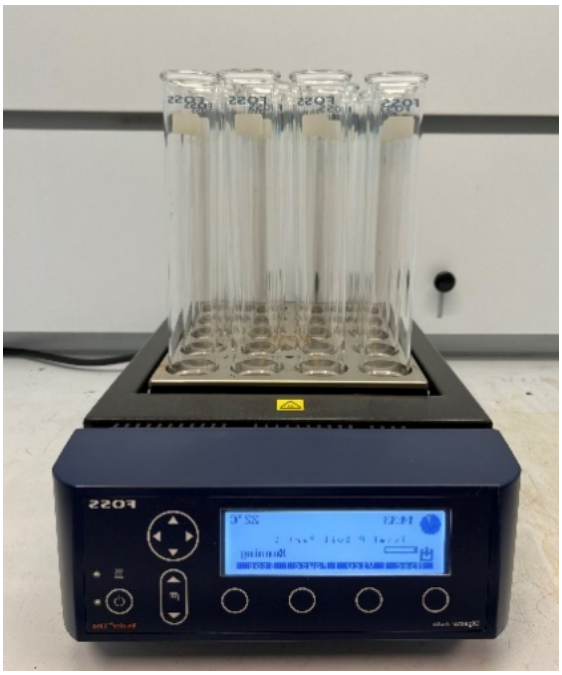

Figure 1. Digestion block for open-vessel acid dissolution of the organic matrix. Photo credit: J. Berkey, USDA-ARS.

Figure 2. Microwave for closed-vessel nitric acid dissolution of the organic matrix. Photo credit: A. Morrow, USDA-ARS.

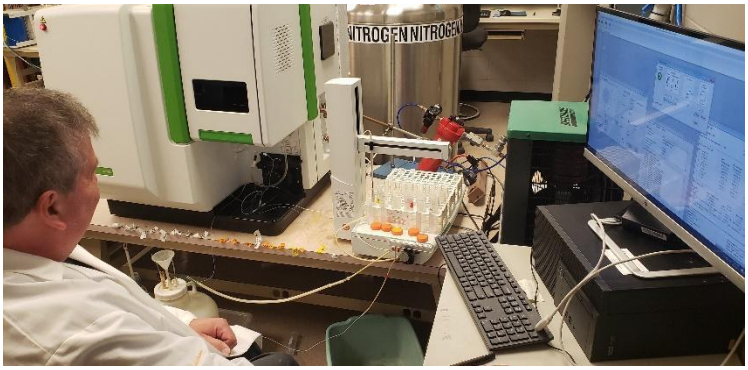

Figure 3. Inductively coupled plasma-optical emission spectrometer (ICP-OES) with an autosampler to determine P, K, and S concentrations in the digestate. Photo credit: A. Morrow, USDA-ARS.

Recommendations for data collection

24 Table 1. Summary of recommendations for measurement of aboveground biomass P, K, and S concentrations.

	A	B	C	D
	Attribute	Preferred	Minimum	Comments
	Spatial scale	Field	Plot	
	Frequency	Every crop harvest	Every crop harvest	Maximum vegetative biomass
	Covariate metrics	Biomass staying and leaving P, K, and S concentrations	Biomass staying and leaving P, K, and S concentrations	ICP analysis of biomass digests allows the determination of other plant-essential elements
	Other	Biomass staying and leaving C and N concentrations	Biomass staying and leaving C and N concentrations	Refer to the aboveground biomass C and N percentages protocol for procedures to determine these elements

Protocol references

Bryson, G., and H.A. Mills. 2014. Plant Analysis Handbook IV. Athens, GA: Micro-Macro Publishing, Inc. (link for purchasing the book: **10.13140/2.1.1693.2646**)

Havlin, J.L., and P. N. Soltanpour. 1980. A nitric acid plant tissue digest method for use with inductively coupled plasma spectrometry. Communications in Soil Science and Plant Analysis, 11:10, 969-980. https://doi.org/10.1080/00103628009367096