



RAPID SOIL FERTILITY SURVEY & SOIL TESTING INSTITUTE

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To

1. Agricultural Chemists

Soil & Water Testing Laboratories for Research in Punjab

2. Assistant Agricultural Chemists

Soil & Water Testing Laboratories in Punjab

Subject: **SFRI-GUIDE-2: SOIL AND WATER ANALYSIS MANUAL (2021)**

By the grace of Almighty Allah, who enabled us to write another excellent booklet, "SFRI-GUIDE-2: SOIL AND WATER ANALYSIS MANUAL (2021)," which is the most pressing need of the time and almost completely covers all aspects of precautions and instructions for analyzing soil and water samples in order to eliminate all flaws in the process of dealing with advisory and project samples. It includes all of the standard test methods, as well as the following, that will be used uniformly in all district and divisional SWT laboratories.

1. Basic guidelines for laboratory activities
2. SOPs: soil sample receiving , handling , analysis and storage
3. SOPs: for water samples under advisory / research services
4. SOPs: for discarding of advisory soil samples
5. SOPs for discarding of advisory water samples.
6. SOPs for discarding of project extension services 2.0 soil samples

You are required to follow all instructions and conduct all tests according to standard procedures in order to obtain consistent results with minimal variations. Please print and distribute this copy to all of your analysts for strict enforcement in order to improve the precision with which soil and water samples are analyzed for accuracy, repeatability, and reliability, which is our primary goal.


(DR MUHAMMAD AKRAM QAZI)
Chief Scientist/ Director
Soil Fertility Research Institute, Punjab,
Lahore

CC.

1. Ps to Additional Secretary (Admin), Agriculture Department, Punjab, Lahore.
2. Chief Scientist/Director General Agriculture (Research), AARI, Faisalabad



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2021

SFRI-GUIDE-2: SOIL AND WATER ANALYSIS MANUAL

Compiled and edited by Dr. Muhammad Akram Qazi

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Funded by the Soil Fertility Research Institute, Punjab, Lahore

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Important Disclaimer

A reasonable care was taken to make the information in this SFRI-Guide accurate and up-to-date and in accordance with the ISO/IEC 17025:2017 international standard and Standard Operating Procedures (SOP) issued by the Directorate, Soil Fertility Research Institute, Punjab, Lahore from time to time. However, authors would appreciate any comments and suggestions for further improvement of this SFRI-Guide.

Authors accept no liability whatsoever, by reason of negligence or otherwise, arising from any use or release of information in, or referred to in, this guide, or any error, inaccuracy, or omission in the information.

The analysis methods described in this guide need verification to ensure that the laboratory is capable of meeting the test method performance specifications. Verification of a test method demonstrates that the laboratory has met the test method's performance specifications and must be completed before the method is used for routine testing.



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■FOREWORD

The goal of this Soil, Water, and Plant Analysis Manual is to ensure that all divisional and district soil and water testing laboratories working under the command of the Rapid Soil Fertility Survey and Soil Testing Institute Punjab, Lahore, follow the standard approved procedures. During an inspection of soil and water testing laboratories throughout Punjab, it was observed that the different work strategies and methods for analysis, storage, handling, and discarding soil and water samples were being used in laboratories. A need was identified for the development of a guide that would assist and assist scientists in performing analysis using this guide in order to harmonize work strategy and analysis methods across all SWT laboratories.

This SFRI-Guide-2 will be followed in all soil and water testing laboratories throughout the province of Punjab, assisting in the harmonization of analytical work.

I hope you will find this SFRI-Guide-2 useful, and any suggestions for improvement will be greatly appreciated.

Director

Soil Fertility Research Institute, Punjab, Lahore



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■ **ACKNOWLEDGEMENT**

We consider it an honour to be able to express our thoughts and feelings, so we put together a guide. Dr. Asad Rehman Gillani, our Department Head, deserves special recognition for improving our work in soil and water analysis. We gratefully acknowledge him for assisting our laboratories to the best of his ability and authority.

FUTURE WORK STRATEGY OF SFRI

- 1. To improve the efficiency of service delivery of all divisional and district labs.*
- 2. In all labs, all laboratory tests will be performed in accordance with uniform standard test methods.*
- 3. To improve the handling of fertilizer samples collected as part of an anti-adulteration campaign for accuracy, repeatability, and reliability.*
- 4. By implementing reforms in the field wing, the institute's original mandate of formulating fertilizer recommendations will be made more efficient and authenticated. InSha Allah*



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■ INTRODUCTION

True understanding and interpretation of any field of study is based on the careful correlation of facts and the logical conclusions that can be drawn from them. Efficient laboratory work will provide you with unique and stimulating opportunities to learn about soil science in painstaking depth. Scientists have established standards that unify the disciplines of soil science and the concepts that offer existing interpretations as a result of such scientific insights. This is the proper scientific procedure document to follow. To summarize, this manual was created with the following goals in mind:

- a. To ensure the safety and health of those who work in research laboratories.
- b. To choose method that is quick, adaptable for routine analysis, and capable of producing consistent results when used by individual and multiple laboratories.
- c. Use uniform methods of soil sample collection, processing, and analysis so that the results obtained by different analysts can be compared.
- d. To establish a standard for interpreting the results of soil, water, and plant tests.
- e. To make site-specific fertilizer recommendations based on the results of soil tests. This will not only reduce the negative effects of any applied nutrient on the environment, but it will also improve production efficiency and quality.

Although a very thorough compilation of these methods has been made in writing, if any recent developments, flaws, or faults are revealed, the opinion will be greatly appreciated.

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■ BASIC GUIDELINES FOR LABORATORY ACTIVITIES

The methods described in this manual may or may not address all safety and health concerns. It is the responsibility of laboratory staff to establish appropriate safety and health practices in due course in order to avoid any laboratory injuries and fatalities.

● IMPORTANT GENERAL INSTRUCTIONS

- 1. Lab notebooks should be painstakingly maintained to ensure complete traceability of results both chronologically and by lab registration, and each and every activity performed in the lab should be depicted and easily traceable. Supervisors will record their visits to the lab and keep track of them in the lab note books.*
2. Each register, log, and record will prominently display the purpose for their formation and be page certified.
3. Log books will be kept for the date of use and the number of samples to be run on each piece of laboratory equipment. The date of use and the number of samples to be run on each piece of laboratory equipment should be depicted and cross-referenced with the number of samples performed in the lab note book.
4. A register of chemical consumption should also be maintained.
5. Each chemical should be clearly labeled with the date it was opened and the date it will expire.
6. Each batch will have a reference sample run and any correction factor that appears to be within limits should be incorporated and documented in the lab note book.
7. If a significant difference in the results is observed, the sample will be repeated and the results documented in the notebook.
8. The same data that was recorded in the lab notebook must be carefully transferred to the data entry register and included in the final report.
9. No lead pencils are to be used in the note book or in the register.
10. No use of loose papers for documenting readings, not even for rough calculations.
11. Each sub lab will be clearly labeled with the purpose for which it is being used, as well as with the lab number.
12. Only authorized people will be allowed in the lab, and no analyst will be allowed to use it as a sitting area for guests.



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- **GUIDELINES FOR PERSONAL PROTECTION**

1. Read the safety precautions and laboratory rules carefully and adhere to them. These will assist you in safeguarding not only your own interests, but also those of other laboratory personnel.
2. During lab work, personal protective equipment (spectacles, goggles, safety shields, etc.) must be worn at all times.
3. If noise is likely to damage or impair hearing, hearing protection should be made to wear.
4. Long hair must be properly covered or restrained.
5. In laboratories, children are not permitted.
6. Before beginning analytical work, read all applicable MSDS and make sure that eyewash facilities, safety showers, spill kits, and a first aid box are readily available.
7. Always wear closed-toed footwear. Thongs, sandals, and bare feet are not permitted.
8. When using machinery such as a mechanical shaker or soil grinder, safeguard loose clothing.
9. In the laboratory, it is forbidden to eat, drink, or use cosmetics.
10. Mobile phones and other devices that could cause a distraction during laboratory operations should be left outside.
11. Do not run or act hastily in laboratories.
12. When handling chemicals or other biological materials, cover all open wounds.
13. Pipette by mouth is not recommended. Instead, use mechanical pipetting devices.
14. Lifting heavy objects should be avoided. When appropriate, use lifting devices and trolleys.
15. Without prior instruction and training from the supervisor or technical staff, do not use any machine, equipment, or laboratory apparatus.
16. Install safety signs and ensure that they are adhered to at all times.
17. Remind yourself to thoroughly wash your hands before leaving the lab/handling food during lunch breaks.

- **CHEMICAL AND HAZARDOUS SUBSTANCE USAGE GUIDELINES**

1. Make sure all containers in the lab are clearly labeled. At the very least, this label should include the chemical/name, solution concentration, intended use, and preparation and expiration dates. Refer to the certificate of analysis in the case of CRM.
2. When transporting glass or containers, always use safety carriers such as buckets.
3. Treat all substances as hazardous unless you have specific evidence to the contrary.
4. Perform work in fume hoods in accordance with the Material Safety Data Sheets (SDS).
5. Avoid accumulating flammable materials in the laboratory.
6. Keep only the bare minimum amount of chemicals required in the laboratory work area.



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7. Avoid storing flammables in domestic refrigerators. Store flammables in a spark-proof refrigerator.

- **CLEANING AND MAINTENANCE GUIDELINES**

1. Read the safety precautions and laboratory rules carefully and follow them. These will assist you in safeguarding not only your own interests, but also those of other laboratory staff.
2. Keep the floor clean and dry. Keep your work bench organized so you can find what you need quickly. If a substance is spilled, clean it up as soon as possible. If you do not, you risk damaging or destroying your clothing, skin, books, or papers.
3. After each use, thoroughly clean the work area and equipment.
4. Maintain clear aisles and exits.
5. Ensure that emergency equipment is easily accessible (fire extinguisher etc.)
6. When leaving the laboratory, turn off all operating equipment (if applicable), extinguish flames, and so on.
7. Maintain the operating manuals for the laboratory equipment provided by the supplier.
8. The laboratory environment must be suitable for laboratory activities and not impair the validity of results. Laboratory activities require specific environmental conditions, which must be documented (ISO-17025: 2017)
9. Areas with incompatible laboratory activities should be separated.
10. Each piece of chemical laboratory equipment serves a specific purpose; for example, a graduated cylinder is intended to be used as a device for measuring liquid volume rather than as a reaction vessel. First, familiarize yourself with the equipment by carefully reading the operating manual and displaying the work instructions for each piece of equipment near it.
11. Electric fan heaters and kerosene heaters are prohibited.
12. Read the research protocol thoroughly before you come to the laboratory. Laboratory work is significantly more valuable, and if you are properly oriented with procedure, you can make more efficient use of your time.
13. Discuss the analysis in order to obtain more precise research work. Scientists gain a great deal from their interactions with one another.
14. Distilled water should be used for all analytical work.
15. Use all reagents in a cost-effective and precise manner. Take care of the laboratory equipment and physical facilities..



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■ **SOPs: SOIL SAMPLE RECEIVING, HANDLING, ANALYSIS AND STORAGE**

1. The head of the laboratory will be the custodian of the sample upon receipt from the source (customer) and will keep a close eye on the activities of the responsible officers/officials.
2. He may appoint any officer or official on his behalf to obtain the sample, and the concerned officer or official shall be held responsible for maintaining the record in accordance with SOPs and ensuring the accuracy and presence of the sample received.
3. The sample is obtained by the laboratory staff at the request of the farmer or brought to the laboratory for analysis by the farmer himself (Appendix-9).
4. Samples will be collected at the Sample Collection Desk of the laboratory.
5. The approved rate list should be prominently displayed in the laboratory's sample receiving room.
6. The individual who receives the samples at the laboratory shall inspect the samples and fill in Proforma, shall charge for the analysis of the samples at the notified rate, shall issue the receipt of the fee and shall record the farmer & the details of the samples.
7. The sample will be entered into the Soil Sample Receipt Register, and the sample analysis order sheet will be prepared and sent to the relevant analyst for analysis, with the analyst being responsible for the analytical results (Appendix-13).
8. Lab notebooks should be painstakingly maintained to ensure complete traceability of results both chronologically and by lab registration, and each and every activity performed in the lab should be depicted and easily traceable. Supervisors will record their visits to the lab and keep track of them in the lab note books.
9. Each register, log, and record will prominently display the purpose for their formation and be page certified.
10. Log books will be kept for the date of use and the number of samples to be run on each piece of laboratory equipment and should be depicted.
11. On the basis of the analytical results, the data will be entered in the Soil Analysis Data Register, and a soil analysis report will be generated and signed by Assistant Agricultural Chemist and then Agricultural Chemist if the Divisional Laboratory (Appendix-12).
12. Within 14 working days, the sample analysis will be completed, and a report will be mailed or emailed to farmers, along with full fertilizer recommendations for the targeted crop (Appendix-5).
13. After the completion of the analysis, after issuing / sending the report to the farmer/research organization, the samples will be disposed of after 30 days.



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■ SOPs: WATER SAMPLES UNDER ADVISORY / RESEARCH SERVICES

1. The Head of the Laboratory will be the custodian of the samples once they have been received from the source (customer) and will keep a close eye on the activities of the responsible officers/officials. He can appoint any officer or official to receive the sample on his behalf, and that officer or official will be held accountable for maintaining the record in accordance with SOPs and ensuring the accuracy and presence of the sample received.
2. A sample will be collected by the lab staff at the request of the farmer or brought to the laboratory by the farmer himself for analysis (Appendix-9).
3. Samples will be collected at the laboratory's Sample Collection Desk.
4. The person receiving the samples at the laboratory will inspect the sample and record the details of the farmer and the sample in a Proforma.
5. The sample will be recorded in the Water Sample Receipt Register (Appendix-10).
6. Data will be entered into the Water Analysis Data Register after the sample analysis is completed (Appendix-11).
7. The sample analysis will be completed in 5 working days, and the farmer will receive a report with all of the recommendations (Appendix-7).
8. After the analysis is completed, the sample will be discarded 30 days after the report is issued / dispatched to the farmer.

■ SOPs FOR DISCARDING OF ADVISORY SOIL SAMPLES

1. Samples will be prepared, result data entered in Data Register to the satisfaction of the analysts (repeating analysis if necessary), and result reports and recommendations to the farmers will be issued within 15 working days.
2. A committee of three members will be formed by the laboratory heads.
3. After 30 days, the committee will compile a list and submit it to the head for approval.
4. Prior to discarding samples, approval from a centrally constructed committee by DRSF is also mandated.
5. Samples will be discarded after the head has given their approval.
6. A proper record will be kept.

■ SOPs FOR DISCARDING OF ADVISORY WATER SAMPLES

1. Within 05 working days, the sample will be analyzed, prepared, and the results data will be entered into the Data Register to the satisfaction of the analysts (repeating the analysis if necessary). Finally, result reports and recommendations to the farmers will be issued.
2. A committee of three members will be formed by the heads of the laboratories.
3. Prior to discarding samples, approval from a centrally constructed committee by DRSF is also mandated.



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4. After 30 days, the committee will compile a list and submit it to the head for approval.
5. Samples will be discarded once the heads have given their approval.
6. A proper record will be kept.

■ SOPs FOR DISCARDING OF PROJECT EXTENSION SERVICES 2.0 SOIL SAMPLES

1. Samples are prepared, analyzed, and result data is entered into the Data Register to the satisfaction of the analysts (repeating the analysis if necessary), and result reports and farmer recommendations are issued.
2. A three-member committee will be formed by the heads of the laboratories.
3. Prior to discarding samples, approval from a centrally constructed committee by DRSF is also mandated.
4. After 60 working days, the committee will compile a list and submit it to the head for approval.
5. Every day, check samples will be used in each batch, and data will be kept.
6. The Atomic Absorption Spectrophotometer / Spectrophotometer / Flame photometer library, as well as any other equipment, will be kept for at least 6 months.
7. Samples will be discarded after approval from the heads.
8. A proper record will be kept.

■ COMMITTEE FOR RECHECKING OF SOIL, WATER AND PROJECT EXTENSION SERVICES 2.0 SAMPLES

1. Agricultural Chemist (Training), Directorate of Rapid Soil Fertility Survey & Soil Testing Institute, Lahore
2. Soil Fertility Officer of the respective region.
3. Assistant Soil Fertility Officer-II, Directorate of Rapid Soil Fertility Survey & Soil Testing Institute, Lahore

TORs OF THE COMMITTEE

4. The committee will recheck the soil and water samples which have previously been analyzed by the lab analysts. The number of samples will be the determined by the committee.
5. It will also be the choice of the committee either he may check the analysis of all the parameters or anyone.
6. Report will be furnished to the Director, SFRI for appropriate action, if required.



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CHAPTER-1

SOIL AND WATER SAMPLING

The error in soil sampling in a field is generally greater than the error in laboratory analysis. The most recommendation call for soil testing of each field is about every 3 years with more frequent testing on lighter soils. Therefore, it is necessary that the soil sample should be representative of the area.

