Soil sample analysis methods: A ready reckoner for soil testing

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High yields of top quality crops require an abundant supply of the 16 essential nutrient elements. In addition to providing a place for crops to grow, soil is the source for most of the essential nutrients required by the crops. Our soil resource can be compared to a bank where continued withdrawal without repayment cannot continue indefinitely. As nutrients are removed by one crop and not replaced for subsequent crop production, yields will decrease accordingly. Accurate accounting of nutrient removal and replacement, crop production statistics, and soil analysis results will help the producer manage fertilizer applications. A soil analysis is used to determine the level of nutrients found in soil sample. As such, it can only be as accurate as the sample taken in a particular field. The results of a soil analysis provide the agricultural producer with an estimate of the amount of fertilizer nutrients needed to supplement those in the soil. Applying the appropriate type of needed fertilizer will give the agricultural a more reasonable chance to obtain the desired crop yield.

Essential nutrient elements in soils

Nutrient	Symbol	Form available	Category	Function in Plants
Carbon	C	CO_2	non- fertilizer element supplied through air, water, and soil nutrients	make up the bulk of the plant weight.
Hydrogen	H	H_2O		
Oxygen	O	O_2		
Nitrogen	N	NO_3, NH^4_+	macronutrients required by plants in large amounts	proteins, amino acids
Phosphorus	P	PO_4^{-3}		nucleic acids, ATP
Potassium	K	K^+		catalyst, ion transport
Calcium	C	C^{++}	secondary nutrients required by plants in moderate amounts	cell wall component
Magnesium	Mg	Mg^{++}		part of chlorophyll
Sulfur	S	$SO_4^=$		amino Acids
Boron	B	HBO_4^-	micronutrients required by plants in small amounts	cell wall components
Chlorine	Cl	Cl^-		Photosynthesis reactions
Copper	Cu	Cu^{++}		component of enzymes
Iron	Fe	Fe^{+2}, Fe^{+3}		chlorophyll synthesis
Manganese	Mn	Mn^{++}		activates enzymes

Molybdenum	Mo	$MoO_4^{=}$		involved in N fixation
Zinc	Zn	Zn^{++}		activates enzymes

Rating of essential elements in soil

Sl. No.	Item	Low	Medium	High
1.	organic carbon used as a measure of available nitrogen.	below 0.5 %	0.5 to 0.75 %	above 0.75 %
2.	available N alkaline permanganate method in kg/ha	below 280	280 - 560	above 560
3.	available P by Olsens method in kg/ha	below 10	10 – 24.6	above 24.6
4.	available K in kg/ha	below 108	108 - 280	above 280
5.	pH	acidic	normal	alkaline above
6.	conductivity (total soluble salts) millimhos			
	below<1	Normal		
	1-2	soluble salt content critical for germination		
	2-3	salt content critical for growth of salt sensitive crops		
	above 3	severe injury to most crops		
Micro Nutrients	zinc	< 0.5 ppm	0.5-1.0 Ppm	> 1.0 ppm
	iron and Manganese	< 5 ppm	5-10 ppm	>10 ppm
	copper	<0.2 ppm	0.2-0.4 ppm	>0.4 ppm

Objectives of soil analysis

The objectives of soil analysis are as follows: to provide an index of nutrient availability or supply in a given soil-the soil extract is designed to evaluate a portion of the nutrients from the same "pool" used by the plant; to predict the probability of obtaining a profitable response to fertilizer application- low analysis soils may not always responds to fertilizer applications due to other limiting factor; to provide a basis for fertilizer recommendations for a given crop; to evaluate the fertility status of the soil and plan a nutrient management program.

