NCDA&CS Methods for Soilless Media Analysis

Plant/Waste/Solution/Media Laboratory

Agronomic Division

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http://www.ncagr.gov/agronomi/uyrsoln.htm

Soilless Media Analysis

Soilless media analysis is used to test the inorganic minerals and other parameters of container mixes used in greenhouse and nursery production. The NCDA&CS Agronomic Division does not perform any testing for microbial agents (e.g., pathogens, algae) or organic contaminants (e.g., pesticides, herbicides, petroleum products).

The standard soilless media analysis includes measurement of nitrate-nitrogen (NO_3-N), ammonium-nitrogen (NH_4-N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron (B), sodium (Na), aluminum (Al), and chloride (Cl) concentrations. Soluble salts (SS or EC) and pH are also measured. Nutrient balances (NH_4-N, NO_3-N, K, Ca, Mg, Na, Cl) as a percent of the electrical conductivity (EC) and nutrient ratios (K:Ca, Ca:Mg, K:Mg) are calculated and reported.

Standard media analysis for **N.C. residents**	$5.00
Standard media analysis for **non-N.C. residents**	$25.00
ǂ Standard media analysis for **N.C. researchers**	$12.00
ǂ Standard media analysis for **non-N.C. researchers**	$25.00

ǂ A completed NCDA&CS Research Project Agreement is **required prior to submission** of research samples. *Please contact Dr. Kristin Hicks at Kristin.Hicks@ncagr.gov to set up a Research Project Agreement. The NCDA&CS Cooperative Research Agreement can be found at: http://www.ncagr.gov/agronomi/documents/Research_Project_Agreement_PWSM.pdf*

In addition to the standard analysis, certain tests are available by request for an additional fee per sample.

Additional tests available by request:

- Molybdenum (Mo) + $2
- Bulk Density (BD) + $10
- ǂ Heavy Metals: cadmium, nickel, lead, arsenic, chromium, selenium + $20

ǂ Note: heavy metals analysis is available only for research purposes with a valid research agreement from a university, government agency or private scientific research institute. NO EXCEPTIONS.)

Sampling instructions can be found here:
https://www.ncagr.gov/agronomi/documents/WaterMediaTestingforNurseryandGreenhouseManagers_.pdf

The Sample Submission form for growers can be found here:
http://www.ncagr.gov/agronomi/pdffiles/Soilless_Media_Sample_Submission_Form_Fillable.pdf

The Sample Submission form for researchers can be found here: http://www.ncagr.gov/agronomi/pdffiles/Soilless_Media_Sample_Submission_Form_Research.pdf

Bulk density sampling and submission instructions can be found here: https://www.ncagr.gov/agronomi/documents/BulkDensityTestingforNurseryandGreenhouseManagers_.pdf

Minimum Sample Volumes

In order to obtain a representative sample, NCDA&CS strongly recommends a sample volume of one quart of media. Two quarts is preferable. If requesting bulk density, submit four quarts. Where this is not possible, please note the minimum sample volume required to perform each analysis (Table 1).

Table 1. Soilless media methods summary with minimum volume required for each method.

Sample Test	Minimum Volume	Analytical Method	Reference
NO_3-N, NH_4-N, Cl^-	200 cm^3	SME extraction; Continuous Flow Analysis	EPA 350.1; EPA 353.1; EPA 325.2
P, K, Ca, Mg, S, Fe, Mn, Zn, Cu, B, Na, Al, Mo, As, Cd, Cr, Ni, Pb, Se	200 cm^3	SME extraction; ICP-OES	EPA 200.7
pH	200 cm^3	As received; pH meter	AOAC 973.41
EC/SS		SME extraction; EC meter	EPA 120.1
Bulk Density	2 L	As received; gravimetric	USDA TPA-103

Analytical Methods

Sample Processing & Storage

Prior to analysis, the primary sample is homogenized by manual mixing. Except during analysis, primary samples are refrigerated at 4 °C. Samples are analyzed as-received for pH and bulk density. All other methods (EC, ionic and elemental analysis) are performed following filtration (Whatman #1 filter paper; Fisher Scientific) according to the Saturated Media Extract (SME) method (Warncke 2011).