■ SOIL SAMPLING

Non-representative samples constitute the largest single source of error in a soil fertility programme. The most important phase of soil analysis takes place not in the laboratory but in the field where the soil is sampled. Soils vary from place to place. In view of this, efforts should be made to take the samples in such a way that they are fully representative of the field. Only 1–10 g of soil is used for each chemical determination and this sample needs to represent as accurately as possible the entire surface 0–22 cm of soil, weighing about 2 million kg/ha.

a. CORRET PROCEDURE

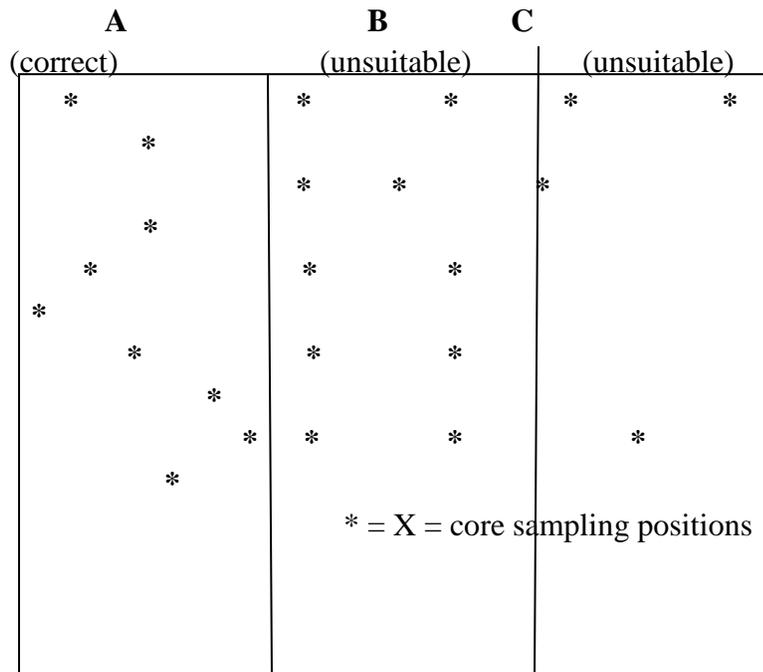
A zigzag pattern across the plot gives a proper composite of 10-30 cores for a single soil sample.

b. UNSUITABLE PROCEDURE

Regular positioning of the core in plot from which samples are likely to be biased by row applications. Analysis of these cores separately is an inefficient practice.

c. UNSUITABLE PROCEDURE

Regular positioning of core with a distinct bias towards the end of the plot, too few cores are presented to make an accurate composite soil sample.





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B. SAMPLING IN FIELDS.

DIVISION OF FIELD INTO AREA FOR SAMPLING

If soils are fairly uniform, an area upto 10 acres can be sampled as a unit. In case of variation, field can be subdivided into uniform sampling units and each sampled separately in the form of composite sample.

SOIL SAMPLING FOR VEGETABLE FIELDS

As vegetables are grown in fertile soil and root remain in furrow slice. So, the depth of 0-15, 15-30 and 30-45 cm is sufficient for soil fertility evaluation.

SOIL SAMPLING FOR FIELD CROPS

As roots of crops like wheat, rice, cotton, and maize etc. go deeper in sub-soil so it is necessary to get knowledge about sub soil nutrient status and concretions. Sampling of soil for this purpose depends on the type of crop grown and study required. Soil sampling should be done upto 0-15 , 15-30 , 30-45, 45-60 and 60-90 cm depth.

SOIL SAMPLING FOR ORCHARDS/GARDENS

In case of fruit trees samples should be taken at the distance not less than three feet from the trunk of plant or under the crown of tree. As root system is more extensive than crops sampling should be done upto the depth of 150 cm to evaluate nutrient status and underlying properties and water logging. Sampling should be done as follow 0-15, 15-30, 30-45,45-60,60-90,90-120 and 120-150 cm depth.

Carefully note the concretions especially. If more that 20%, it will hinder nutrient availability and free penetration of roots through the soil. In case of sandy soil, it will not have positive effect on fruit trees from nutrient supplying point of view.

PRECAUTIONS FOR SOIL SAMPLE COLLECTION

When sampling a soil, bear in mind the following:

1. Do not sample unusual areas, such as unevenly fertilized areas, marshy areas, old paths, old channels, old bunds, areas near trees, sites of previous compost piles, and other unrepresentative sites.
2. For a soft and moist soil, the tube auger or spade is considered satisfactory .For harder soil, a screw auger may be more convenient.
3. Where crops have been planted in rows, collect samples from the middle of the rows in order to avoid the area where fertilizer has been band placed.
4. Avoid any type of contamination at all stages. Soil samples should never be stored with fertilizer materials and detergents. Contamination is likely when the soil samples are spread out to dry in the vicinity of stored fertilizers or on floor where fertilizers were stored previously.
5. Before putting soil samples in bags, they should be examined for cleanliness as well as for strength.
6. The information sheet should be filled in clearly with a copying pencil.
7. Samples should not be taken:
 - i) Along the water channel, roadside, pathway, and boundaries



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- ii) Under the tree shade.
- iii) Close to the heaps (manure).
- iv) Areas where plant population is not uniform.
- v) Areas where domestic refuse is thrown.
- vi) Places in the field where fertilizers are dumped.
- vii) Places where organic matter or plant residues have been accumulated by natural agencies like wind or water, sampling should be avoided especially for fertility evaluation.
- viii) For salinity evaluation, if salt crust on the surface is present it should be removed before soil sample collection or it should be sampled separately.

PREPARATION OF COLLECTED SOIL SAMPLES

1. BAGGING: Samples are placed in polyethylene bags and then in cloth bags.

2. LABELLING: Labelling should be made with lead pencil or waterproof ink. One small label should be placed inside the bag and same number should be written on the bag.

3. DRYING: Soil samples after collection should be brought to the laboratory. Dry under shade and where fumes are not present. Higher temperature should be avoided as reversible change may take place. Samples received in the laboratory may be moist. They should be dried in wooden or enamelled trays. Care should be taken to maintain the identity of each sample at all stages of preparation. During drying, the trays can be numbered or a plastic tag could be attached. The samples are allowed to dry in the air. Alternatively, the trays may be placed in racks in a hot-air cabinet, whose temperature should not exceed 35 °C and whose relative humidity should be 30–60 percent. Oven drying a soil can cause profound changes in the sample. This step is not recommended as a preparatory procedure despite its convenience. Drying has a negligible effect on total N content, but the nitrate content in the soil changes with time and temperature. Drying at a high temperature affects the microbial population. With excessive drying, soil K may be released or fixed depending on the original level of exchangeable K. Exchangeable K will increase if its original level was less than 1 me/100 g soil (1 cmol/kg) and vice versa, but the effect depends on the nature of clay minerals in the soil. In general, excessive drying, such as oven drying of the soil, affects the availability of most of the nutrients present in the sample and should be avoided. Only air drying is recommended.

Nitrate, nitrite and ammonium determinations must be carried out on samples brought straight from the field. These samples should not be dried. However, the results are expressed on an oven-dry basis by estimating separately the moisture content in the samples.

4. GRINDING: After drying, the air-dried samples are ground with a wooden pestle and mortar so that the soil aggregate is crushed but the soil particles do not break down. Samples of heavy clay soils may have to be ground with an end-runner grinding mill fitted with a pestle of hard wood and rubber lining to the mortar. Pebbles, concretions and stones should not be broken during grinding.



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5. SIEVING: After grinding, Air dried soil should be screened through a sieve of 2mm mesh opening for general purpose and 100 mesh (0.45mm) for trace elements and for other critical analysis. The practice of passing only a portion of the ground sample through the sieve and discarding the remainder is erroneous. This introduces a positive bias in the sample as the rejected part may include soil elements with differential fertility. Therefore, the entire sample should be passed through the sieve except for concretions and pebbles of more than 2 mm. The coarse portion on the sieve should be returned to the mortar for further grinding. Repeat sieving and grinding until all aggregate particles are fine enough to pass the sieve and only pebbles, organic residues and concretions remain. After the sample has passed through the sieve, it must be mixed again thoroughly.

6. STORAGE: Soil samples, after passing through 2mm sieve, should be transferred into wide mouthed screw topped, numbered plastic bottles/jars. If the soil is to be analysed for trace elements, containers made of copper, zinc and brass must be avoided during grinding and handling. Sieves of different sizes can be obtained in stainless steel. Aluminium or plastic sieves are useful alternative for general purposes.

6. Before analysis samples must be uniformly mixed.

REFERENCE / RELATED DOCUMENTS:

Methods of Soil, Plant and Water Analysis Laboratory Manual ICARDA Third Edition, 2013

Guide to laboratory establishment for plant nutrient analysis, Food and Agriculture Organization of the United Nations Viale delle Terme di Caracalla 00153 Rome, Italy, 2008.



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WATER SAMPLING

■PURPOSE

To analyze the quality of irrigation

■EQUIPMENTS/APPARATUS REQUIRED:

Plastic bottles

■ METHOD:

A representative water sample is collected in a glass or polyethylenebottle, which should be properly washed/rinsed with the same water that is being sampled. The minimum quantity of water needed for the chemical analysis is about half liter. Obtain a representative sample. Collect sample from pipe after the pump has been running for some time. In general, shorter the elapsed time between collection and analysis of a sample of a sampler, the more reliable will be the analytical data. Changers resulting from chemical and biological activity may alter the composition of the sampler

■PRECAUTIONS/ SAFETY REQUIREMENTS:

- i) Bottle should be clean, never wash bottle with soap. Give detail of tube-well and along with location and depth. Floating debris or any other contaminant should be avoided while collecting the sample.
- ii) After proper labeling (e.g. source of water, date of collection, and type of analysis required), the sample should be sent to the laboratory without undue delay.
- iii) Some of the anions such as SO₄ and NO₃ may be quite low in irrigation waters. Hence, the large volume of the sample has to be first concentrated by evaporating it to about 100 ml in order to obtain their detectable amounts.

■ REFERENCE / RELATED DOCUMENTS:

Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington.

Guide to laboratory establishment for plant nutrient analysis, Food and Agriculture Organization of the United Nations Viale delle Terme di Caracalla 00153 Rome, Italy, 2008.



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CHAPTER-2

SOIL ANALYSIS

Soil testing is a chemical method for estimation of nutrient supplying power of a soil/ soil fertility evaluation. Soil fertility may be defined as the capacity of soil to furnish available plant nutrients to the plants in proper amount and appropriate balance, under ideal condition of plant growth. Whereas Soil productivity is the capacity of soil to produce under specific condition of crop production. Advantages of soil testing:

- More rapid method as compare to biological or deficiency symptoms/ plant analysis.
- One may determine the need of the soil before the planting of crop.
- To study/maintain fertility status of a field.
- To predict the probability of obtaining a profitable response of lime & fertilizers.
- To provide basis for recommendation of fertilizers.
- To evaluate fertility status of soil of an area/state/country for development of plans for research and education work.
- To study the alkalinity and salinity problems.
- To determine the suitability of the soil for laying gardens.
- CaCO₃ problems.
- Soil survey.



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DETERMINATION OF ELECTRICAL CONDUCTIVITY (EC_e) OF SOIL

■ **PURPOSE:**

Determination of Electrical Conductivity (EC_e) of Soil

■ **PRINCIPLE:**

Electrical Conductivity (EC) is the measure of the ability of a solution to conduct an electric current which is proportionate to the concentration of soluble salts in the sample at any particular temperature. The EC is measured in the field or in the laboratory using a conductivity meter, which is basically a Wheatstone bridge that measures the resistance of the solution between two parallel platinum electrodes.

It is normally measured by extracting the soil sample with water (1:5 or 1:10 soil: water ratio, w/v) or in an extract saturated paste.

■ **EQUIPMENTS/APPARATUS REQUIRED:**

Conductivity Meter, beaker, suction pump, or filter press

■ **REAGENTS/CHEMICAL REQUIRED:**

0.01 N KCl: Dissolve 0.7456 g KCl in 1 liter of distilled water.

■ **METHOD:**

SATURATION PASTE:

Weigh approx. 350g of soil (air dried pulverized to pass 10 mesh sieves < 2mm) in to 500ml plastic container. Gradually add deionized water and mix uniformly (free of partially wetted clumps) until a saturated paste is obtained. At saturation, the soil paste.

- Doesn't have free standing water on the surface of the paste
- Slides freely and cleanly of a spatula
- Will flow slightly when container tipped to 45 degree from horizontal
- Glistens as it reflects light
- Consolidate easily by tapping after a trench is formed in the paste with the flat side of spatula (may not apply to sandy soils > 70%)

SATURATION EXTRACT:

Saturation extract from the soil paste can easily be obtained with the help of suction pump using negative pressure (pressure is applied from below the soil paste) of 1 atmosphere. However, if the soil is highly sodic and contains high amount of sodium and clay, saturation extract cannot easily be obtained by the negative suction. For such soils filter press apparatus (pressure from above the paste) with positive pressure of any desirable magnitude can be used.

PROCEDURE:

Adjust moist filter paper inside the funnel, add paste into it, place bottle below the funnel, apply vacuum to collect the extract.

In case of filter, place a moist filter paper in stainless steel cup, add paste to it, closed and airtight the cup, apply pressure from above the paste gradually till all the extract is collected in the bottle placed under the cup.



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DETERMINATION OF ELECTRICAL CONDUCTIVITY OF SATURATION

EXTRACT(EC_e)

■PROCEDURE:

Wash the conductance cell of EC meter with distilled water and then with extract (if extract is in reasonable amount). Dip the cell into the extract making sure that there is no air bubble in it. Conductivity corrected at 25 °C will be displayed in $mS\ cm^{-1}$ or $dS\ m^{-1}$.

If the sample is very small either use the micro cell or dilute the sample. Sometime salt concentration is too high to be measured. So, make appropriate dilutions and multiply the final reading with the dilution factor.

■ EC DETERMINATION OF SOIL WATER RATIO:

In case of saline sodic or sodic soils, to get a sufficient amount of extract is difficult. In such cases soil: water ratio of 1:2, 1:5 or 1:10 should be used.

■CALCULATIONS:

$$\text{Dilution Factor} = \frac{V_f \text{ (total volume of diluted solution)}}{V_i \text{ (volume of aliquot taken for dilution)}}$$

■ CONVERSION OF 1:10 TO EC_e

Divide reading of 1:10 EC by 1.33 to convert into EC_e

$$\text{So } EC_1 (1:10) / 1.33 = EC_2 \text{ i.e } EC_e$$

■PRECAUTIONS/ SAFETY REQUIREMENTS:

Standardize the conductivity meter before analysis. Calibrate the instrument with 0.01 N KCl solution. The EC of this standard solution is $1.413\ dS\ m^{-1}$ at 25 °C. The cell constant (K) can be calculated by the formula.

$$K = \frac{1.413\ dS\ m^{-1}}{\text{Observed reading in } dS\ m^{-1}}$$

The cell constant is necessary when 0.01 N KCl solution gives an EC values different from $1.413\ dS\ m^{-1}$. In this case, samples readings are multiplied by the cell constant.

■REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 89-91.



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DETERMINATION OF SOIL REACTION (pH) USING SATURATED PASTE

■ PURPOSE:

Determination of soil reaction (pH)

PRINCIPLE:

pH is a measure of how acidic or alkaline a solution is; or the power or potential of the hydrogen $[H_3O^+]$ ion. pH stands for the negative logarithm of H_3O^+ -or in short H^+ ion activity, in mol/L. $pH = -\log [H^+]$. Soil pH is a crucial soil indicator and is defined as the negative log of the hydrogen ion activity. Since pH is logarithmic, the H-ion concentration in solution increases ten times when its pH is lowered by one unit.

■ EQUIPMENTS/APPARATUS REQUIRED:

- Plastic beaker
- Spatula
- pH meter

■ REAGENTS/CHEMICALS REQUIRED:

Standard buffer solutions of pH 7.01 and 10.01

Deionized water

■ CALIBRATION OF pH METER

- Calibrate the pH meter using at least two buffer solutions of different pH values, usually (7.01 and 10.01) or company specified buffers.
- Calibrate accordingly to the company specified protocols. For alkaline Soil, its better to calibrate pH meter in between 7-10pH buffers

■ PROCEDURE:

Make a soil saturated paste as explained in E_{Ce} method. Give overnight stay to the soil paste so that it may stabilized. Rejuvenate the paste next morning if desired. Dip the electrode in the paste, note the reading when it is stabilized. Wash the electrode with distilled water and dry with tissue paper before dipping in new soil paste sample.

■ PRECAUTIONS / SAFETY REQUIREMENTS:

After completion of work, wash and immerse the electrode in distilled water or in saturated KCl solution.

■ REFERENCE / RELATED DOCUMENTS:

Schofield, R. K. and A. W. Taylor. 1955. The measurement of soil pH. Soil Sci. Soc. Amer. Proc. 19:164-167.



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DETERMINATION OF SOIL REACTION (pH) (1:1)

■ PURPOSE:

Determination of soil reaction (pH) using 1:1 soil –water ratio

■ EQUIPMENTS/APPARATUS REQUIRED:

- Plastic beaker
- Spatula
- pH meter

■ REAGENTS/CHEMICALS REQUIRED:

- Standard buffer solutions of pH 7.01 and 10.01
- Deionized water

■ CALIBRATION OF pH METER

- Calibrate the pH meter using at least two buffer solutions of different pH values, usually (7.01 and 10.01) or company specified buffers.
- Calibrate pH meter accordingly to company specified protocols. For alkaline soils, it's better to calibrate pH meter in-between 7-10 pH buffers

■ PROCEDURE:

- Weigh 50 g air-dry soil (< 2-mm) into a 100-mL glass beaker.
- Add 50 mL DI water using a graduated cylinder or 50-mL volumetric flask.
- Mix well with a glass rod and allow to stand for overnight.
- Next day, put the combined electrode in suspension (about 3-cm deep). Take the reading after 30 seconds with one decimal.
- Remove the pH electrode from the suspension, and rinse thoroughly with DI water in a separate beaker, and carefully dry excess water with a tissue.

■ PRECAUTIONS / SAFETY REQUIREMENTS:

After completion of work, wash and immerse the electrode in distilled water or in saturated KCl solution.