Preparation and use of chemical reagents

The best quality chemical reagents available should be used normally, "analytical reagent grade" (AR) or in some case "laboratory grade" (LR). For most laboratory purposes, water distilled in a borosilicate glass still or in a tin still will be satisfactory. For preparing some reagents, dilution water requires special treatment like second distillation, boiling to drive off excess carbon

dioxide, etc. where such special treatment is necessary, it will be stated. Procedures for the preparation of reagents usually give direction for the preparation of a one liter volume. For those reagents that are not often used, smaller volume should be prepared by mixing proportionally smaller quantities than those give in the procedure. Where a working standard or working solution is to be made by dilution of stock solution, no more of the stock solution should be prepared than will be used within the next six months. Furthermore, only amount of stock solution necessary to meet the immediate need for the working or standard solution will be diluted at one time. Reagents solution should be kept in tightly stoppered glass bottles, (except where they are incompatible with glass, as with silica solutions). Rubber or neoprene stoppers or screw tops with gaskets are suitable, provided that the reagents do not react with these materials. For short term storage, small quantities of sample may be transported in plastic bottles with plastic screw cap. Reagent containers should always be accurately labeled with the name of the reagent, its concentration, the date that it was prepared and the name or the initials of the person who prepared it.

Soil sampling format for different land use

Sample	Depth	Parameter	Methods
Soil from different land use	0-15 cm, 15-30 cm, 30-60 cm and 60-1 m	pH	potentiometric method
		bulk Density	core Method
		soil Moisture	gravimetric Method
		soil Texture	hydrometer Method
		soil Organic Carbon	walkley and Black Method
		available nitrogen	alkaline permanganate method
		available phosphorus	brays method
		available potassium	neutral normal ammonium acetate method
		available micronutrients (Zn, Cu, Fe, Mn),	DTPA extract method
		soil Microbial Biomass Carbon	chloroform Fumigation method
		total organic carbon	TOC analyser
		carbon stock	OC × BD × Depth

Soil pH (potentiometric method)

Reagents: buffer solution of ph 4.0, 7.0 AND 9.2

Procedure:

1. Weight 10 g soil

2. Add 25 ml distilled water and stir for 30 minutes
3. Calibrate the pH meter with two standard buffer solutions
4. Immerse the electrodes of the pH meter in the soil sample and read the pH of the sample
5. Wash the electrodes with distilled water every time for new sample

Soil Texture (hydrometer method)

Apparatus:

1. Hydrometer
2. Mechanical stirrer
3. Thermometer
4. Suspension cylinder with 34=+-2 cm height upto 1000ml mark

Reagent:

1. 10% sodium hexametaphosphate/calgon solution: Dissolve 100g of sodium hexametaphosphate in distilled water and make to 1L. Filter through Whatman No. 1 filter paper.

Procedure:

1. Take 100 g of soil sample in 500ml beaker. Give H2O2 treatment to destroy the organic matter. Skip the step if the sample had < 1% organic matter.
2. Add 200 ml of distilled water, 100 ml of the solution hexametaphosphate solution, stir well with the help of glass rod, cover and keep for 4-5 hrs (preferably over night).
3. Transfer the contents quantitatively to the cup of mechanical stirrer, giving 4-5 washing of distilled water to transfer all soil particles, bringing the volume to about 500 ml.
4. Run the stirrer for 10minutes and transfer the contents to the suspension cylinder (1000ml), giving 4-5 washing of distilled water and make the volume to the mark.
5. Put the rubber stopper tightly and invert the cylinder carefully. Vigorously shake several times to allow the soil particles to disperse completely.
6. Keep the cylinder on the table, remove the stopper and bring back adhering liquid on the stopper by touching it to the side of the cylinder. Immediately place the hydrometer in the suspension, gently checking any up and down movement.
7. Record the reading exactly 40 seconds after placement of hydrometer.

8. Replace the rubber stopper and invert several times again to ensure complete dispersion of particles. Keep it on the table.
9. Exactly after 2 hours, place the hydrometer again into the suspension and note the hydrometer reading.
10. Simultaneously run a blank without soil and record the room temperature in °F [(temperature recorded in °C × 1.8) + 32 = Temperature in °F].
11. In case the surface of the suspension in the cylinder is not clear due to foam, add 2-3 drop of amyl alcohol.

Calculation:

Correction factor (CF) = (Actual room temperature in °F -68) × 0.2

Where, Sand B stands for sample and blank reading, respectively, taken at 40 seconds (first reading)

Where, s and b stand for sample and blank reading, respectively, taken after 2 hours (second reading)

Per cent sand content can be calculated by difference.

i.e. percent sand= 100-(per cent silt + clay)

With the help of then triangular diagram, the textural class name is assigned using the percent content of sand, silt and clay particles.