Inorganic Nitrogen: NO_3-N and NH_4-N

Nitrate-nitrogen (NO_3-N) and ammonium-nitrogen (NH_4-N) are determined on a 15 mL aliquot of filtrate which is manually shaken prior to analysis.

NO_3-N is determined by the hydrazine reduction method, where nitrate is reduced to nitrite with hydrazinium sulfate catalyzed by Cu^{2+}, under alkaline conditions and at elevated temperature (Kempers and Luft 1988). The NO_2-N concentration (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with α-naphthyl-ethylenediamine dihydrochloride to form a highly-colored azo dye which is measured at 540 nm (modified Griess reaction) (USEPA 1978b; Kempers and Luft 1988; Skalar Analytical 2018c). NH_4-N is based on the modified Berthelot reaction where, after oxidation and oxidative coupling, a green-colored complex is formed, which is then measured at 660 nm (Krom 1980; Skalar Analytical 2018a; USEPA 1993).

Both NO_3-N and NH_4-N are quantified by continuous flow analysis using an auto-flow spectrophotometric analyzer (San^{++} Segmented Flow Auto-Analyzer, Skalar Instruments; Breda, The Netherlands). Nitrate-nitrogen (NO_3-N) and nitrite-nitrogen (NO_2-N) are reported as NO_3-N on the Soilless Media Analysis Report. Ammonium-nitrogen (NH_3-N + NH_4-N) is reported as NH_4-N on the Soilless Media Analysis Report. Results are expressed in parts per million (ppm) [equivalent to mg L^{-1}].

Inorganic Nitrogen Quality Controls
Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually. **The MDLs for NH_4 and NO_3 are 0.87 ppm and 0.45 ppm, respectively.**

Samples are quantified using nine calibration standards. A method blank (DI water, filtered) is analyzed with each batch. A calibration verification solution and calibration blank are analyzed at the beginning and end of each batch and after every 10 samples. A duplicate aliquot of a filtered solution sample is spiked and analyzed for analytical recovery with each batch. Four independent calibration verification solutions are analyzed at the beginning and end of each run. Drift checks are analyzed at the beginning and end of each run and every 20 samples. Two nitrite checks (NO_2-N) are analyzed to verify the completeness of the nitrate reduction reaction at the beginning and end of each run.

Chloride (Cl^-)

Chloride concentration is determined on a 15 mL aliquot of filtrate which is manually shaken prior to analysis.

Cl^- is determined by the thiocyanate displacement method where thiocyanate is liberated from mercury (II) thiocyanate by the formation of soluble mercuric chloride. The liberated thiocyanate forms a red colored complex with ferric iron ions also present in solution (USEPA 1978a; Skalar 2018b). This complex is measured at 470 nm on a segmented flow analyzer (San^{++} Segmented Flow Auto-Analyzer, Skalar Instruments; Breda, The Netherlands). Results are expressed in parts per million (ppm) [equivalent to mg L^{-1}].

Cl Quality Control

Method detection limits (MDL) are determined when a new instrument or method is put into use and verified annually. **The MDL for Cl is 1.27 ppm.**

Samples are quantified using nine calibration standards. A method blank (DI water, filtered) is analyzed with each batch. A calibration verification solution and calibration blank are analyzed at the beginning and end of each batch and after every 10 samples. A duplicate aliquot of a filtered solution sample is spiked and analyzed for recovery. Four independent calibration verification solutions are analyzed at the beginning and end of each run. Drift checks are analyzed at the beginning and end of each run and every 20 samples.

Phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron (B), sodium (Na), aluminum (Al), molybdenum (Mo), cadmium (Cd), nickel (Ni), lead (Pb), arsenic (As), chromium (Cr), and selenium (Se)

Total elemental concentrations are determined on a 15 mL aliquot of filtrate, which is manually shaken prior to analysis, using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES) (Spectro Arcos EOP and Arcos II EOP, Spectro Analytical: A Division of Ametek; Mahwah, NJ) (USEPA 2001). Elements are measured at the wavelengths listed in Table 2. Results are expressed in parts per million (ppm) [equivalent to mg L^{-1}].

Table 2. Wavelengths used to quantify total elemental concentrations in soilless media by ICP-OES.

Element	Wavelength (nm)
Aluminum (Al)	396.152
Arsenic (As)	189.042
Boron (B)	208.959
Cadmium (Cd)	214.438
Calcium (Ca)	183.801, 315.887, 318.128
Chromium (Cr)	267.716, 357.869
Copper (Cu)	324.754
Iron (Fe)	259.941
Lead (Pb)	220.353, 405.778
Magnesium (Mg)	279.079
Manganese (Mn)	257.611
Molybdenum (Mo)	202.095
Nickel (Ni)	341.476
Phosphorus (P)	178.287
Potassium (K)	404.721, 766.491
Selenium (Se)	196.090
Sodium (Na)	330.237, 589.592
Sulfur (S)	182.034
Zinc (Zn)	213.856

ICP-OES Quality Controls
Elements are measured using a curve with at least five calibration points.

A method blank, calibration blank and reference material are analyzed with each batch. A calibration verification solution is run after the daily calibration, after every 10 samples and at the end of each run. An independent calibration verification solution is analyzed at the beginning and end of each run. The method detection limits (MDLs) for each analyte are listed in Table 3.

Table 3. Method detection limits (MDL) of total elemental concentrations in soilless media by ICP-OES.

Element	MDL (ppm)
Aluminum (Al)	0.025
Arsenic (As)	0.003
Boron (B)	0.025
Cadmium (Cd)	0.002
Calcium (Ca)	2.362
Chromium (Cr)	0.004
Copper (Cu)	0.006
Iron (Fe)	0.047
Lead (Pb)	0.006
Magnesium (Mg)	1.059
Manganese (Mn)	0.003
Molybdenum (Mo)	0.001
Nickel (Ni)	0.004
Phosphorus (P)	0.022
Potassium (K)	0.193
Selenium (Se)	0.004
Sodium (Na)	0.657
Sulfur (S)	0.048
Zinc (Zn)	0.010

pH
The pH of soilless media samples is determined directly on saturated samples at 25 °C according to Warncke 2011, using a Thermo Scientific Orion Versa Star Pro pH meter with a green epoxy non-fillable pH electrode with BNC connection (APHA 2012; AOAC 1990b). pH is a measure of acidity or alkalinity on a scale of 1 to 14 and is reported on this scale (unitless).

pH Quality Control
A three-buffer calibration is performed daily with a slope maintained between 98% and 102%. The three buffers are read back and recorded at the beginning and end of each day.

Electrical conductivity (EC)

Electrical conductivity (EC) is a measure of the ability of an aqueous solution to carry a current. The EC of an aqueous solution depends on the total concentration, mobility, and valence of ions and on the temperature of the sample. EC is measured on filtered samples at 25 °C (Warnke 2011). EC is measured using a conductivity meter and probe (SevenMulti; Mettler-Toledo, LLC; Columbus, OH) (AOAC 1990a). EC is also referred to as soluble salts (SS). EC is expressed in units of mS/cm and SS is expressed in units of 10^{-5} S/cm.

Electrical conductivity Quality Control

The EC meter is calibrated daily with a 1000 µS conductivity standard.

Bulk Density

Bulk density is determined on soilless media primarily for the purpose of calculating insecticide rates for the control of fire ants in nurseries and greenhouses. The media sample is dried at 80 °C for a minimum of 8 hours and a maximum of 24 hours and until sample weight is stable. Three 1 L sample aliquots are weighed, and the average reported as g cm_3^{-1} (USDA 2019).

References

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