■ REFERENCE / RELATED DOCUMENTS:

George Estefan, Rolf Sommer, and John Ryan 2013. Methods of Soil, Plant, and Water Analysis: A manual for the West Asia and North Africa region. International Center for Agricultural Research in the Dry Areas (ICARDA)



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DETERMINATION OF SOIL SATURATION PERCENTAGE

■ **PURPOSE:**

Determination of Soil Saturation Percentage

■ **PRINCIPLE:** This method soil water content of saturated soil. At saturation all soil pore space is occupied by water. Texture can be made from saturated moisture contents

■ **EQUIPMENTS/APPARATUS REQUIRED:**

Balance, Beakers, China Dish, Spatula, Plastic beaker

■ **METHOD:**

PREPARATION OF SATURATED SOIL PASTE

Weigh 100g of soil (air dried pulverized to pass 10 mesh sieves < 2mm) in to 500ml plastic container. Gradually add deionized water and mix uniformly (free of partially wetted clumps) until a saturated paste is obtained. At saturation, the soil paste.

- Doesn't have free standing water on the surface of the paste
- Slides freely and cleanly of a spatula
- Will flow slightly when container tipped to 45 degree from horizontal
- Glistens as it reflects light
- Consolidate easily by tapping after a trench is formed in the paste with the flat side of spatula (may not apply to sandy soils > 70%)

Record mass of soil and total water added

■ **CALCULATIONS:**

$$\text{Saturation \% (SP \%)} = \frac{\text{Water Added (ml)}}{\text{Weight (g) of soil (air dried)}} \times 100$$

Table 1: Saturation % vs Soil Texture

SP (%)	Soil Texture
0-20	Sand or Loamy Sand
20-35	Sandy Loam
35-50	Loam or Silt loam
50-65	Clay loam
65-135	Clay
>81	Organic Soils

■ **PRECAUTIONS / SAFETY REQUIREMENTS:**

Soil paste must meet all criteria of soil paste parameters

■ **REFERENCE / RELATED DOCUMENTS:**

Robert O Miller, Colorado State University, USA



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DETERMINATION OF CARBONATES AND BICARBONATES IN SOIL

■ **PURPOSE:**

Determination of Carbonates and Bicarbonates in soil

■ **PRINCIPLE:**

Carbonate (CO_3) and bicarbonate (HCO_3) ions are species of the same acid, carbonic acid. Their proportionate content is a function of pH. The CO_3 starts to form as pH rises above 8.4. Carbonate and bicarbonate are generally determined in soil saturation extract by titration with 0.01 N H_2SO_4 to pH 8.3 and 4.5, respectively.

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Pipette, 50 ml
- Conical flask, 100ml
- Cylinder, 50 ml
- Wash bottle
- Burette, 50 ml

■ **REAGENTS/CHEMICALS REQUIRED:**

- Phenolphthalein 1%: Dissolve 1.0 g phenolphthalein in 100 ml ethanol with constant stirring. Filter if needed.
- Methyl orange 0.1%: Dissolve 0.1 g methyl orange in 100 ml distilled water.
- Sulphuric Acid 0.01N (standardized).
-

■ **METHOD:**

A. Extraction

1. Soluble carbonate and bicarbonate can be obtained in a water extract from a saturated paste as for pH and EC determinations.
2. Filter suspension using Whatman No.1 filter paper to exclude any soil particles.

B. Measurement

1. Pipette 10 – 15 mL soil saturation extract in a wide-mouthed porcelain crucible or a 150-mL Erlenmeyer flask.
2. Add 1 drop phenolphthalein indicator. If pink color develops, add 0.01 N H_2SO_4 by a burette, drop by drop, until the color disappears.
3. Take the reading, R1.
4. Continue the titration with 0.01 N H_2SO_4 after adding 2 drops 0.1 % methyl orange indicator until the color turns to orange.
5. Take the reading, R2.
6. Always run two blanks containing all reagents but no soil and treat them in exactly the same way as the samples. Subtract the blank titration reading from the readings for all samples.



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■ **CALCULATIONS :**

$$\text{CO}_3^{-2} \text{ (me L}^{-1}\text{)} = \frac{2R_1 \times \text{Normality of H}_2\text{SO}_4}{\text{aliquot(ml)}} \times 1000$$

or

Volume of aliquot taken = × ml (10 ml)

Volume of 0.01 N H₂SO₄ used = Final reading - blank reading = R₁

Actual volume of 0.01 N H₂ SO₄ used = 2× R₁ = 2R₁*

$$\text{CO}_3^{-2} \text{ (me L}^{-1}\text{)} = \frac{2R_1 \times 0.01 \text{ H}_2\text{SO}_4}{10} \times 1000$$

$$\text{Bicarbonates (me L}^{-1}\text{)} = \frac{(R_2 - R_1) \times \text{Normality of H}_2\text{SO}_4}{\text{aliquot (ml)}} \times 1000$$

or

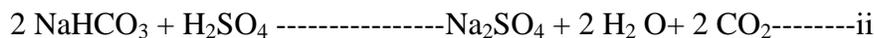
Volume of aliquot taken = 10 ml

Volume of 0.1 N H₂SO₄ used = Final reading -Initial reading

** = R₂

Actual volume of 0.1 N H₂SO₄ used = R₂ - R₁

* This reaction is completed in two steps as shown by the chemical reaction



R₁ is the amount of acid used to convert the carbonates to bicarbonates (equation i, phenolphthalein part of reaction, while for the completion of the reaction, same amount of acid will be still needed to further convert bicarbonates so produced during step i to form CO₂ and H₂O as shown in equation ii. Thus, R₁ is the amount of acid for half reaction and 2R₁ will represent the amount of complete reaction. Hence R₁ is multiplied by a factor of 2.

** R₂ is the total amount of acid used to neutralize the bicarbonates present in the sample after titration. Which means that sample contains bicarbonates coming from conversion of CO₃ as



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well as originally present in the sample. Therefore, the actual amount of acid used for neutralizing the originally present HCO_3 would be $R_2 - R_1$.

■ PRECAUTIONS / SAFETY REQUIREMENTS:

Always run blank for accuracy.

■ TECHNICAL REMARK

Carbonate and bicarbonate should be determined soon after preparing extracts because CaCO_3 precipitates on standing. If other analyses (Ca, Mg, K, Na, Cl, and SO_4) cannot be performed immediately, refrigerate the extracts.

■ REFERENCE / RELATED DOCUMENTS:

- United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA. Handbook 60. Washington, D.C. pp. 89-91.
- George Estefan, Rolf Sommer, and John Ryan 2013. Methods of Soil, Plant, and Water Analysis: A manual for the West Asia and North Africa region. International Center for Agricultural Research in the Dry Areas (ICARDA)



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DETERMINATION OF SOIL SOLUBLE CALCIUM AND MAGNESIUM

■ **PURPOSE:**

Determination of Soluble Calcium and Magnesium of Soil

■ **PRINCIPLE:**

The method is based on the principle that calcium, magnesium and a number of other cations form stable complexes with versenate (ethylenediaminetetraacetic acid disodium salt, EDTA) at different pH. A known volume of standard calcium solution is titrated with standard 0.01N EDTA solution using Murexid (ammonium purpurate) indicator in the presence of NaOH solution. The end point is a change of color from orange red to purple at pH 12 when the whole of Ca forms a complex with EDTA. Calcium plus Mg are also present in the solution and can be titrated with EDTA using buffer solution and a few drops Eriochrome Black T indicator. The end point is a change of color from red to blue at pH 10. Beyond pH 10, magnesium is not bound strongly to Eriochrome Black-T indicator to give a distinct end point

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- China dish
- Pipette, 10 ml
- Cylinder, 50 ml
- Glass rod
- Wash bottle

■ **REAGENTS/CHEMICALS:**

A. NH₄Cl -NH₄ OH buffer solution

Dissolve 67.5 g NH₄Cl in 570 ml concentrated NH₄OH, make the volume up to 1 liter.

B Sodium hydroxide (NaOH), 4 N

Dissolve 160 g NaOH in distilled water, make the volume of the solution to 1 liter .

C. Standard calcium chloride solution (CaCl₂) , 0.01 N

Dissolve 0.5 g CaCl₂ pure (calcite crystals) in 10 ml of approx. 3 N (1+3) HCl and dilute to a volume of exactly 1 liter.

D. Eriochrome black-T indicator (EBT)

Dissolve 0.4 g of EBT and 4.5 g hydroxyl amine hydrochloride (NH₂O.H. HCl, H₃NO.HCl, m. wt. 69.49) in 100 ml of 95 % ethanol.

E. Ammonium Purpurate indicator (C₈H₄N₅O₆. NH₄)

Thoroughly mix 0.5 g ammonium Purpurate with 100 g of powdered potassium sulphate (K₂SO₄)

F. EDTA (Versenate) solution, 0.01 N

Dissolve 2.0 g of disodium salt and 0.05 g magnesium chloride hexahydrate (MgCl₂. 6H₂ O) in distilled water and dilute to a volume of 1 liter . Standardized the solution against reagent C using given titration procedure.



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■ **METHOD: Extraction**

1. Soluble Ca and Mg can be obtained in a water extract from a saturated paste as for pH and EC determinations.
2. Filter suspension using a Whatman No.1 filter paper to exclude any soil particle

■ **CALCIUM**

- Pipette 10-20 mL soil extract, into a 250-mL Erlenmeyer flask.
- Dilute to 20-30 mL with DI water, add 2-3 mL 2 N NaOH solution.
- Add about 50 mg ammonium purpurate indicator.
- Titrate with 0.01 N EDTA until the color changes from red to lavender or purple. Near the end point, EDTA should be added one drop every 10 second since the color change is not instantaneous.
- Always run a blank containing all reagents but no soil and treat it in exactly the same way as the samples; and subtract the blank titration reading from the readings for all samples.

CALCIUM PLUS MAGNESIUM

- Pipette 10 – 20 mL soil extract into a 250-mL Erlenmeyer flask.
- Dilute to 20 – 30 mL with DI water, add 3-5 mL buffer solution.
- Add a few drops eriochrome black indicator.
- Titrate with 0.01 N EDTA until the color changes from red to blue

■ **CALCULATIONS :**

$$\text{Soluble Ca}^{++} = \frac{\text{ml of EDTA sol. for sample -ml of EDTA for blank} \times N \times 1000}{\text{aliquot in ml}}$$

$$\text{Sol. Ca}^{++} + \text{Mg}^{++} \text{ (me L}^{-1}\text{)} = \frac{\text{ml of EDTA sol. for sample -ml of EDTA for blank} \times N \times 1000}{\text{aliquot in ml}}$$

$$\text{Soluble Mg}^{++} \text{ (me L}^{-1}\text{)} = (\text{Ca}^{++} + \text{Mg}^{++}) - \text{Ca}^{++} \text{ (All expressed as meL}^{-1}\text{)}$$

* multiply values with dilution Factor if any dilution made

■ **REFERENCE / RELATED DOCUMENTS:**

George Estefan, Rolf Sommer, and John Ryan 2013. Methods of Soil, Plant, and Water Analysis: A manual for the West Asia and North Africa region. International Center for Agricultural Research in the Dry Areas (ICARDA)



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DETERMINATION OF CHLORIDE IN SOIL

■ **PURPOSE:**

Determination of soil soluble chloride

■ **PRINCIPLE:**

Soluble chloride (Cl) is obtained in the saturation extract (as prepared for soluble Ca, Mg and anions), and its concentration in the extract is determined by silver nitrate titration. The determination of Cl is done by AgNO₃ (Mohr's titration) method, which is based upon the fact that in solution containing Cl and chromate (Cr). Silver reacts with all the Cl and precipitates before the reaction with chromate begins. The appearance of the brick-red colour of the silver chromate precipitate is the end-point of the titration

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Pipette, 50 ml
- Conical flask, 100ml
- Cylinder, 50 ml
- Wash bottle
- Burette, 50 ml

■ **REAGENTS/CHEMICALS REQUIRED:**

- Potassium Chromate (K₂CrO₄), 5 % solution
- Dissolve 5.0 g of potassium chromate in 50 ml distilled water and add 1 N (saturated solution) AgNO₃ drop wise until red precipitate is produced. The solution is filtered, and filtrate is diluted to 100 ml.
- Silver Nitrate (AgNO₃), 0.01 N (F. wt. 169.888)
- Dissolve 1.696 g (oven dried) silver nitrate in distilled water and dilute to 1 litre. Keep in a brown bottle away from light to avoid photolysis.

■ **METHOD**

A. Extraction

1. Soluble Cl can be obtained in a water extract from a saturated paste as for pH and EC determinations.
2. Filter suspension using Whatman No.1 filter paper to exclude any soil particles.

B. Measurement

1. Pipette 5-10 mL soil saturation extract in a wide-mouth Erlenmeyer flask (150-mL).
2. Add 4 drops 5 % K₂CrO₄ solution.
3. Titrate against 0.01 N AgNO₃ until a permanent reddish-brown color appears.
4. Always run two blanks containing all reagents but no soil and treat them in exactly the same way as for the samples. Subtract the blank titration reading from the readings for all samples.
6. In order to standardize the AgNO₃ solution used in the determination of Cl:
 - Pipette 10 mL 0.01N NaCl solution in a wide-mouth Erlenmeyer flask.



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- Add 4 drops 5 % K_2CrO_4 solution.
- Titrate against 0.01 N $AgNO_3$ solution, until a permanent reddish-brown color appears.
- Take the reading, and calculate $AgNO_3$ normality:

■ **CALCULATIONS:**

$$Cl \text{ (me L}^{-1}\text{)} = \frac{(\text{AgNO}_3 \text{ used for sample} - \text{AgNO}_3 \text{ used for blank}) \times \text{Normality of AgNO}_3 \times 1000}{\text{aliquot (ml)}}$$

■ **PRECAUTIONS/ SAFETY REQUIREMENTS.**

Always run a blank with actual titration.

■ **REFERENCE / RELATED DOCUMENTS:**

George Estefan, Rolf Sommer, and John Ryan 2013. Methods of Soil, Plant, and Water Analysis: A manual for the West Asia and North Africa region. International Center for Agricultural Research in the Dry Areas (ICARDA)



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DETERMINATION OF SOIL ORGANIC MATTER

■ PURPOSE:

Organic matter determination in soil

■ PRINCIPLE:

Organic matter determination in soil involves reduction of potassium dichromate ($K_2Cr_2O_7$) by Organic Carbon compounds and subsequent determination of the unreduced dichromate by oxidation-reduction titration with ferrous ammonium sulfate. This method is referred to as the *Walkley-Black method*

■ EQUIPMENTS/APPARATUS REQUIRED:

- 1 N Potassium dichromate ($K_2Cr_2O_7$, f. wt. 294. 18): Dissolve oven dried 49.04 g potassium dichromate in distilled water, mix well and dilute to 1litre.
- 1.0 N ferrous sulphate standardized: Dissolve 278.02 g ferrous sulphate ($FeSO_4.7H_2O$), in distilled water, add 15 ml concentrated Sulphuric acid and dilute to one liter.
- Sulphuric Acid (96 %, specific gravity 1.84)
- Phosphoric acid (H_3PO_4) 85 % or sodium fluoride (NaF)
- Barium diphenylamine sulphonate, 0.16 %: Dissolve 0.16 g barium diphenylamine sulphonate indicator in 100 ml conc. H_2SO_4 of 4:1 ratio.

■ REAGENTS/CHEMICALS REQUIRED:

- 500 ml conical flask
- Pipette, 10 ml
- Burette, 50 ml
- Beaker, 500 ml
- Cylinder, 50 ml
- Wash bottle
- Weighing balance

■ METHOD:

Weigh 1.0 g air dried, ground soil sample into 250- or 500-ml conical flask, add 5 ml $K_2Cr_2O_7$ and mix well. Add 10 ml Sulphuric acid and mix. Allow the flask to stand for 30 minutes to cool. Then add 100-150 ml distilled water. Add 3 ml phosphoric acid or 0.5 g sodium fluoride, 5-10 drops of indicator and finally titrate against standardized ferrous sulphate to sharp green end point through blue colour. Run a blank and subtract sample reading from blank to get the actual volume of ferrous sulphate used to reduce potassium dichromate.

■ CALCULATIONS REQUIRED:

$$O M (\%) = \frac{\text{ml for blank} - \text{ml for sample}}{\text{weight of sample (g)}} \times 0.698^*$$



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$$\text{O M (\%)} = \frac{\text{me of K}_2\text{Cr}_2\text{O}_7 \text{ reduced}}{\text{weight of sample (g)}} \times 0.698$$

me of $\text{K}_2\text{Cr}_2\text{O}_7$ reduced = (me of $\text{K}_2\text{Cr}_2\text{O}_7$ added - me of FeSO_4 used)

me of $\text{K}_2\text{Cr}_2\text{O}_7$ added = (ml of $\text{K}_2\text{Cr}_2\text{O}_7$ added x N)

me of FeSO_4 used = (ml of FeSO_4 used x N)

$$\text{O C (\%)} = \frac{\text{ml for blank - ml for sample}}{\text{weight of sample (g)}} \times \text{N FeSO}_4 \times 0.337$$

O C (%) = % organic matter / 1.724

$$*0.698 = 0.003 \times \frac{100}{74} \times \frac{100}{58} \times 100$$

How = 0.003

1 me of C of OM in soil = 1 x Eq. wt. of C / 1000

$$\text{Eq. Wt. of C} = \frac{\text{M.wt.}}{\text{valency}} \times \frac{12}{4} = \frac{3}{1000} = 0.003 \text{ g of C of OM in soil}$$

So

1 ml of 1 N $\text{K}_2\text{Cr}_2\text{O}_7$ when reduced = 0.003 g of C of OM in soil

$100 / 74 = 1.3514$, efficiency factor for determining O. C. of soil

$100 / 58 = 1.724$, for conversion of % OM of soil to % O.C. (If divided).