For using the triangular diagram, take the following steps:

1. Locate the per cent clay content of the sample on the left hand side and move horizontally.
2. Locate the per cent silt content of the sample on the right hand side and move on slanting line. Alternatively, sand content instead of silt may be located (on the bottom side).
3. Find the point where the two lines cross each other to get the textural class.

Bulk Density (core method)

Procedure:

1. Remove 1-2 cm of surface soil from the spot where sample will be taken, and level the spot.
2. Drive a 5 cm diameter thin sheet metal tube of known weight (W1) and volume (V) 5 cm into the soil.

3. Excavate the soil from around the tube and cut the soil beneath the tube bottom.
4. Trim excess soil from the tube end.
5. Dry at 105°C for two days and weight (W2)

Calculation:

Soil Moisture (gravimetric method)

Procedure:

1. Weight 10 to 20 g of fresh sample into a dry weighed (or tare) evaporating basin.
2. Placed in an air circulation oven at 105°C and dry to a constant weight successive weighing should not differ by more than 1 to 2 mg)
3. Cool in Desiccators and weigh.
4. Calculate percentage fresh moisture from loss in weight.

Note: 1. The sample should not be sieved before weighing but large stones sand roots should be removed.

1. Solution 'B' to 100 ml.

Soil organic carbon (walkley black method)

Reagent:

1. 1N $K_2Cr_2O_7$ (49.04 g in 1 lt.).
2. 85% H_3SO_4.
3. Concentration H_2SO_4.
4. Diphenylamine indicator (0.5 g diphenylamine in a mixture of 20 ml H_2O and 100 ml conc. H_2SO_4).
5. 0.5 N FAS (Mohr's salt), add 40 ml conc. H_2SO_4 and make vol. to 2 lt.

Procedure:

0.5 g soil + 10 ml 1N $K_2Cr_2O_7$

↓

Add 20 ml conc. H_2SO_4 and wait for half an hour

↓

Pour 200 ml H_2O

↓

Add 10 ml 85% H3PO4 and 1 ml diphenylamine indicator

↓

Back titrate 0.5 N FAS

↓

Carry a blank titration

Where B= Blank reading, T= sample reading

Available Nitrogen (Alkaline Permanganate Method)

Reagent:

2% Boric Acid (heat & mix, cool put mix indicator @ 20ml/lt)

Mixed indicator = 0.1 g bromocresol green & 0.04 g methyl red in 100 ml C_2H_5OH)

Boric acid pH 4.5 (NaOH/ H_2SO_4)

0.32 % KMnO4

2.5% NaOH

Procedure:

a. 5 g soil + little paraffin-wax + little water from the side.
b. Add 25 ml KMnO4 + 25 ml NaOH put in distillation tube.
c. Put 25 ml boric acid in conical flask & place it under the pipe to collect ammonia. Distilled for 9 mins. Back titrate with N/50 N H_2SO_4.

Calculation:

Available phosphorus (brays method modified)

Extracting solution:

Dissolve 2.775 g of NH_4F in 2.5 lt of 0.025 N HCl; 5.36 ML concentrated HCl make up the volume to 2.5 lt. (4.29 ml to 2 lts.) 100 ppm P standard solution: - 0.4434 g KH_2PO_4 (potassium dihydrogen phosphate) in 1lt. volumetric flask and dilute it and add 408 ml of concentrated H_2SO_4 and make up the volume to 1 lt.

2 ppm P solution: - 5 ml of 100 ppm P solution in 250 ml volumetric flask and make up the volume by distilled water.

Pipette out 0, 1, 2.5, 5, 7.5, 10, 12.5 ml of 2 ppm P solution (0, 0.08, 0.2, 0.4, 0.6, 0.8 & 1 ppm) + 4 ml ascorbic acid indicator and make up the volume to 25 ml by distilled water.

