

NCDA&CS Methods for Soilless Media Analysis



Plant/Waste/Solution/Media Laboratory

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Introduction

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 NCDA&CS Soilless Media Analysis Lab performs the following analyses:

- Phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), sulfur (S), iron (Fe), manganese (Mn), zinc (Zn), copper (Cu), boron (B), molybdenum (Mo), sodium (Na), aluminum (Al), arsenic (As), cadmium (Cd), chromium (Cr), nickel (Ni), lead (Pb) and selenium (Se)
- Nitrate-nitrogen (NO₃-N), ammonium-nitrogen (NH₄-N), and chloride (Cl)
- Soluble salts (SS or EC), pH, and bulk density

Sample Collection and Minimum Sample Masses

No matter how precise and accurate an analytical method is, meaningful results are only as good as the sample itself. The most significant cause of poor statistical results in analysis is due to imprecise sample collection and preparation rather than analytical measurement.

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). Sample collection guidelines are listed on our website: [Soilless Media Sample Collection](#).

Table 1. Soilless media methods summary and minimum volume required

Sample Test	Minimum Volume	Analytical Method	Reference
NO ₃ -N, NH ₄ -N, Cl ⁻	200 cm ³	SME extraction; Flow Injection Analysis	EPA 350.1; EPA 353.2; 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

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).

Analytical Methods

Inorganic Nitrogen: NO₃-N and NH₄-N

Nitrate-nitrogen (NO₃-N) and ammonium-nitrogen (NH₄-N) are determined on a 15 mL aliquot of filtrate which is manually shaken prior to analysis.

NO₃-N is determined by cadmium reduction, where nitrate is reduced to nitrite with copperized cadmium, under alkaline conditions. The NO₂-N concentration (that originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-Naphthyl) ethylenediamine dihydrochloride to form a magenta-colored azo dye which is measured at 520 and 600 nm (USEPA 1993a; FIA NO3-W-1-1).

NH₄-N is determined by the modified Berthelot reaction where hypochlorite and sodium salicylate react with ammonia in a two-step reaction, converting it to 5-aminosalicylate. The aminosalicylate is oxidized in the presence of sodium nitroferricyanide to form a blue-green colored complex, which is then measured at 660 nm (USEPA 1993b; FIA NO3-W-1-2).

Both NO₃-N and NH₄-N are quantified by flow injection analysis (FIAlyzer-3000, FIA Lab). Nitrate-nitrogen (NO₃-N) and nitrite-nitrogen (NO₂-N) are reported as NO₃-N on the Soilless Media Analysis Report. Ammonium-nitrogen (NH₃-N + NH₄-N) is reported as NH₄-N on the Soilless Media Analysis Report. Results are expressed in 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.

Samples are quantified using nine calibration standards. A method blank (DI water, filtered) is analyzed with each batch. A duplicate aliquot of a filtered solution sample is spiked and analyzed for analytical recovery 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. 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 (NO₂-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; FIA CL-W-1-1) This complex is quantified at 480 nm by flow injection analysis (FIAlyzer-1000, FIA Lab; Seattle, WA). Results are expressed in 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.

Samples are quantified using nine calibration standards. A method blank (DI water, filtered) is analyzed with each batch. A duplicate aliquot of a filtered solution sample is spiked and analyzed for analytical recovery 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. 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.

Elemental Analysis

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 mg kg⁻¹.

ICP-OES Quality Controls

Elements are measured using a curve with at least five calibration points.

A method blank (DI water, filtered) and calibration blank 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.

Table 2. Wavelengths used to quantify total elemental concentrations 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

pH

The pH of soilless media samples is determined directly on saturated samples at room temperature 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 and an automatic temperature compensation probe (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 at the beginning of each day.

Electrical conductivity

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 room temperature (Warncke 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. A duplicate solution sample is analyzed daily as a quality control sample with an acceptance criteria of <2% relative standard deviation.

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).

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