100 = for converting O.M. in percentage

**0.337

Walkley, s rapid method (1935, 1947) for the determination of organic carbon in soils has been found to give app. 89 % recovery of carbon as compared to dry combustion method. The conversion factor, 0.337 was obtained by dividing 0.003, the milliequivalent weight of carbon by 89 and multiplying by 100 to convert to percent.

■ PRECAUTIONS/ SAFETY REQUIREMENTS:

Always run blank along with samples.

■ REFERENCE / RELATED DOCUMENTS:

Walkley, A. 1947. A critical examination of a rapid method for determining organic carbon in soils – effect of variations in digestion conditions and of inorganic soil constituents. Soil Sci. 63:251-264.



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DETERMINATION OF SOIL TEXTURAL CLASS

■ **PURPOSE:**

Soil Textural Class Determination

■ **PRINCIPLE:**

The hydrometer method of silt and clay measurement relies in the effect of particle size on the differential settling velocities within a water column. By this method (using Hydrometer with Bouyoucos scale in g/L) after 40 second all sand-sized particles (0.02 mm and larger) settle out of the suspension and after 4 h, particles larger than clay (0.002 mm) settle out of the suspension

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Hydrometer
- Graduated cylinder 1000 ml
- Thermometer
- Stopwatch
- Stirrer with cup
- Beaker 500 ml
- Perforated brass plunger
- Wash bottle

■ **REAGENTS/CHEMICALS REQUIRED:**

- Sodium hexametaphosphate (4%), dispersing reagent (NaPO_3)₆: Dissolve 40 g of sodium hexametaphosphate and 10 g of sodium carbonate in distilled water, mix well and make the volume one liter (new solutions should be prepared after 1 – 2 weeks).
- Amyl Alcohol

■ **METHOD:**

1. Weight 40g air-dry soil (2-mm) into a 600mL beaker.
2. Add 60-mL dispersing solution
3. Cover the beaker with a watch-glass, and leave overnight
4. Quantitatively transfer contents of the beaker to a soil-stirring cup and fill the cup to about three-quarters with water.
5. Stir suspension at high speed for 3 minutes using the special stirrer. Shake the suspension overnight if no stirrer is available.
6. Rinse stirring paddle into a cup and allow to stand for 1 minute.
7. Transfer suspension quantitatively into a 1-L calibrated cylinder (hydrometer jar) and bring to volume with water.

A. Determination of Blank

- Dilute 60 mL dispersing solution to 1-L hydrometer jar with water
- Mix well, and insert hydrometer, and take hydrometer reading **Rb**.
- The blank reading must be re-determined for temperature changes of more than 2°C from 20°C



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B. Determination of Silt plus Clay.

- Mix suspension in the hydrometer jar, using a special paddle carefully, withdraw the paddle, and immediately insert the hydrometer.
- Disperse any froth, if needed, with one drop of amyl alcohol, and take hydrometer reading 40 seconds after withdrawing the paddle. This gives reading **R_{sc}**.

■ CALCULATIONS

Percentage **Silt plus Clay** in soil

$$\% \text{ [Silt +Clay] (w/w)} = (R_{sc}-R_b) \times \frac{100}{\text{Oven -dry soil (g)}}$$

C. Determination of Clay

- Mix suspension in the hydrometer jar with paddle, withdraw the paddle, with great care, leaving suspension undisturbed
- After 4 hours, insert the hydrometer, and take hydrometer reading **R_c**.

Percentage **Clay** in soil:

$$\% \text{ [Clay] (w/w)} = (R_c-R_b) \times \frac{100}{\text{Oven -dry soil (g)}}$$

Percentage **Silt** in soil:

$$\% \text{ Silt (w/w)} = \% \text{ [Silt +Clay (w/w)]} - [\% \text{ Clay (w/w)}]$$

D. Determination of Sand

- After taking readings required for clay and silt, pour suspension quantitatively through a 50µm sieve.
- Wash sieve until water passing the sieve is clear.
- Transfer the sand quantitatively from sieve a 50 mL beaker of known weight.
- Allow the sand in the beaker to settle and decant excess water.
- Dry beaker with sand overnight at 105°C
- Cool in a desiccator, and re-weigh beaker with sand.

Percentage **Sand** in soil:

$$\% \text{ Sand (w/w)} = \frac{\text{Sand weight}}{\text{Oven -dry soil (g)}} \times 100$$

Where: Weight of sand follows from

$$\text{Sand weight (g)} = [\text{Beaker + Sand (g)}] - [\text{Beaker (g)}]$$

Note:

- If possible, all hydrometer jars should be placed in a water bath at constant temperature (20°C); in that case, temperature corrections are not needed.
- For temperature correction, use a value of 0.4 for each degree temperature difference from 20°C. Add or subtract this factor if the temperature is more or less than 20°C, respectively.

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- All results of mechanical analysis should be expressed on the basis of oven-dry soil (24 hours drying at 105°C).
- In the above procedure, carbonates and organic matter are not removed from the soil.
- The Hydrometer method, as described in this section, cannot be applied to soils that contain free gypsum (gypsiferous soils). For gypsiferous soils, see Hesse (1971).
- Sum of % silt and clay + % sand should be 100%. The magnitude of deviation from 100 is an indication for the degree in accuracy.

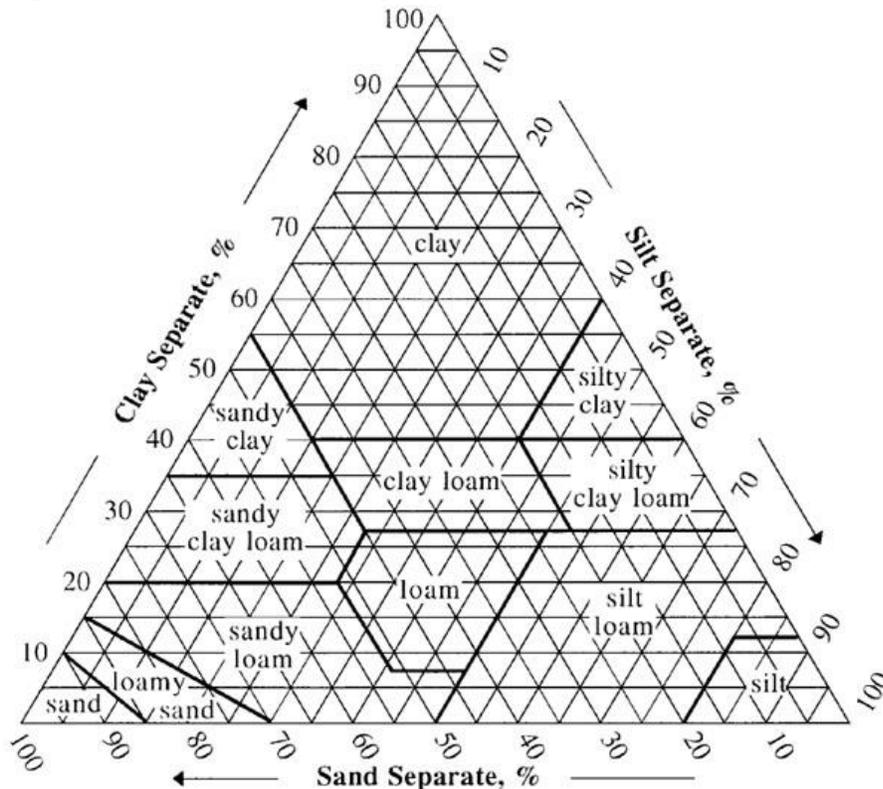
Soil Texture

Once the percentage of sand, silt, and clay is measured, the soil may be assigned a textural class using the USDA textural triangle (Fig.1). Within the textural triangle are various soil textures which depend on the relative proportions of the soil fractions.

■ REFERENCE / RELATED DOCUMENTS:

Bouyouces, G. J. 1962. Hydrometer method improved for making particle size analysis of soils. *Agron. J.*, 53:464-465.

Fig no .1: Soil Textural Triangle





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DETERMINATION OF EXTRACTABLE POTASSIUM IN SOIL

■ **PURPOSE:**

Extractable potassium determination in soil

■ **PRINCIPLE:**

Extractable K is the sum of water-soluble and exchangeable K. The method uses a neutral ammonium acetate solution (1 N) to replace the cations present on the soil exchange complex. This is considered as plant available K in the soils, and is commonly measured by the flame photometer.

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Mechanical Shaker, reciprocating
- Flame photometer
- Beaker, 50 ml
- Pipette, 10 ml
- Wash bottle
- Volumetric flasks

■ **REAGENTS/CHEMICALS REQUIRED:**

- A.1 N NH₄OAC: Dissolve 77.1 g ammonium acetate (F.W. 77.1, CH₃COONH₄) in 800 ml distilled water and make the volume to 1 liter. Adjust pH 7.0 using either NH₄OH or HCl.
- B. Stock solution : Dissolve 1.907 g oven dried KCl in distilled water and make volume 1-2. This solution contains 1000ppm K.
- Prepare standard solutions from stock solution in 100ml volumetric flask.

■ **METHOD:** Wight 2.5 g air dried, ground, passed through 2 mm sieve soil sample into a 250ml conical flask, add 50 ml extracting reagent, shake on a flatbedreciprocal shaker for 30 minutes and filter the extract. Determine K by flame photometer in ppm using graph readings.

■ **CALCULATIONS REQUIRED:**

Extractable K (ppm) = reading (ppm) × 20

■ **REFERENCE / RELATED DOCUMENTS:**

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 96-97.



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DETERMINATION OF EXTRACTABLE SOIL PHOSPHORUS (OLSEN'S METHOD)

■ **PURPOSE:**

Soil Extractable Phosphorus Determination (The Olsen Method)

■ **PRINCIPLE**

The sodium bicarbonate (NaHCO_3) procedure of Olsen et al. (1954) is generally accepted as a suitable index of P “availability” for alkaline soils, where the solubility of calcium phosphate is increased because of the precipitation of Ca as CaCO_3 .

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Spectrophotometer
- Standard laboratory glassware, Beaker, Volumetric flasks pipettes funnels
- Extraction bottles

■ **REAGENTS/CHEMICALS REQUIRED:**

- Sodium bicarbonate solution, 0.5 M NaHCO_3 : Dissolve 42.0 g NaHCO_3 in app. 700 ml distilled water, shake well and make the volume 1000 ml . Adjust pH 8.5 using 5 N NaOH.
- 5 N NaOH = Dissolve 200g NaOH in distilled water and let it cool and make volume 1 L.
- Mixed reagent:
 - a) Ammonium hepta molybdate 4.8 % $(\text{NH}_4)_6\text{MO}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$ (f. wt. 1235.9) Dissolve 12.0 g in distilled water and make volume 250 ml
 - b) Potassium antimony tartrate ($\text{K}_2\text{Sb}_2\text{O}_7 \cdot 4\text{H}_2\text{O}$) Dissolve 0.291 g make volume to 100 ml with distilled water
 - c) 5N H_2SO_4 . Dilute 148 ml concentrated sulfuric acid (in fume hood) in distilled water, let it cool and make volume 1 L.
 - d) Add both the dissolved reagents (a + b) in 1000 ml 5 N H_2SO_4 and make volume 2000 ml with distilled water. Store in a Pyrex bottle in a dark, cool place
- Colour developing reagent: Weigh 0.528g ascorbic acid to 100 ml of mixed reagent. This reagent should be prepared freshly as required because it does not give accurate results after 24 hours.
- D. Stock solution (1000 ppm): Dissolve 4.3937 g potassium dihydrogen phosphate (KH_2PO_4) in distilled water and make volume to 1 liter.
- **STANDARDS** (0.05-3.0ppm):
Making 5 ppm sub-stock solution



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■ **METHOD:**

Weigh 2.5 g air dried and ground soil, add 50 ml extracting solution. Shake for 30 minutes and filter with Whatman No. 42. Pipette out 5 ml aliquot. Add 5 ml of colour developing reagent, in 25 ml volumetric flask. Shake to remove gas bubbles. Let stand for 15 minutes. Make volume up to mark. Bluish colour will develop. Concentration of phosphorus in soil is directly proportional to the intensity of blue color developed. Take reading on concentration mode at 880 nm wavelength on Spectrophotometer.

■ **CALCULATIONS:-** Extractable phosphorous in soil (ppm)=ppm x dilution factor

■ **REFERENCE / RELATED DOCUMENTS:**

Olsen, S. R., C. V. Cole, F. S. Watanabe, and L. A. Dean. 1954. Estimation of available phosphorus in soils by extraction with sodium bicarbonate. USDA Circ. 939. pp. 19.



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DETERMINATION OF SOLUBLE SODIUM IN SOIL

■ **PURPOSE:**

Soluble Sodium Determination of Soil

■ **PRINCIPLE:**

This fraction is a measure of the amount of Na extracted from the soil by water (can be obtained in a water extract from a saturated paste as for pH and EC).

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Flame Photometer
- Volumetric flask
- Conical flask

■ **REAGENTS/CHEMICALS REQUIRED:**

Preparation of stock solutions

Stock solution of sodium (1000 ppm) can be prepared by dissolving oven dried NaCl (2.5435 g) in distilled water and making the volume to exactly one litre. Store in a cool and dry place.

Preparation of working standards:

Stock solution so prepared will be used for the preparation of working standards by using the formula:

$$C_1V_1 = C_2V_2$$

Whereas

C_1 = Concentration of stock solution in ppm

V_1 = Volume to be taken of stock solution in ml

C_2 = Concentration of Na to be required in ppm

V_2 = Total volume to be required in ml

$$C_2 \text{ (ppm required)} = \frac{V_1(\text{ml of known solution}) \times C_1 \text{ (ppm of known solution)}}{V_2 \text{ (Total volume to be made)}}$$

Working standard solution can be prepared of 10,20,30 ppm concentration



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■ **METHOD:**

A. Extraction

1. Soluble Na can be obtained in a water extract from a saturated paste as for pH and EC determinations.
2. Filter suspension using a Whatman No.1 filter paper to exclude any soil particles.

B. Measurement

1. Operate Flame Photometer according to the instructions provided for the equipment.
2. Run a series of suitable Na standards and draw a calibration curve.
3. Measure Na in the samples (soil extracts) by taking the emission readings on the Flame.
4. Calculate Na concentrations by inferring to the calibration curve.

■ **CALCULATIONS REQUIRED:**

Soluble Na (ppm) = from calibration curve x dilution factor

■ **PRECAUTION**

Clean the table, wash all the apparatus and place at proper place before leaving the laboratory.

■ **REFERENCE / RELATED DOCUMENTS:**

George Estefan, Rolf Sommer, and John Ryan 2013. Methods of Soil, Plant, and Water Analysis: A manual for the West Asia and North Africa region. International Center for Agricultural Research in the Dry Areas (ICARDA).



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DETERMINATION OF SULPHATE IN SOIL

■ **PURPOSE:**

Sulfate in soil extractant is determined normally by barium sulfate precipitation

■ **PRINCIPLE:**

The commonly used method for S determination in alkaline soils is the extraction of SO₄-S with 0.15% CaCl₂.2H₂O and measurement of SO₄-S concentration in the extracts by a turbidimetric procedure using barium chloride

■ **APPARATUS**

- Mechanical shaker reciprocal
- Spectrophotometer or colorimeter

■ **REAGENTS**

- Calcium Chloride Dihydrate Solution (CaCl₂.2H₂O), 0.15% Dissolve 1.5 g CaCl₂.2H₂O in about 700 mL DI water, and bring to 1-L volume.
- Hydrochloric Acid Solution (HCl), 6 M Dilute 496.8 mL concentrated HCl (37%, sp. gr. 1.19) in DI water, mix well, let it cool, and bring to 1-L volume.
- Barium Chloride (BaCl₂.2H₂O), crystal
- Sorbitol, 70% aqueous solution
- Standard Stock Solution • Dissolve 0.5434 g potassium sulfate (K₂SO₄) in DI water, and bring to 1-L volume. This solution contains 100 ppm SO₄-S (Stock Solution).
- Prepare a series of Standard Solutions from the Stock Solution as follows: Dilute 5, 10, 20, 30, 40, and 50 mL Stock Solution to 100-mL numbered flasks by adding 0.15 % calcium chloride dihydrate solution, and then bring to volume. These standards contain 5, 10, 20, 30, 40 and 50 ppm SO₄-S, respectively.

■ **PROCEDURE**

A. Extraction

- Weigh 5 g air-dry soil (2-mm) into a 150-mL Erlenmeyer flask.
- Add 25 mL 0.15% CaCl₂.2H₂O solution (do not use a rubber stopper, or wrap the rubber stopper in thin polyethylene. Errors result from gradual oxidation of S compounds present in the stopper).
- Shake for 30 minutes on a reciprocal shaker (180+ oscillations per minute).
- Filter the suspension through Whatman No. 42 filter paper. This procedure yields almost colorless extracts.

B. Measurement

- Pipette 10-mL aliquot of the extract into a 50-mL test tube, or a smaller aliquot diluted to 10 mL with DI water.
- Add 1 mL 6 M HCl solution followed by 5 mL 70 % sorbitol solution from a pipette with an enlarged jet.