Reagent:

1. 27.78 ml concentrated H_2SO_4 + H_2O = 200 ml.
2. Ammonium molybdate: 20 g Ammonium Molybdate dilute to 500 ml of d. H_2O.
3. Ascorbic acid (0.1 M): 0.2743 g of pot. Antimony tartarate, dilute to 100 ml with 100 d. H_2O.
4. Potassium antimony tartarate: 0.2743 g of pot. Antimony tartarate, dilute to 100 ml with 100 d. H_2O.
5. Mixed indicator: 125 ml of 5 N H_2SO_4 + 37.5 ml ammonium molybdate + 75 ml ascorbic acid + 12.5 ml of potassium antimony tartarate.

Procedure:

a. 2.5 g soil + 25 ml extractant (shake 5 min).
b. Filter it no. 42.
c. 5 ml aliquot + 5 ml ascorbic acid indictor (mixed reagent).
d. Volume makes up upto 25 ml d. H_2O.
e. Take reading @ 882 nm.

Available potassium (neutral normal ammonium acetate method)

Reagent:

Neutral 1 N NH_4OAC solution: dissolve 77.09 g of ammonium acetate in distilled water H_2O and make up the volume to 1lt. pH 7 (acetic acid or NH4OH)

Procedure:

a. 5 g soil + 25 ml 1 N NH4OAC.
b. Shake for 5 min.
c. Feed the sample in flame photo metre.

Standard curve:

Stock solution = dissolve 1.908 g KCl (dried @60°1 hrs.) 1 lt make up by distilled water (1000 ppm). From the stock make 20, 40, 60, 80 and 100ppm by adding 2, 4, 6, 8 and 10 ml and make up 100 ml.

Calculation:

1. Avail. K ppm = c (reading) × dilution/5).
2. Avail. K kg/ha = c × 5 × 2.24.
3. Avail. K2O kg/ha = c × 5 × 2.24 × 1.20.

DTPA- $CaCl_2$ –TEA extraction method for Zn, Cu, Fe and Mn available Zn

Instruments:

1. Atomic absorption spectrometer.
2. Mechanical shaker.

Reagents:

1. Dilute HCl: dilute AR grade HCl 5 times with double distilled water (DDW).
2. DTP extractant: AR grade diethyline-triamine-penta acetic acid (DTPA) 1.967 + 1.47 g CaCl2, 2H2O + 25 ml DDW + 13.3 ml triethanolamine (TEA) + 100 ml DDW transfer the solution to 1 lt. volumetric flask before make the volume. Adjust pH 7.3 (adjust by diluted HCl).
3. Standard stock solution 'A' (100 mg Zn L^{-1}): weigh exactly 1 g of pure Zn metal (AR grade) and dissolve it in minimum volume (about 10 ml) of dil. HCl (1:1) and make the volume 1 lt.
4. Standard stock solution 'B': dilute 5 ml of sol^n A to 100 ml to get sol^n B containing 50 mg Zn L^{-1}.
5. Standard working solution: dilute 0.5, 1.0, 1.5, 2, 2.5 and 5.0 ml portions of solution B to 50 ml to get working standard containing 0.5, 1.01.5, 2, 2.5 and 5.0 mg Zn L^{-1} the working standard should be prepared in the medium of the extracting solution after every few days as these can't be preserved for long.

Procedure:

1. 10 g soil in 100 ml conical flask.
2. Add 20 ml of DTPA.
3. Shake for 2 hrs. (Mechanical Shaker).
4. Filtration through whatman no. 42.
5. Use the filtrate for Zn measurement on AAS.
6. Feed the standard working soln and prepare a standard curve by ploting AAS reading against Zn concentration.

Calculation:

Where, 'A, standard for the Zn concentration in aliquot as read from X-axis of standard curve against the sample reading.

Available Cu

1. 1 g Cu + 50 ml HNO3 (1 Acid: 1 DDW) → make up the volume 1 lt.
2. Solution 'A' containing 1000 mg Cu L^{-1}
3. Solution 'B' 50 mg by diluting appropriate volume of solution 'A' finally prepare working solution containing 0.25, 0.50, 1.0, 1.5, 2.0 and 2.5 mg Cu L^{-1} from solution 'B'.

Available Fe

1. 1 g Fe metal + 50 ml HNO3 (1 Acid: 1 DDW) → make up the volume 1 lt.
2. Solution 'B': 50 ml of solution A to 500 ml to get 100 mg Fe L^{-1}.
3. 1, 2, 3, 5 &10 mg Fe L^{-1} from B.