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- Add about 1 g BaCl₂.2H₂O crystals (using a measuring spoon).
- Shake vigorously (on a test tube shaker for 30 seconds) to dissolve the BaCl₂.2H₂O and obtain a uniform suspension.
- Prepare a standard curve as follows: • Pipette 10 mL of each standard (0 – 50 ppm), and proceed as for the samples. • Also make a blank with 10 mL 0.15 % CaCl₂.2H₂O solution, and proceed as for the samples
- Read the absorbance (turbidity) of the blank, standards, and samples on the Spectrophotometer at 470-nm wavelength.
- Prepare a calibration curve for standards, plotting absorbance against the respective SO₄-S concentrations.
- Read SO₄-S concentration in the unknown samples from the calibration curve.
-

■ **CALCULATIONS REQUIRED:**

$$\text{Soluble SO}_4\text{-S (ppm)} = \text{ppm SO}_4(\text{from calibration curve}) * V/Wt$$

where: V = Total volume of the soil extract (mL)

Wt = Weight of air-dry soil (g)

■ **REFERENCE / RELATED DOCUMENTS:**

George Estefan, Rolf Sommer, and John Ryan 2013. Methods of Soil, Plant, and Water Analysis: A manual for the West Asia and North Africa region. International Center for Agricultural Research in the Dry Areas (ICARDA)



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DETERMINATION OF CALCIUM CARBONATE IN SOIL

■ **PURPOSE:**

Soil Calcium Carbonate

■ **PRINCIPLE:**

The CaCO₃ content is calculated from the weight of CO₂ lost after treating a sample with excess hydrochloric acid.

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Pipette, 10 ml
- Flask
- Balance

■ **REAGENTS/CHEMICALS REQUIRED:**

- Hydrochloric Acid (1 + 3, Acid + Water)

■ **METHOD:**

Weigh the flask. Add 10 ml HCl 3 N (1:3 acid, water) and weigh it. Add 10 g soil and weigh again. Shake gently and stay for two hours. Note the final weight of flask after 2 hours.

■ **CALCULATIONS REQUIRED:**

$$\begin{aligned} \text{Wt. of flask} &= A \\ \text{Wt. of flask + acid} &= B \\ \text{Wt. of flask + acid + soil} &= C \\ \text{Wt. of flask + acid + soil after 2 hours} &= D \\ \text{Wt. of CO}_2 \text{ lost} &= \text{Initial Wt. (C)} - \text{Final Wt. (D)} = E \end{aligned}$$

$$\% \text{ CaCO}_3 = \frac{E \times 227.4}{\text{Wt. of soil taken}}$$

■ **REFERENCE / RELATED DOCUMENTS:**

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C.



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GYPSUM REQUIREMENT OF SALINE SODIC AND SODIC SOIL

■ **PURPOSE:**

Assessment of Gypsum Requirement for Saline Sodic and Sodic Soil

■ **PRINCIPLE**

The main principle involve for the gypsum requirement soils is to replace exchangeable Na by cation calcium (Ca^{2+}) by agitating soil in Saturated Gypsum solution. Amount of Calcium adsorbed refer to soil gypsum requirement.

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Weighing balance
- Volumetric flask, 1000 ml
- Mechanical shaker
- Conical flask, 250 ml
- Plastic bottle, 250 ml
- Funnel with stand
- Pipette, 10 ml
- Burette, 50 ml

■ **REAGENTS/CHEMICALS REQUIRED:**

- Saturated gypsum solution: Dissolve 5.0 g $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ in 1-liter distilled water. Stopper the volumetric flask and shake by hand for one hour or for 10 minutes on reciprocating shaker. Filter and determine the calcium concentration of solution by Versenate method. The calcium concentration should be at least 28 me L⁻¹.
- EDTA 0.01 N. Dissolve 2.0g of EDTA and 0.05g $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ in water and dilute to volume of 1-L.
- Buffer solution: Ammonium chloride – ammonium hydroxide solution. Dissolve 67.5g of ammonium chloride in 570ml of concentration ammonium hydroxide and make volume to 1-L.
- EBT indicator. Dissolve 0.5g of EBT and 4.5g of hydroxylamine hydrochloride in 100ml of 95% ethanol.

■ **METHOD:**

Weigh 5.0 g air dried soil in a 250 ml capacity plastic bottle, add 100 ml saturated gypsum solution, stopper the bottle, shake by hand several times for half an hour or for 5 minutes in a mechanical shaker. Filter the suspension and. Take 5 ml of aliquot, add 7 – 10 drops of C and D each. Titrate against B from wine red to bluish end point.



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■ **CALCULATIONS :**

$$\text{Gypsum Req. (me100 g}^{-1}\text{)} = \frac{\text{Blank Reading} - \text{Sample Reading} \times 0.01 \times 1000 \times 2}{5.0}$$

5 g soil was added in 100 ml of gypsum saturated solution.

So, 1000 ml soil filtrate will correspond to 50 g air dry soil.

Therefore, in 50 g soil, Ca conc. = Ca conc. (me L⁻¹) of gyp. sol. - Ca+Mg conc. (me L⁻¹) of filtrate.

Ca conc. in 100 g soil = 100/50 = 2

Conversions:

Say

100 g soil has gypsum requirement = M me

Then gyp. req. for

Tonnes per acre 15 cm of soil = M x 0.86

Tonnes per ha 15 cm of soil = M x 2.12

Tonnes per acre 30 cm of soil = M x 1.72

Tonnes per ha 30 cm of soil = M x 4.25

■ **PRECAUTIONS/ SAFETY REQUIREMENTS:**

Never use oven dry soil sample as heating promotes the conversion of CaSO₄ 2H₂O to CaSO₄.1/2 H₂O (plaster of Paris) which is more soluble than that of CaSO₄. 2H₂O. Hence the gypsum requirement will be more than the actual.

■ **REFERENCE / RELATED DOCUMENTS:**

Schoonover, W. R. 1952. Examination of soil for alkali. Univ. of Calif. Ext. Service Berkely California. (Mimeographed).



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DETERMINATION OF TOTAL NITROGEN IN SOIL

■ **PURPOSE:**

Total Nitrogen Evaluation in Soil

■ **PRINCIPLE:**

This procedure involves digestion and distillation. The soil is digested in concentrated H_2SO_4 with a catalyst mixture to raise the boiling temperature and to promote the conversion from organic-N to NH_4-N . The NH_4-N from the digest is obtained by steam distillation, using excess NaOH to raise the pH. The distillate is collected in saturated H_3BO_3 , and then titrated with dilute H_2SO_4 .

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Kjeldhal distillation unit
- Balance
- Volumetric flasks, 100, 1000 & 2000ml
- Pipette, 10 ml
- Conical flask, 250 ml
- Burette, 50 ml
- Beaker, 50 & 1000 ml
- Wash bottle
- Digestion Block

■ **REAGENTS/CHEMICALS REQUIRED:**

- Sulphuric acid Conc. (reagent grade N free).
- Digestion mixture: $K_2SO_4 + CuSO_4$ in 9:1
- Sodium hydroxide solution (40%): Dissolve 400g sodium hydroxide in one-liter distilled water.
- Bromocresol green and Methyl red indicator : Dissolve 0.5 g BCG and 0.1 g methyl red in 100 ml of 95 % ethanol
- Boric acid solution (4%): Dissolve 40 g of boric acid in 1.0 liters of CO_2 free distilled water.
- 0.1 N H_2SO_4 solution
- Phenolphthalein indicator

STANDARDISATION OF NaOH 0.1 N:

REAGENTS:

A Potassium acid phthalate 0.1 N: Weigh 20.423 g of oven dried potassium acid phthalate, dissolve in distilled water and make the volume 1.0 liter.

B. Phenolphthalein indicator. Dissolve 1 g of phenolphthalein in 100 ml 50% ethanol (50 ml of ethanol and 50 ml distilled water) with constant stirring. Filter if precipitates are formed.

C. Sodium hydroxide 0.1 N: 4.0 g of sodium hydroxide is dissolved in distilled water and make volume one liter.



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Pipette 10 ml of 0.1 N potassium acid phthalate into a titration flask and add 1-2 drops of phenolphthalein, titrate against solution of sodium hydroxide to light pink color end point. Note the reading and adjust the normality of the base by

$$N_a \times V_a = N_b \times V_b$$

Standardization of H₂SO₄ 0.1 N:

Pipette out 10 ml of 0.1 N standardized base in a titration flask, add 1-2 drops of phenolphthalein indicator titrate against the acid to be standardized, to colorless endpoint. Note the volume of acid used. By using the above formula adjust the normality i.e., 0.1N. If less volume of acid to be standardized is used for 10 ml of standardized base, then dilute the acid or vice versa.

■ METHOD:

Weigh 5 g air dried, passed through 2.0 mm sieve soil sample and 1.0 g digestion mixture into clean and dry digestion tube. Add 10-15 ml concentrated commercial Sulphuric acid and heat at 420⁰C. Continue to heat until the color of the material changes from black to white/greenish white. Solidification should be avoided.

Distil digested material in same flask on the distillation unit using pre-defined parameters. Add few drops of indicator into collector solution(Boric Acid). Purple color will form and will be changed to golden yellow on distillation. Which is then titrated against 0.1 N H₂SO₄ for soil from golden yellow to a purple end point.

■ CALCULATIONS REQUIRED:

$$\%N = \frac{14.1 \times \text{ml of titrant for sample} - \text{ml of titrant for blank} \times N \text{ of acid}}{\text{weight of sample (g)} \times 10}$$

$$= \frac{0.00141 \times R \times 100}{5}$$

Factor for soil if weight of soil is 5.0 g

$$\% N = R - \text{blank} \times 0.0282$$

■ PRECAUTIONS/ SAFETY REQUIREMENTS:

Always run blank along with samples.

■ REFERENCE / RELATED DOCUMENTS:

Bremner, M. 1960. Kjeldhal Method. Agronomy – 9, 2nd Ed.



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DETERMINATION OF CATION EXCHANGE CAPACITY OF SOIL

■ **PURPOSE:**

Determination of the Capacity of the Cation Exchange of Soil

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Flame photometer
- Centrifuge machine
- Volumetric flask, 100 ml
- Cylinder, 50 ml
- Weighing balance
- Funnel with stand
- Burette, 50 ml

■ **REAGENTS/CHEMICALS REQUIRED:**

- Sodium acetate solution (saturating solution), 1.0 N: Dissolve 136 g sodium acetate trihydrate in distilled water and make volume upto 1 liter. Adjust pH approximately 8.2 using NaOH or CH₃COOH.
- Ethanol 95%
- Ammonium acetate solution (replacing solution) 1.0 N: Dissolve 77.08g in distilled water and make volume up to 1L and adjust pH at 7.0 with Ammonium Sulphate.
- **Sodium Stock Solution (1000 ppm)** Dissolve dried 2.5418 g of NaCl in 1 L deionized water (Desired working standards be prepared from it).

4. METHOD:

Weigh 5.0 g soil sample in a centrifuge tube. Add 33 ml of reagent A, stopper the tube, shake for 5-7 minutes, centrifuge for 5 minutes or till the clear supernatant solution in centrifuge at 1200-2000 rpm. Decant and discard. Repeat same process further two times with 33 ml of reagent A. Centrifuge, decant and discard the supernatant solution. Now add 33ml of B reagent to the tube, stopper, shake for 5-7 minutes. Decant and discard supernatant solution. Repeat the same procedure further two times with 33 ml of reagent B. Now add 33 ml of reagent C to the same tube, stopper, shake and centrifuge at 2000 rpm for 5-7 minutes. Decant the supernatant solution in the 100 ml volumetric flask. Repeat the same procedure for further two times with 33 ml of reagent C. Make the volume 100 ml with distilled water. This solution is ready for the determination of replaced Na by flame photometer.

■ **CALCULATIONS:**

$$\text{Cation exchange capacity (CEC) } C \text{ mol (+) Kg}^{-1} = \frac{\text{Na conc. of ext. (me L}^{-1})}{\text{wt. of sample (g)}} \times 10$$

■ **REFERENCE / RELATED DOCUMENTS:** United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 101.



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DETERMINATION OF EXTRACTABLE MICRONUTRIENTS IN SOIL

■ PURPOSE:

Determination of Plant Available Micronutrients in Soil

■ PRINCIPLE

The DTPA method is an important and widely used chelating agent, which combines with free metal ions in the solution to form soluble complexes of elements. The DTPA method has a capacity to complex each of the micronutrient cations as 10 times of its atomic weight

■ EQUIPMENTS/APPARATUS REQUIRED:

- Atomic Absorption Spectrophotometer
- Plastic beaker
- Mechanical Shaker
- Filter paper
- Funnel
- Test tubes

■ REAGENTS/CHEMICALS REQUIRED:

- 0.005 M DTPA, 0.01 M CaCl₂, and 0.1 M TEA (tri-ethanol amine, adjusted to pH 7.3 with dilute HCl. Dissolve 3.934 g of DTPA and 2.94 g of CaCl₂ and 25.3 ml of TEA in approximately 200 ml of distilled water. After sufficient time for DTPA to dissolve, make the volume 2 liter. Adjust the pH 7.3 with 1:1 HCl while stirring.

■ METHOD:

Weigh 10 g of soil and add 20 ml of DTPA solution. Shake continuously for 2 hours on horizontal shaker and filter. A blank solution (0 ppm) containing all reagents except soil should be run with samples as blank. Read on concentration mode by atomic absorption spectrophotometer Prepare at least 3-4 standard using DTPA as matrix for each element with a range as following

For Zn and Cu 0.05,1.0,1.5,2.0 ppm

For Fe and Mn 0,5,10,20, 30, ppm

■ CALCULATIONS:

Micronutrients (ppm) = reading × 2

■ PRECAUTIONS/ SAFETY REQUIREMENTS:

Always run blank along with samples.

■ REFERENCE / RELATED DOCUMENTS:

Lindsay and Norvell. 1978. Development of a DTPA Soil Test for Zinc, Iron, Manganese, and Copper, Soil Science Society of America Journal. Volume 42, Issue 3



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DETERMINATION OF AVAILABLE BORON IN SOIL

■ **PURPOSE:**

Boron Assessment in Soil

■ **PRINCIPLE:**

■ Boron in soil extracts is measured calorimetrically using reagent Azomethine-H in presence of buffer solution

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Spectrophotometer.
- Weighing balance
- Poly propylene volumetric flask
- Pipette
- Funnel with stand
- Wash bottle
- Water bath
- Burner
- Beaker, 50 ml

■ **REAGENTS/CHEMICALS REQUIRED:**

A. Buffer solution: Dissolve 250 g ammonium acetate and 15 g of ethylenediamine tetra acetic acid (EDTA) in 400 ml of distilled water. Slowly add 125 ml glacial acetic acid and mix.

B. Azomethine – H reagent: Dissolve 0.45 g of reagent in 100 ml of 1 % L-ascorbic acid solution. Fresh reagent should be prepared and store in a refrigerator.

C. Boron standard solution: Dissolve 5.709 g of boric acid (H_3BO_3) in distilled water (for hot water extraction) or in 0.05 M HCl for HCl extraction and adjust the volume 1000 ml . This will be of 1000 ppm of boron solution. Make sub-stock solution to 100 ml of 100 ppm B. Then make working standard as desired.

$$C_1V_1 = C_2V_2$$

$$100 \times V_1 = 0.5 \times 100$$

$$5 \times 100$$

$V_1 = \frac{0.5 \times 100}{1000} = 0.5$ ml of sub-stock solution to make 0.5 ppm B working standard.

$$1000$$

D. 0.05 M HCl

4. METHOD:

■ **HCl EXTRACTION**

Ten grams soil was shaken with 20 ml of 0.05 M HCl for 5 minutes and then filter. Transfer 1 ml aliquot in 50 ml polypropylene volumetric flask, add 2 ml buffer solution, add 2 ml azomethine-H reagent and mix. After 30 minutes read absorbance at 430 nm on spectrophotometer at concentration mode or by graph using standard curve prepared Boron standards are prepared in



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0.05 M HCl for HCl extraction and in distilled water for hot water extraction. Using 420 nm wavelength determine boron (ppm) by spectrophotometer using color developing method.

■ CALCULATIONS:

$$B \text{ (ppm)} = R \times 100^*$$

Whereas

R = spectrophotometer reading

100* = total dilution factor.

■ PRECAUTIONS/ SAFETY REQUIREMENTS:

Always run blank for accuracy.

■ REFERENCE / RELATED DOCUMENTS:

Bingham. 1982. Methods of soil analysis. Part II. Amer. Soc. Agron. USA.



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CHAPTER-3 WATER ANALYSIS

ELECTRICAL CONDUCTIVITY (EC) OF WATER

■ **PURPOSE:**

Determination of Electrical Conductivity (EC)

■ **PRINCIPLE:**

Electrical Conductivity (EC) is the measure of the ability of a solution to carry an electric current or the concentration of soluble salts in the sample at any particular temperature. This ability is directly related to the concentration of ions in the water. These conductive ions come from dissolved salts and inorganic materials such as alkalis, chlorides, sulphides and carbonate compounds. The EC is measured in the field or in the laboratory using a conductivity meter, which is basically a Wheatstone bridge that measures the resistance of the solution between two parallel platinum electrodes.

■ **EQUIPMENTS/APPARATUS REQUIRED:**

Conductivity Meter

■ **REAGENTS/CHEMICALS REQUIRED:**

0.01 N KCl: Dissolve 0.7456 g KCl in 1 liter of distilled water.

■ **METHOD:**

The instrument must be standardized with 0.01 N KCl solution. The EC of this standard solution is 1.413 dS m⁻¹ at 25 °C. The cell constant (K) can be calculated by the formula.

$$K = \frac{1.413 \text{ dS m}^{-1}}{\text{Observed reading in dS m}^{-1}}$$

The cell constant is necessary when 0.01 N KCl solution gives an EC values different from 1.413 dS m⁻¹. In this case, samples readings are multiplied by the cell constant.

Wash the conductance cell with water sample Dip the cell (EC probe) into the water sample making sure that there is no air bubble in it. Conductivity corrected at 25 °C will be displayed in mS cm⁻¹ or dS m⁻¹.

■ **CALCULATIONS REQUIRED:**

EC of water = Observed EC × K

■ **PRECAUTIONS/ SAFETY REQUIREMENTS:**

The conductivity meter should be standardized daily or as desired depending upon the number of samples being tested.

■ **REFERENCE / RELATED DOCUMENTS:**

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 89-91.



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DETERMINATION OF CARBONATES AND BICARBONATES IN WATER

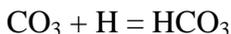
■PURPOSE:

Determination of the concentration of carbonates and bicarbonates in water

■PRINCIPLE

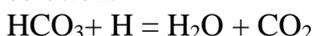
Carbonates

When the pH value of a sample of natural water is above 8.4, the CO₃ is present, normally as sodium carbonate. The carbonate ion is converted to HCO₃. The amount of acid used a measure of the carbonate present.



Bicarbonate

Bicarbonate ions react with mineral acid and release carbon dioxide (CO₂) into the solution.



The pH value at complete neutralization being about 3.8, HCO₃ ions are present. Thus HCO₃ may be measuring by titration with mineral acid to a pH 3.8, either potentiometrically or using an indicator unaffected by CO₂. Methyl orange is suitable and gives a good color change from green through grey (end-point) to red, which avoids the need for a matching buffer solution.

■ EQUIPMENTS/APPARATUS REQUIRED:

- Pipette, 50ml
- Conical flask, 100ml
- Cylinder, 50ml
- Wash bottle
- Burette, 50ml

■ REAGENTS/CHEMICALS REQUIRED:

- A. Phenolphthalein 1%: Dissolve 1.0 g phenolphthalein in 100 ml ethanol with constant stirring, Filter if needed.
- B. Methyl Orange 0.1%: Dissolve 0.1g methyl orange in 100ml distilled water.
- C. Sulphuric Acid 0.1 N (standardized)

■ METHOD:

Pipette 50ml water sample in conical flask, add 1-3 drops of reagent A. If no color appears, carbonates are absent. If pink color appears, carbonates are present. Titrate it against 0.1 N H₂SO₄ to colorless end point. Take three readings. Reserve the flask along with its contents for bicarbonates determination.

To the same conical flask (after colorless end point), add 1-2 drops of methyl orange, titrate against 0.1 N H₂SO₄ from golden yellow to a light pink or light orange color end point. Take three readings. Reserve the flask for chloride determination.

■CALCULATIONS REQUIRED:

$$\text{CO}_3^{-2} \text{ (me L}^{-1}\text{)} = \frac{2R_1 \times \text{Normality of H}_2\text{SO}_4}{\text{-----}} \times 1000$$



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Aliquot (ml)

Or

Volume of aliquot taken = 50 ml

Volume of 0.1 N H₂SO₄ used = Final Reading - Blank Reading
 = R₁

Actual Volume of 0.1 N H₂ SO₄ used = 2 x R₁ = 2R₁

* 2R₁ x 0.1 N H₂SO₄

Carbonates (me L⁻¹) = $\frac{2R_1 \times 0.1 \text{ N H}_2\text{SO}_4}{50} \times 1000 = 4 R_1$

(R₂-R₁) x Normality of H₂SO₄

Bicarbonates (me L⁻¹) = $\frac{(R_2 - R_1) \times \text{Normality of H}_2\text{SO}_4}{\text{Aliquot (ml)}} \times 1000$

Or

Volume of aliquot taken = 50 ml

Volume of 0.1 N H₂SO₄ used = Final reading -Initial reading
 = R₂

Actual volume of 0.1 N H₂SO₄ used = R₂ - R₁

(R₂-R₁) x 0.1 N H₂SO₄

Bicarbonates (me L⁻¹) = $\frac{(R_2 - R_1) \times 0.1 \text{ N H}_2\text{SO}_4}{50} \times 1000$
 = 2 (R₂-R₁)

* This reaction is completed in two steps:

R₁ is the amount of acid used to convert the carbonates to bicarbonates.

R₂ is the total amount of acid used to neutralize the bicarbonates present in the sample after titration. Therefore, the actual amount of acid used for neutralizing the originally present HCO₃ would be R₂-R₁.

■ PRECAUTIONS/ SAFETY REQUIREMENTS:

- Always run blank for accuracy.

■ REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 89-91.



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DETERMINATION OF CHLORIDE IN WATER

■ **PURPOSE:**

Determination of Chloride in Water

■ **PRINCIPLE**

The determination of Cl is done by AgNO₃ (Mohr's titration) method, which is based upon the fact that in solution containing Cl and chromate (Cr). Silver reacts with all the Cl and precipitates before the reaction with chromate begins. The appearance of the brick-red colour of the silver chromate precipitate is the end-point of the titration.

■ **EQUIPMENTS/APPARATUS REQUIRED:**

- Pipette, 50ml
- Conical flask, 100ml
- Cylinder, 50ml
- Wash bottle
- Burette, 50ml
-

■ **REAGENTS/CHEMICALS REQUIRED:**

A. Potassium Chromate (K₂CrO₄), 5 % solution: Dissolve 5.0 g of potassium chromate in 50 ml distilled water and add 1 N (saturated solution) AgNO₃ drop wise until red precipitate is produced. The solution is filtered, and filtrate is diluted to 100 ml.

B. Silver Nitrate (AgNO₃), 0.05 N. Dissolve 8.494 g silver nitrate in distilled water and dilute to 1 liter. Keep in a brown bottle to avoid photolysis.

■ **METHOD:**

To the same conical flask after light orange yellow end point, add 3-4 drops of reagents A. While stirring, titrate under bright light with reagent B., to a brick red precipitate./permanent reddish-brown color.

■ **CALCULATIONS REQUIRED:**

$$\text{Cl (me L}^{-1}\text{)} = \frac{(\text{ml of AgNO}_3 \text{ for sample -ml of AgNO}_3 \text{ for blank)} \times \text{N}}{\text{Aliquot (ml)}} \times 1000$$

Or

Volume of aliquot taken = 50 ml

Volume of 0.05 N AgNO₃ used = Final reading - initial reading
 = R₃

$$\text{Cl (me L}^{-1}\text{)} = R_3 \times 0.05 \text{ N AgNO}_3 \times 1000/50$$

$$= R_3$$

Note: Always run a blank along with actual titration.



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Q. Why we first neutralize CO_3 & HCO_3 before Cl titration?

If we don't neutralize CO_3 & HCO_3 , AgNO_3 will react with CO_3 & HCO_3 to form AgHCO_3 . So more AgNO_3 will be utilize for chloride titration.

■ PRECAUTIONS/ SAFETY REQUIREMENTS:

Always run blank for accuracy.

■ REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 89-91.



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DETERMINATION OF CALCIUM AND MAGNESIUM IN WATER

■ PURPOSE:

Determination of Calcium and Magnesium in Water

■ PRINCIPLE

EDTA-disodium salt solution is used to chelate Ca + Mg. Calcium is separately estimated by the versenate method using ammonium purpurate (Murexide) indicator, when the pH is made sufficiently high that the Mg is largely precipitated as hydroxide and an indicator is used that combines with Ca only.

■ EQUIPMENTS/APPARATUS REQUIRED:

- Burette
- China dish
- Pipette, 10 ml
- Cylinder, 50 ml
- Glass rod
- Wash bottle

■ REAGENTS/CHEMICALS REQUIRED:

A. NH_4Cl - NH_4 OH buffer solution: Dissolve 67.5 g NH_4Cl in 570 ml concentrated NH_4OH , make the volume up to 1 liter.

B Sodium hydroxide (NaOH), 4 N: Dissolve 160 g NaOH in distilled water, make the volume of the solution to 1 liter .

C. Standard calcium chloride solution (CaCl_2) , 0.01 N: Dissolve 0.5 g CaCl_2 pure (calcite crystals) in 10 ml of approx. 3 N (1+3) HCl and dilute to a volume of 1 litre.

D. Eriochrome black-T indicator (EBT): Dissolve 0.5 g of EBT and 4.5 g hydroxyl amine hydrochloride ($\text{NH}_2\text{O.H. HCl}$, $\text{H}_3\text{NO.HCl}$) in 100 ml of 95 % ethanol.

E. Ammonium Purpurate indicator ($\text{C}_8\text{H}_4\text{N}_5\text{O}_6 \cdot \text{NH}_4$): Thoroughly mix 0.5 g ammonium purpurate with 100 g of potassium sulphate (K_2SO_4).

F. EDTA (Versenate) solution, 0.01 N: Dissolve 2.0 g of EDTA and 0.05 g magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) in distilled water and dilute to a volume of 1 litre . Standardized the solution against reagent C using given titration procedure.

■ METHOD:

Calcium: Pipette 10 ml of water sample in a conical flask. Add 5 drops of reagent B and add 50 mg of E. Titrate against F from orange red to lavender/purple end point. When closed to the endpoint, reagent F should be added drop wise (each drop after 5-10 second) as the colour change is not instantaneous.

Calcium + Magnesium: Pipette 10 ml of aliquot in a conical flask. Add 10 drops of reagent A, 3-4 drops of reagent D. Titrate against reagent F to a change in colour from wine red to bluish green.



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■ CALCULATIONS REQUIRED:

$$\text{Ca}^{++} \text{ or } \text{Ca}^{++} + \text{Mg}^{++} \text{ (me L}^{-1}\text{)} = \frac{\text{ml of ETDA sol. for sample} - \text{ml of EDTA for blank} \times \text{N}}{\text{Aliquot in ml}} \times 1000$$

$$= \frac{R_1 - R_2 \times 0.01 \times 1000}{10}$$

$$= (R_1 - R_2) = R \text{ me L}^{-1}$$

$$\text{Mg}^{++} \text{ (me L}^{-1}\text{)} = (\text{Ca}^{++} + \text{Mg}^{++}) - \text{Ca}^{++} \text{ (All expressed as meL}^{-1}\text{)}$$

■ PRECAUTIONS/ SAFETY REQUIREMENTS:

Always run a blank.

■ REFERENCE / RELATED DOCUMENTS:

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C.



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DETERMINATION OF SULPHATE IN WATER

■ **PURPOSE:**

Determination of Sulphate in water by precipitation with barium chloride

■ **EQUIPMENTS/APPARATUS**

- Spectrophotometer
- Electric balance
- Volumetric flask
- Pipette

■ **REAGENTS/MEDIAS:**

A. Acid Mixture: 125 ml HNO₃ + 250 ml Acetic Acid + 100 ml H₃PO₄ (85 %) Dilute to one litre with deionized water.

B. Acid Sulphate solution (20 ppm S): 86 ml conc. HCl + 100 ml S solution (100 ppm). Make volume upto 500 ml with deionized water.

C. BaCl₂.2H₂O Crystal: 20-60 mesh crystal

E. Gum Acacia Solution: Dissolve 0.5 g gum acacia in 50 ml deionized water. Mix and filter. Add 50 ml acetic acid. Filter if necessary.

F. SO₄ – Standards: Standards of 5, 10, 15 and 20 ppm from 100 ppm stock solution

■ **METHOD:**

Take 5 ml sample in a 50 ml volumetric flask. Add 5 ml mixed acid reagent and 1 ml acid sulphate solution and mix. Add 0.5 g BaCl₂.2H₂O crystals. Let stand undisturbed for 3 minutes and then mix. Add 1 ml gum acacia reagent and mix. Make total volume 50 ml. Take readings of samples and standards at 420-450 nm between 3 to 8 minutes after final shaking. Run reagent blank.

■ **CALCULATIONS:**

Sulphate concentration (ppm) = Sulphate conc. (ppm) × Dilution factor

■ **REFERENCE / RELATED DOCUMENTS:**

Verma, B. C. 1977.



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DETERMINATION OF SODIUM (Na) AND POTASSIUM (K) IN WATER

■ PURPOSE:

Determination of Sodium (Na) and Potassium (K) in water

PRINCIPLE:The estimation of Na and K is based on the emission spectroscopy, which deals with excitation of electrons from ground state to a higher energy state and coming back to its original state with the emission of light

■ EQUIPMENTS/APPARATUS REQUIRED:

Flame Photometer

■ REAGENTS/CHEMICALS REQUIRED:

Preparation of stock solutions

- Sodium: Dissolve 2.5435g dry NaCl in 1-L, it will be 1000ppm Na.
- Potassium: Dissolve 1.9103 g dry KCl in 1-L, it will be 1000ppm K.
- However, both the stock solutions in combined form can easily be prepared by dissolving the said amounts of both the salts in exactly one litre distilled water. Store in a cool and dry place.

Preparation of working standards:

Stock solutions so prepared will be used for the preparation of working standards by using the formula:

$$C_1V_1 = C_2V_2$$

Whereas

C_1 = Concentration of stock solution in ppm

V_1 = Volume to be taken of stock solution in ml

C_2 = Concentration of Na or K to be required in ppm

V_2 = Total volume to be required in ml

$$C_2 \text{ (ppm required)} = \frac{V_1 \text{ (ml of known solution)} \times C_1 \text{ (ppm of known solution)}}{V_2 \text{ (Total volume to be made)}}$$

Working standard solution can be prepared of 10, 20, 30, 50 Na and 2,4,6, 8,10 for K, or According the instrument sensitivity and accuracy



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■ **METHOD:**

For Na

1. Operate Flame Photometer according to the instructions provided for the equipment.
2. Run a series of suitable Na standards and draw a calibration curve.
3. Measure Na in the samples (water samples) by taking the emission readings by direct feeding water sample to Flame Photometer. Dilute sample, if required, to make it fit into calibration curve range

For K

1. Operate Flame Photometer according to the instructions provided for the equipment.
2. Run a series of suitable Na standards and draw a calibration curve.
3. Measure Na in the samples (water samples) by taking the emission readings by direct feeding water sample to Flame Photometer. Dilute sample, if required, to make it fit into calibration curve range

■ **CALCULATIONS REQUIRED:**

K or Na (ppm) = ppm from calibration cure of standards.

■ **REFERENCE / RELATED DOCUMENTS:**

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C. pp. 96-97.



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DETERMINATION OF SODIUM ADSORPTION RATIO (SAR) AND RESIDUAL SODIUM CARBONATE (RSC) OF WATER

■ **PURPOSE:**

Determination of Sodium Adsorption Ratio (SAR) and Residual Sodium Carbonate (RSC) of Water

■ **CALCULATIONS REQUIRED:**

$$\text{SAR} = \text{Na}^+ / [(\text{Ca}^{++} + \text{Mg}^{++})/2]^{1/2}$$

All the cations are expressed as me L⁻¹.

Residual sodium carbonate can be calculated by using following equation

$$\text{RSC, me L}^{-1} = (\text{CO}_3^{-} + \text{HCO}_3^{-}) - (\text{Ca}^{++} + \text{Mg}^{++})$$

All expressed in me L⁻¹.

■ **REFERENCE / RELATED DOCUMENTS:**

United States Salinity Laboratory Staff. 1954. Diagnosis and improvement of saline and alkali soils. USDA Handbook 60. Washington, D.C.

DETERMINATION OF HEAVY METALS IN WATER

■ **PURPOSE:**

Determination of Heavy Metals in Water

■ **EQUIPMENTS/APPARATUS REQUIRED:**

Atomic Absorption Spectrophotometer / ICP-OES

■ **REAGENTS/CHEMICALS REQUIRED:**

Standard solutions for the respective heavy metals

■ **METHOD:**

Determine the heavy metals concentration using atomic absorption spectrophotometer / ICP-OES after standardizing. Dilute the sample if necessary.

■ **CALCULATIONS REQUIRED:**

Direct concentration is given on these instruments

■ **REFERENCE / RELATED DOCUMENTS:**

Standard method for the examination of water and wastewater. (APHA, AWWA WEF) 20th Edition, 1998.



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DETERMINATION OF BORON IN WATER

■ PURPOSE:

Determination of Boron in Water

■ PRINCIPLE:

The B is measured calorimetrically using Azomethine-H (Bingham, 1982)

■ EQUIPMENTS/APPARATUS REQUIRED:

- Poly propylene volumetric flask, 50 ml
- Pipette, 2 ml
- Weighing balance
- Funnel with stand
- Wash bottle
- Beaker, 50 ml
- Spectrophotometer.

■ REAGENTS/CHEMICALS REQUIRED:

A. Buffer solution: Dissolve 250 g ammonium acetate and 15 g of ethylenediamine tetra acetic acid (EDTA) in 400 ml of distilled water. Slowly add 125 ml glacial acetic acid and mix.

B. Azomethine – H reagent: Dissolve 0.45 g of reagent in 100 ml of 1 % L-ascorbic acid solution. Fresh reagent should be prepared and store in a refrigerator.

C. Boron standard solution: Dissolve 5.709 g of boric acid (H_3BO_3) in distilled water (for hot water extraction) and in 0.05 M HCl for HCl extraction and adjust the volume 1000 ml . This will be of 1000 ppm of boron solution. Make sub-stock solution to 100 ml of 100 ppm B. Then make working standard as desired.

$$C_1V_1 = C_2V_2$$

$$100 \times V_1 = 0.5 \times 100$$

$$5 \times 100$$

$$V_1 = \frac{0.5 \times 100}{1000} = 0.05 \text{ ml of sub-stock solution to make 0.5 ppm B working standard.}$$

1000

4. METHOD:

Filter water sample in boron free apparatus (Polypropylene). If boron free apparatus is not available, then Pyrex glass apparatus should be dipped in concentrated HCl for a week and then used.

Boron standards (0.5, 1.0, 1.5, 2.0 and 2.5 ppm) were prepared in distilled water. At 420 nm wavelength determine boron (ppm) by spectrophotometer after developing color using azomethine – H.

Transfer 1 ml sample in 50 ml polypropylene volumetric flask, add 2 ml buffer solution, add 2 ml azomethine- H reagent and mix. After 30 minutes read absorbance by spectrophotometer directly at concentration mode or by graph using standard curve prepared from standards readings.



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■ **CALCULATIONS REQUIRED:**

$$B \text{ (ppm)} = R \times \text{dilution factor}$$

Whereas

R = spectrophotometer reading

■ **PRECAUTIONS/ SAFETY REQUIREMENTS:**

Always run blank for accuracy.