Available Mn

1. 1.583 g MnO_2 + 1 g metal Mn + 50 ml HNO_3 (1 Acid: 1 DDW) → make up the volume 1 lt.
2. Solution 'A' containing 1000 mg Mn L^{-1}.
3. Solution 'A' dilutes 25 ml to 250 ml with DDW to get solution 'B' having 100 mg Mn L^{-1}.
4. 0.5, 1, 2, 2.5, & 5 mg Mn L^{-1} concentration by diluting 0.5, 1, 2, 2.5 and 5 ml portions of solution 'B' to 100 ml.

Estimation of Microbial Biomass Carbon (Chloroform fumigated method)

Reagent:

1. 0.5 M K_2SO_4: 174.25 g in 2 lt.
2. 0.2 M $K_2Cr_2O_7$: 9.18 g in 1 lt.
3. 0.5 M FAS:
4. 8.5 % H_3PO_4:
5. Diphenylamine indicator = 0.5 g Diphenylamine + 20 ml water + 100 ml H_2SO_4

Procedure:

25 g soil sample (2 replican) (one fumigated and other non-fumigated)

↓

Fumigate for 24 hour by keeping in vacuumed incubates-chloroform (pressure 0.4 for, 10-15 min)

↓

After fumigate add 40 ml K_2SO_4 and shake for half an hour and filter

↓

Take 10 ml aliquot

↓

Add 2 ml $K_2Cr_2O_7$ + 10 ml H_2SO_4

↓

Leave for ½ hour

↓

Add 10 ml d. H2O + 5 ml H_3PO_4 + 1 ml indicator

↓

Titrate with FAS (dark blue to light green)

↓

Carry a blank titration

1. N_1= actually normality of FAS
2. Volume of free K2Cr2O7 consumed by FAS in any sample V1
3. V_1 (NF)=
4. V_2(F) = 2 – V_1 (F) ; V_2 (NF); 2 for $K_2Cr_2O_7$ 1 ml of 1 M $K_2Cr_2O_7$ oxidised 0.0006 g C So, µg C – 1 ml × 100
5. VS = Volume of soil Moisture (%) × volume of extracting (40 ml)
6. Formal EC (/g) of soil
7. Extractable carbon, EC (/ml) EC (F) -; EC (NF) =; Because, 10 ml aliquot
8. Where, K = constant and EC = 0.48

TOC (modified wet oxidation titration)

1. Digestion mixture: 39.22 $K_2Cr_2O_7$ in 800-900 ml DW. Add 1000ml concentration H_2SO_4. Make up volume upto 2 lt. with DW.
2. 0.2 N FAS: 157 g FAS in 1000 ml DW. Add 100 ml concentration H_2SO_4. Make the volume to 2 lt. with DW.
3. Indicator: 3 g O-Phenanthraline monohydrate: Add 1.4 g FAS (heptahydrate). Dissolve in 200 ml DW

   or

   0.1 g N phenylanthrahilic acid + 0.1 g Na2CO3. Make volume to 100 ml with DW.

Procedure:

1. 0.5 g soil in 250 ml digestion tube adds 15 ml digestion mixture.
2. Place the sample into a preheated block digestor for 45 mins (150). Cool it
3. Add 50 ml DW
4. Add 5 ml ortho-phosphoric acid
5. Add 4 drops indicator
6. Titrate (0.2 N FAS)

Blank: 2 heated and 2 unheated blank

Calculation:

Carbon Stock:

Conclusion

Fertility of a soil can be assessed by analyzing various available nutrients present in the soil. Fertilizer recommendations for various crops and cropping sequences can be made on the basis of fertility status of a soil and targeted yield equations. Besides this, problematic soil can be ameliorated on the basis of soil test values. Proper identification of the sources (point/diffuse) is required before interpretation of the data set. However, soil being the major source of nutrients for crops can also provide support to the plant growth. Hence, soil health and its maintenance are the key issues to sustain crop productivity, which is assessed by the quality indicators and sustenance of the crops grown on them. However, the policy may be framed on the platform

based on “strategic and fundamental research” for developing innovative models in agricultural systems.

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