■ **REFERENCE / RELATED DOCUMENTS:**

Gaines, T. P. and G. A. Mitchell. 1979. Commun. Soil Sci. Plan Anal. 10:1099-1108.



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CHAPTER NO 4

PLANT ANALYSIS

Plant and soil testing enables scientific assessment of the needs of the plant for nutrient elements and of the capacity of the soil to supply them. The nutrient elements enter the plant in ionic form from the soil solution. Ion transport to the root surface may take place through ion diffusion and bulk transport (mass flow). Mass flow is the sweeping along of ions as water moves to the root. It is particularly important for ions that are not absorbed on soil colloids such as nitrates and sulphates. Because of mass flow, a plant deficient in N can extract all the nitrates from the soil. Potassium, phosphate and the micronutrient cations are absorbed on soil colloids with various degrees of affinity and are greatly retarded in movement with the soil water. Diffusion (movement along a concentration gradient) is the main mode of transport from the solid phase to the root surface for these non-mobile ions. There is also a possibility that some of them are absorbed by a direct exchange of an ion, usually hydrogen, between the root surface and ions absorbed on soil colloids. Most ions are sorbed by the root against a concentration gradient and, thus, the process involves the use of metabolic energy. The fact that nutrient uptake is an active process explains some of its peculiarities. Plants not only accumulate nutrients against a concentration gradient, they are also able to select from the nutrients at the root surface according to their requirements (preferential uptake).

Knowledge of nutrient concentration in growing plants can serve as a tool for correcting any deficiencies where carried out early enough to safeguard yield. The nutrient uptake by a healthy crop, which has attained its growth and yield potential, is taken as the effective requirement for the crop. It can also be used to evaluate the efficacy of a recent application. The information it provides can help to plan nutrient application in subsequent crops on that field or to compute nutrient removals in relation to productivity and nutrient balance sheets.

Whole plant analysis is conducted in order to determine the total nutrient uptake (which is usually carried out on the shoot). For plant analysis to be meaningful as a diagnostic tool, the collection of particular plant parts (tissue) at the right stage of growth for analysis is very important. Plant leaves are considered the focus of physiological activities. The concentrations of leaf nutrients appear to reflect changes in mineral nutrition.



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Determination of Boron in Plant

■ PURPOSE:

Determination of Boron in Plant

■ EQUIPMENTS/APPARATUS:

- Poly propylene volumetric flask
- Pipette
- Weighing balance
- Funnel with stand
- Wash bottle
- Water bath
- Burner
- Beaker
- Spectrophotometer.

■ REAGENTS/MEDIAS:

A. Buffer solution: Dissolve 250 g ammonium acetate and 15 g of ethylenediamine tetra acetic acid (EDTA) in 400 ml of distilled water. Slowly add 125 ml glacial acetic acid and mix.

B. Azomethine – H reagent: Dissolve 0.45 g of reagent in 100 ml of 1 % L-ascorbic acid solution. Fresh reagent should be prepared and store in a refrigerator.

C. Boron standard solution: Dissolve 0.114 g of boric acid (H₃BO₃) in distilled water the volume 1000 ml. This will be of 20 ppm of boron stock solution. Then make working standard as desired.

$$C_1V_1 = C_2V_2$$

■ METHOD:

Weigh 1.0g plant sample in crucible. Ash at 550 0C in furnace. Cool and add 10 ml 0.36 N H₂SO₄. Make volume as desired. Filter through Whatman No. 42. Adopt same steps as in soil analysis for color development and procedure.

■ CALCULATIONS:

$$B(\text{ppm}) = \frac{\text{ppm B (from calibration curve)} \times A}{Wt}$$

A= Total volume of extract (ml)

Wt= Wt of dry plant (g)

■ CAUTIONS / SAFETY REQUIREMENTS:

Always run a blank for accurate results.

■ REFERENCE / RELATED DOCUMENTS:

Gaines, T. P. and G. A. Mitchell. 1979. Commun. Soil Sci. Plan Anal. 10:1099-1108



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Determination of Micronutrients and Heavy Metals in Plant

■ PURPOSE:

Determination of Micronutrients and Heavy Metals in Plant

■ EQUIPMENTS/APPARATUS REQUIRED:

- Atomic Absorption Spectrophotometer / ICP – OES
- Digestion tubes
- Digestion block
- Fume hood
- Volumetric flask
- Pipette
- Grinder
- Drying oven
- Weighing balance.

■ REAGENTS/MEDIAS REQUIRED:

A. Perchloric acid and Nitric Acid mixture (1:2): Add 500 ml (70%) perchloric acid in 1000 ml HNO₃ (69-71 %), mix well, cool and store in an amber glass bottle.

B. Standard solutions for the desired micronutrients

■ METHOD:

Weigh 1 g sample and add 25 ml Nitric acid + Perchloric acid (2:1) solution. Digest at 150 to 175 0C until the clear liquid obtained. Make the volume 50ml with distilled water and filter. Prepare the standards as following:

Zn and Cu 0, 0.25, 0.50, 1.00 ppm*

Fe and Mn 0, 0.5, 1.0, 2.0 ppm*

* Range may be extended according to the sample nature.

Analyse on atomic absorption spectrophotometer using respective lamp.

■ CALCULATIONS REQUIRED:

Micronutrients (ppm) = Reading x 50

■ CAUTIONS / SAFETY REQUIREMENTS:

Always run a blank for accurate results.

■ REFERENCE / RELATED DOCUMENTS:

Jones, Jr. J. B., B. Wolf and H. A. Mills. 1991. Micro-Macro Publishing Inc., Athens, GA, USA.



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Determination of Nitrogen in Plant

■ PURPOSE:

Determination of Nitrogen in Plant

■ EQUIPMENTS/APPARATUS REQUIRED:

- Plant Grinder
- Drying oven
- Digestion tubes
- Digestion block
- Weighing balance
- Distillation unit

■ REAGENTS/MEDIAS REQUIRED:

A. Sulphuric acid Conc. (reagent grade N free).

B. Digestion mixture: $K_2SO_4 + CuSO_4 :: 9:1$

C. Sodium hydroxide solution (40%): Dissolve 400g sodium hydroxide in one litre distilled water.

D. Bromocresol green and Methyl red indicator : Dissolve 0.5 g BCG and 0.1 g methyl red in 100 ml of 95 % ethanol

E. Boric acid solution (4%): Dissolve 400 g of boric acid (H_3BO_3 f. wt. 61.83) in 1.0 litres of CO_2 free distilled water.

F. 0.1 N Standardised H_2SO_4 solution

G. Phenolphthalein indicator

■ METHOD:

Digestion

Weigh 1.0 g or less dried, ground, sieved and plant sample in digestion tube. Add 15 ml conc. Analar H_2SO_4 and 1.0 g digestion mixture ($K_2SO_4 + CuSO_4 @ 9:1$). Heat at $450^\circ C$ on digestion block for two hours after attaining the required temperature or till clear transparent to light yellowish or light green solution in the digestion tubes. Make volume as desired for further analysis.

Distillation



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Distillate appropriate volume on the distillation unit. Collect distillate in 20-25 ml 4% boric acid in the receiver. Add few drops of indicator. Purple color will form and will be changed to golden yellow on distillation. Which is then titrated against 0.1 N H₂SO₄ from golden yellow to a purple end point.

■ **CALCULATIONS:**

14.1 x ml of titrant for sample - ml of titrant for blank x N of acid x d.f.

%N= -----

weight of sample (g) x 10

■ **CAUTIONS / SAFETY REQUIREMENTS:**

Always run blank for accurate results

■ **REFERENCE / RELATED DOCUMENTS:**

Jones, Jr. J. B. 1991. Micro-Macro Publishing Inc., Athens, GA, USA.



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Determination of Phosphorus in Plant

■ PURPOSE:

Determination of Phosphorus in Plant

■ EQUIPMENTS/APPARATUS REQUIRED:

- Spectrophotometer
- Digestion tubes
- Digestion block
- Fume hood
- Volumetric flask
- Pipette
- Grinder
- Drying oven
- Weighing balance.

■ REAGENTS/MEDIAS REQUIRED:

A. Ammonium Heptamolybdate-Ammonium Vanadate in Nitric Acid is prepared by mixing the following solutions in equal amounts.

(a) 100 ml of 5% ammonium molybdate solution

(b) 100 ml of 0.25% ammonium vanadate solution.

(c) 100 ml of dilute HNO₃ (HNO₃ : H₂O::1:3)

B. Ammonium molybdate solution 5%

Dissolve 50 g ammonium molybdate, in warm distilled water (50°C), transfer to one litre volumetric flask and make up the volume after cooling

C. Ammonium vanadate solution, 0.25%

Dissolve 2.5 g ammonium vanadate in litre boiling distilled water, cool and add 20 ml nitric acid and make up the volume upto the mark.

D. Perchloric acid and Nitric Acid mixture (1:2): Add 500 ml (70%) perchloric acid in 1000 ml HNO₃ (69-71 %), mix well , cool and store in an amber glass bottle.

Stock Solution

Dissolve 4.3937 g potassium dihydrogen phosphate (KH₂PO₄) in 1000 ml distilled water it will be 1000ppm phosphorus solution. Prepare a series of standard containing 0.25,0.50,1.0, 2.0 3.0, 4.0, ppm phosphorus by the formula.



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C1V1=C2V2

■ **METHOD:**

Digestion

Weigh 1.0 g, ground, sieved and free of contamination plant sample in digestion tube. Add acid mixture. Heat at 150-175°C on digestion block till white to slight yellowish plant material color in the digestion tube. Make volume 100 ml with distilled water.

Pipette 10 ml of the aliquot in 100 ml volumetric flask and add 10 ml Ammonium-Vanadomolybdate reagent and make the volume upto mark with distilled water. After one hour absorbance is measured at 430 nm on Spectrophotometer.

■ **CALCULATIONS REQUIRED:**

$$\%P = \text{ppm P (from calibration curve)} \times \frac{R \times 100}{\text{wt} \times 10000}$$

Where R = Ratio between total volume of digest / aliquot and the digest / aliquot volume used for measurement.

Wt = wt of dry plant

■ **CAUTIONS / SAFETY REQUIREMENTS:**

Always run a blank for accurate results.

■ **REFERENCE / RELATED DOCUMENTS:**

Jones, Jr. J. B., B. Wolf and H. A. Mills. 1991. Micro-Macro Publishing Inc., Athens, GA, USA.



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Determination of Potassium in Plant

■ PURPOSE:

Determination of Potassium in Plant

■ EQUIPMENTS/APPARATUS REQUIRED:

- Flame photometer
- Electronic balance
- Volumetric flask;
- Pipette;
- Wash bottle
- Digestion tubes
- Digestion block
- Fume hood
- Volumetric flask
- Pipette
- Grinder
- Drying oven
- Weighing balance.

■ REAGENTS/MEDIAS REQUIRED:

A. Perchloric acid and Nitric Acid mixture (1:2): Add 500 ml (70%) perchloric acid in 1000 ml HNO₃ (69-71 %), mix well , cool and store in an amber glass bottle.

B. Standard solutions of potassium

■ METHOD:

Digestion

Weigh 1.0 g, ground, sieved and free of contamination plant sample in digestion tube. Add 20 ml acid mixture. Heat at 150-1750C on digestion block till white to slight yellowish plant material color in the digestion tube. Make volume 100 ml with distilled water or as desired for analysis.

Make dilution as desired of the digested material. Determine K (ppm) by flame photometer using standard and graph reading.

■ CALCULATIONS REQUIRED: % K = K ppm x d.f./10000.

■ CAUTIONS / SAFETY REQUIREMENTS: Always run a blank for accurate results.

■ REFERENCE / RELATED DOCUMENTS:

Jones, Jr. J. B., B. Wolf and H. A. Mills. 1991. Micro-Macro Publishing Inc., Athens, GA, USA.



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APPENDIX 1. SUMMARIZED SOIL TEST METHODS AND INTERPRETING FOR SOIL FERTILITY EVALUATION

Parameter Property/ Nutrient(s)	Olsen P	NH₄OAc K, Mg, Na, Ca		DTPA Zn, Cu, Fe, Mn	HCL B
Sample size (g)	2.5	5		10	10
Volume-extractant (mL)	50	25		20	20
Extracting Solution	0.5 M NaHCO ₃ at pH 8.5	1 N NH ₄ Oac pH 7.0		0.005 M DTPA ⁺	HCL
Shake/boil (minutes.)	30	5		0.01 M TEA ⁺	
Shaking action and speed:	All use reciprocating, 180+ oscillations/minutes., except for B				
Extraction method	Colorimetry, at 880nm (Molybdenum blue)	K: Flame emission	K & Na: Flame emission Mg & Ca: AAS	AAS Zn, Cu, Fe, Mn: AAS	Colorimetry, at 450 nm (Azomethine-H)
Soil nutrient conc., no dilution (ppm)	P, 2 – 200	K, 5 – 750;	K, 50 – 1000; Ca, 500 – 2000; Mg, 50 – 500; Na, 10 – 250	Zn, 0.5 – 20	B, 1 – 10
Primary reference	Olsen <i>et al.</i> (1954)	Soltanpour & Schawb (1977)	Schollenberger & Simon (1945)	Lindsay & Norvell (1978)	Berger & Truog (1939)

Source: Soil and Plant Analysis Council (1992). AAS = atomic absorption spectrophotometer.



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APPENDIX 2. INTERPRETING SOIL pH LEVELS AND ASSOCIATED CONDITIONS

<u>Soil pH</u>	<u>Indications</u>	<u>Associated Conditions</u>
< 5.5	Soil is deficient in Ca and /or Mg, and should be limed	Poor crop growth due to low cation exchange capacity and possible Al ³⁺ toxicity. Expect P deficiency.
5.5 – 6.5	Soil is lime-free, should be closely monitored.	Satisfactory for most crops
6.5 – 7.5	Ideal range for crop production.	Soil cation exchange capacity is near 100% base saturation.
7.5 – 8.4	Free lime (CaCO ₃) exists in soil.	Usually excellent filtration and percolation of water due high Ca content on clays. Both P and micronutrients are less available.
>8.4	Invariably indicates sodic soil.	Poor physical conditions. Infiltration and percolation of soil water is slow. Possible root deterioration and organic matter dissolution.

Source: Hach Company, USA (1992).



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APPENDIX 3. GENERALIZED GUIDELINES FOR INTERPRETATION OF SOIL ANALYSIS DATA

Nutrient /Organic Matter	Soil Test	Low	Marginal	Adequate
		----- % -----		
Organic matter	Walkley- Black	<0.86%	0.86–1.29%	>1.29
		----- ppm -----		
Phosphate	NaHCO ₃	<8	8 –15	>15
Potassium	NH ₄ OAc	<100	100-150	>150
Zinc	DTP	<0.5	0.5 –1.0	>1.0
Copper	DTP	<0.2	0.2 –0.5	>0.5
Iron	DTP	<4.5		>4.5
Manganese	DTP	<1.0	1.0 –2.0	>2.0
Boron	HCl	<0.45	0.45 –1.0	>1.0

DTPA= diethylene triamine pentoacetic acid. NaHCO₃ = Sodium bicarbonate.

Sources: FAO (1980); Soltanpour (1985); Ludwick (1995); Martens and Lindsay (1990); Johnson and Fixen (1990); Soil and Plant Analysis Council (1992)



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APPENDIX 4. GENERALIZED GUIDELINES FOR INTERPRETATION OF WATER ANALYSIS DATA

Sr No.	Parameters	Fit	Marginal	Unfit
1.	EC(uS/cm)	<1000	1000-1200	>1200
2.	RSC (meq/l)	<1.25	1.25-2.5	>2.5
3.	SAR	<6	6-10	>10
	Nutrient (ppm)	Low	Normal	High
4.	K	<5	5-20	>30
5.	Ca	<20	20-60	>80
6.	Cl	<4	4-10	>10



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APPENDIX 6. Template (Soil Analysis Report overleaf)

Chemical Analysis

اس کیلئے تمام ضروریات کا معیار یہ ہیں اور ان کے مطابق اس رپورٹ میں نتائج دیئے گئے ہیں۔

کیمیائی	کمیت
نائٹروجن (N)	0.1 سے 0.3
فسفور (P)	0.1 سے 0.3
پوٹاش (K)	0.1 سے 0.3
سولفیر (S)	0.1 سے 0.3

Chemical Analysis

اس کیلئے تمام ضروریات کا معیار یہ ہیں اور ان کے مطابق اس رپورٹ میں نتائج دیئے گئے ہیں۔

کیمیائی	کمیت
نائٹروجن (N)	0.1 سے 0.3
فسفور (P)	0.1 سے 0.3
پوٹاش (K)	0.1 سے 0.3
سولفیر (S)	0.1 سے 0.3

Available Zn

پوٹاش کی کمی کے باعث اس میں پوٹاش کی کمی کا مشاہدہ کیا گیا ہے۔

شمارہ	کمیت
1	0.1 سے 0.3
2	0.1 سے 0.3

Electrical Conductivity

پانی کی موصلیت اس میں پوٹاش کی کمی کا مشاہدہ کیا گیا ہے۔

شمارہ	کمیت
1	0.1 سے 0.3
2	0.1 سے 0.3

Soil Test Results

	Poor	Medium	Fertile		Poor	Medium	Fertile
Organic Matter (%)	< 0.15	0.15-1.25	> 1.25	Cu (mg/kg)	< 0.1	0.1-0.2	> 0.2
Available P (mg/kg)	< 7.0	7-21	> 21.0	Pb (mg/kg)	< 2.0	2.0-4.5	> 4.5
Available K (mg/kg)	< 80	80-180	> 180	Mn (mg/kg)	< 0.5	0.5-1.0	> 1.0
Zn (mg/kg)	< 0.5	0.5-1.0	> 1.0	S (mg/kg)	< 0.2	0.2-0.5	0.5-1.0

کھادوں کی سفارشات

Sr #	Crop	Soil Condition	Fertilizer Recommendation (Kg/acre)			Fertilizer Recommendation (Bags / acra)		
			N	P ₂ O ₅	K ₂ O	Urea	DAP	SOP
1	Wheat	Poor	50	40	25	1.5	2.5	1.0
		Medium	45	35	25	1.5	1.5	1.0
		Fertile	30	20	20	1.1	1.0	1.0
2	Rice (Coarse)	After Wheat	57	30	25	2.0	1.5	1.0
		After Legumes	41	32	25	1.25	1.5	1.0
3	Rice (Fine)	After Wheat	50	41	32	2.5	1.75	1.25
		After Legumes	37	41	32	2.0	1.75	1.25
4	Cotton (BT)	Early Sowing	100-110	45	37.50	3.00-3.25	3.0	1.50-2.0
		Medium / Late Sowing	80-100	45	37	2.0-4.0	1.5	1.5
5	Maize	Poor	50	40	37	3.0	2.5	1.5
		Medium	45	40	35	2.5	2.0	1.0
6	Potato	Poor	100	60	50	4.0	3.5	3.0
		Medium	80	55	50	3.5	3.5	3.0
		Fertile	67	45	47	3.0	3.0	2.5
7	Sugarcane	Poor	120	60	50	6.0	3.0	3.0
		Medium	90	55	50	3.5	3.0	3.0
		Fertile	60	33	35	2.75	1.0	1.0
8	Vegetables	Poor	50	40	35	1.0	1.0	1.0
		Medium	30	34	35	1.0	1.0	1.0
Special Remarks								

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APPENDIX 8. Template (Water Analysis Report overleaf)

1- اصل پذیرشکیات: (TOTAL SOLUBLE SALTS)

اصل پذیرشکیات کا اندازہ برقی سولیت (Electrical Conductivity) کے ذریعے لگایا جاسکتا ہے۔ اس کی اکائی مائیکرو سکنری سنٹی میٹر ہوتی ہے اس کو اصل پذیرشکیات میں تبدیل کرنے کے لئے 0.7 ہے ضرب دے کر حاصل ضرب کو حصے میں لاکھ میں (ppm) دکھایا جاسکتا ہے پانی کی سوزدہیت کی وجہ سے EC کے حساب سے کی جاتی ہے جو حسب ذیل ہے۔

(i) 0 سے 1000 مائیکرو سکنری آب پاشی کے لئے سوزوں ہے اور تمام فصلیں کاشت کی جاسکتی ہیں۔

(ii) 1001 تا 1250 مائیکرو سکنری اس میں خشکیات کی مقدار درمیانی ہوتی ہے اور نہری پانی میں لاکھ آ پاشی کے لئے استعمال کیا جاسکتا ہے اس پانی کے استعمال کے ساتھ درمیانی تک برداشت کرنے والی فصلیں مثلاً گندم، چاول، کئی، جوار، جوی نماثر، آلو، کوکھی، اگا، برمنز، بیاز وغیرہ کاشت کی جاسکتی ہے۔

(iii) 1250 سے زیادہ اس میں خشکیات کی مقدار زیادہ ہے اور آ پاشی کے لئے سوزوں نہیں تاہم یہ پانی زرعی زمینوں یا زیادہ نمک آبی والی زمینوں میں استعمال ہو سکتا ہے اور اس میں زیادہ خشکیات برداشت کرنے والی فصلیں مثلاً کپاس، جود، رایا، بیٹنگر پانک وغیرہ کاشت ہو سکتی ہے۔

2- سوزیم کی جذب سلی نسبت: (SAR)

پانی کی خاصیت سوزیم اور سلیئم + میگنیم سے معلوم کی جاتی ہے سوزیم کی جذب سلی نسبت کی وجہ سے پانی سوزیم میں ہوتا ہے۔

6 سے کم: سوزیم سے نفوز پذیریری (Permeability) کا کوئی مسئلہ نہیں۔

6 تا 10: ہر ایک ہفتہ والی زمین مثلاً چکنی اور چکنی میراجن کی فیصد سیرشدگی (Saturation % age) 50 سے زیادہ ہو سکتی ہے پیری کا مسئلہ پیدا ہو سکتا ہے۔

10 سے زیادہ: ان زمینوں میں جہاں ہامیاتی مادہ کی کمی ہے نفوز پذیریری کا خطرہ ہے لیکن سوزی ہفتہ والی زمینیں (زرعی یا زرعی میرا) جن کی فیصد سیرشدگی 20 سے کم ہو میں کم خطرہ ہوتا ہے۔

3- ذرا سوزیم پانی کا ریسٹ: (RESIDUAL SODIUM BICARBONATE)

ذرا سوزیم پانی کا ریسٹ پذیریری کی کاربائیٹ تفریق اور پانی کا ریسٹ کی حاصل میں سے سلیئم + میگنیم کو منہا کرنے سے حاصل ہوتی ہے اس کی اکائی meq/L ہوتی ہے اور درجہ بندی حسب ذیل ہے۔

1.25 سے کم: پانی آب پاشی کے لئے سوزوں ہے۔

1.25 تا 2.5: وہ فصلیں جن میں خشکیات برداشت کرنے کی درمیانی صلاحیت ہو ان کیلئے پانی آب پاشی کے لئے استعمال ہو سکتا ہے۔

2.5 سے زیادہ: پانی آب پاشی کے لئے سوزوں نہیں ہے۔

4- کلورائیڈ: (CHLORIDE)

آب پاشی کے لئے اس کی مقدار پانی میں 4.5 me/L سے زیادہ نہیں ہونی چاہیے۔

نوٹ: خیال رہے کہ درجہ بندی کے لئے گورنمنٹ کے سوزوں ہونا ضروری ہے۔ لیکن سوزدہیت کا زیادہ تر انحصار زمین کی ہفت پر ہے یا نہری پانی کے ساتھ لگ کر دینے کی صورت میں ان دونوں کا سوزوں کا حساب ہے۔



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APPENDIX 9. PROFORMA FOR COLLECTION OF SOIL & Miscellaneous SAMPLES

Date of sample collection:

Form No.

Sr No	Description	
1.	Name of Farmer	
2.	CNIC No. (optional)	
3.	Telephone No.(If available)	
4.	E-mail ID. (If available)	
5.	Village	
6.	UC	
7.	Tehsil	
8.	Longitude (If available)	
9.	Latitude (If available)	
10.	Square/ Killa No/Any other.	
11.	No. of soil samples	
12.	Depth of soil sample	
13.	No. of Water samples	
14.	Depth of water samples	
15.	No. of plant samples	
16.	Detail of plant samples	
17.	Signature of customer	
18.	Signature of Incharge Sample Receiving Unit (SRU)	



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APPENDIX 10. WATER SAMPLE RECEIPT REGISTER

Sr. No.	Registered No	No. of samples received	Date of Receipt	Name and Address of Farmer	No. of Samples handed over to the analyst	Date of Handing Over	Date of receipt of analysis report from the analyst	Depth of tube well	Amount Received	Dispatch No. & date



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APPENDIX 11. WATER ANALYSIS DATA REGISTER

Name of Farmer	Serial No.	Registration No.	EC (μScm^{-1})	[Ca+Mg] ⁺⁺ (meL^{-1})	Na ⁺ (meL^{-1})	CO ₃ ⁻⁻ (meL^{-1})	HCO ⁻ (meL^{-1})	Cl ⁻ (meL^{-1})	SAR	RSC	Status	Depth of tube well	Sign & Date



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APPENDIX 12. SOIL ANALYSIS DATA REGISTER

Name of Farmer	Serial No.	Registration No.	Depth of Sample	ECe	pH	OM	P	K	SP	Texture	GR (If required)	Sign & Date



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APPENDIX-13 SOIL SAMPLE RECEIPT REGISTER

Serial No.	Lab. Regd. No.	No. of Samples Received	Name and Address of Farmer	Date of Receipt	Sample handed over to the analyst	Date of Handing Over	Date of receipt of Analysis Report from the analyst	Depth of soil sample	Amount Received (Rs.)	Dispatch No. of Report & Date



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APPENDIX-14: TEMPLATE : INTERPRETING WATER ANALYSIS REPORT (URDU)

پانی برائے آبپاشی کی رپورٹ کو سمجھیں!

سوال ایگزٹو ڈائریکٹ لیاہری سرگودھا پانی کا تجربہ کر کے ایک سستی اور جامع رپورٹ فراہم کرتی ہے۔ جس سے فصلات کو لگائے جانے والے پانی کے معیار کو جاننا جاتا ہے۔ پانی کی قیمت کی وجہ سے زمیندار ٹیوب ویل کا پانی لگانے پر مجبور ہو جاتے ہیں۔ ٹیوب ویل، پانی کی کمی تو پوری کر دیتا ہے لیکن پانی کے معیاری ہونے کی کوئی گارنٹی نہیں۔ غیر معیاری پانی کے استعمال سے فصل اور زمین دونوں تباہ ہو سکتے ہیں۔ اس لیے ٹیوب ویل کے پانی کو لگانے سے پہلے اس کے معیار کو جاننا بہت ضروری ہے۔

ناموزوں پانی کیسے نقصان دہ ہوتا ہے؟

ناموزوں پانی کے استعمال سے فصل اور زمین کو درپیشوں سے نقصان ہو سکتا ہے۔ (۱) پانی میں نمکیات جب ایک خاص مقدار سے زائد ہو جاتے ہیں تب فصلات کو اس کا لگا تا نقصان دہ ہو سکتا ہے۔ زائد مل پڑنے پر نمکیات کی وجہ سے پودوں کو زمین سے خوراک اور پانی حاصل کرنے میں مشکل پیش آتی ہے۔ جس کی وجہ سے پودوں کی نشوونما رک جاتی ہے اور وہ مرنے لگتے ہیں۔ (۲) اکثر ناموزوں پانی میں ایسے نمکیات ہوتے ہیں جو زمین میں جا کر اسے سخت کر دیتے ہیں۔ ایسی زمینوں میں نفوذ پڑنے کی صلاحیت کم ہونے سے پانی کم جذب ہوتا ہے۔ ایسی صورت میں پودے ہوا اور پانی دونوں کی کمی سے مرنے لگتے ہیں۔ تجرباتی رپورٹ میں ان دونوں خصوصیات کو معلوم کیا جاتا ہے جس کے معیار کا تعین کیا جاتا ہے۔

مل پڑنے پر نمکیات (EC) :-

سب سے پہلے پانی میں موجود تمام مل پڑنے پر نمکیات کی مقدار کا اندازہ، پانی میں برقی رو کو لگا کر لگایا جاتا ہے۔ پانی میں جتنے زیادہ نمکیات ہوں گے اس کی برقی موصلیت (Electrical Conductivity) (EC) اتنی ہی زیادہ ہوگی۔ مل پڑنے پر نمکیات کی مقدار کے مطابق آبپاشی کے پانی کی درجہ بندی درج ذیل ہے۔

پانی کا معیار	برقی موصلیت (EC)	استعمال / نقصانات
موزوں	1000 سے کم	ہر قسم کی فصلات کے لئے استعمال کیا جا سکتا ہے۔
درمیانہ موزوں	1000 سے 1250 تک	ایسے پانی کو نہری پانی کے ساتھ ملا کر استعمال کیا جا سکتا ہے۔ درمیانہ تک برداشت کرنے والی فصلات مثلاً گندم، چاول، کئی جوار، جوی، لہنا، گھی، گاجر، پیاز، کاشت کی جا سکتی ہیں۔
ناموزوں	1250 سے زیادہ	یہ پانی آبپاشی کیلئے ناموزوں ہے لیکن نمکیات، برداشت کرنے والی فصلات (کیاں، جوار، رایا، بیٹنگر، باگد وغیرہ) ریشمی یا زیادہ لٹاسی آب والی زمینوں میں کاشت کی جا سکتی ہیں۔

سوداگ کی جذب صلاحیت (SAR) :-

مل پڑنے پر نمکیات کی مقدار معلوم کرنے کے بعد ان میں سے ایسے نمکیات کو بھی معلوم کیا جاتا ہے جس سے زمین سخت ہو سکتی ہے۔ زمین کو سخت بنانے میں سب سے اہم کردار سوداگ (Na) ہے۔ جبکہ زمین کو نرم کرنے کیلئے کالشیئم (Ca) کی ضرورت ہوتی ہے۔ یہ دونوں عناصر آبپاشی کے پانی میں موجود ہوتے ہیں اور ان کا تناسب سب سے اہم بات کا تعین کرتا ہے۔ ایسے پانی کے استعمال سے زمین سخت ہوگی یا نہیں۔ اس تناسب کو سوداگ کی جذب صلاحیت (SAR) کہتے ہیں۔ یہ

مقدار جتنی زیادہ ہوگی اتنی ہی زمین میں ایسے پانی کے استعمال سے سختی زیادہ ہوگی۔

پانی کا معیار	SAR	استعمال / نقصانات
موزوں	6 سے کم	نفوذ پڑنے پر پانی کو کوئی خطرہ نہیں۔ ہر قسم کی زمینوں کے لئے استعمال کیا جا سکتا ہے۔
درمیانہ موزوں	6 سے 10 تک	کچھ زمینوں یا باریک بانٹ والی زمینوں میں سختی ہو سکتی ہے۔ ایسے پانی کو نہری پانی کے ساتھ ملا کر استعمال کیا جا سکتا ہے۔
ناموزوں	10 سے زیادہ	یہ پانی آبپاشی کیلئے ناموزوں ہے۔ ریشمی زمینوں کے علاوہ تمام زمینوں میں سختی ہو سکتی ہے۔

زائد سوداگ یا کاربونیٹ (RSC) :-

آبپاشی کے پانی میں سوداگ کے بعد دوسرا اہم جز زمین کو سخت کرنے کا باعث بن سکتا ہے۔ وہ زائد سوداگ یا کاربونیٹ (RSC) ہے۔ جس پانی میں یہ تک زیادہ ہو گا اس کے استعمال سے زمین میں موجود کالشیئم (Ca) بھی ختم ہو جاتا ہے۔ ایسے پانی کے مسلسل استعمال سے زمین سے کالشیئم ختم ہونے کی صورت میں زمین سخت ہوتے جاتے ہیں۔ RSC کی مقدار کے مطابق آبپاشی کے پانی کی درجہ بندی درج ذیل ہے۔

پانی کا معیار	RSC	استعمال / نقصانات
موزوں	1.25 سے کم	نفوذ پڑنے پر پانی کو کوئی خطرہ نہیں۔ ہر قسم کی زمینوں کے لئے استعمال کیا جا سکتا ہے۔
درمیانہ موزوں	1.25 سے 2.5 تک	کچھ زمینوں یا باریک بانٹ والی زمینوں میں سختی ہو سکتی ہے۔ ایسے پانی کو نہری پانی کے ساتھ ملا کر استعمال کیا جا سکتا ہے۔
ناموزوں	2.5 سے زیادہ	یہ پانی آبپاشی کیلئے ناموزوں ہے۔ ریشمی یا زیادہ لٹاسی آب والی زمینوں کے علاوہ تمام زمینوں میں سختی ہو سکتی ہے۔

نوٹ :-

- 1- موزوں پانی کیلئے تینوں خاصیتوں (EC, SAR, RSC) میں سے ہر ایک کا موزوں ہونا ضروری ہے۔ اگر پانی کسی ایک خاصیت میں بھی ناموزوں ہے تو ایسا پانی ناموزوں قرار دیا جائے گا۔
- 2- پانی کی موزونیت کا انحصار زمین کی بانٹ اور کاشت کی جانے والی فصل پر بھی ہوتا ہے۔
- 3- نہری پانی کے ساتھ ملا کر دینے کی صورت میں، نہری اور ٹیوب ویل کے پانی کا تناسب بھی پانی کی موزونیت پر اثر انداز ہوتا ہے۔

پانی کا نمونہ لینے کا طریقہ

ٹیوب ویل کو لگا کر دھاگہ چلا کر ایک صاف بوتل میں ایک لیٹر پانی بھر لیں۔ تجربے کی رپورٹ دوسرے دن دینی جاتی ہے۔ مزید معلومات کیلئے رابطہ: ایگزٹو ڈائریکٹ لیاہری سرگودھا، سلیڈ ان پورٹ سرگودھا

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APPENDIX-15: TEMPLATE: WATER ANALYSIS PROCEEDINGS

WATER ANALYSIS PROCEEDINGS

Page No. 1

Sample Reg. No.	Receipt Date	Farmer Name	T. Well Detail Depth (ft)	Electrical Conductivity ($\mu\text{S cm}^{-1}$)	Calcium + Magnesium	Sodium	Carbonate		Bi-Carbonate	Chloride	Residual Sodium Carbonate	Sodium Adsorption Ratio	Water Quality Category	Report Date with Analyst Sign
					Sample=10 ml		Sample=50 ml							
					$\text{Ca}^{+2} + \text{Mg}^{+2} =$ ml of 0.01N EDTA used	$\text{Na}^+ = \text{TSS} -$ (Ca + Mg)	$R_1 = \text{ml}$ of 0.1N H_2SO_4 used	$\text{CO}_3^{-2} = 4 \times$ R_1	$R_2 = \text{ml}$ of H_2SO_4	$\text{HCO}_3^{-1} =$ $2 \times (R_2 - R_1)$	$\text{Cl}^{-1} = \text{ml}$ of 0.05N AgNO_3	RSC = ($\text{CO}_3 + \text{HCO}_3$) - (Ca + Mg)		

