Chapter 3 Protocol for Measuring Soil Salinity

Abstract A simple protocol is described that tests soil salinity. Water-soluble salts are extracted from soil samples and salt content measured. Accurate field evaluations require sampling at various field locations and various depths and over time take into account the crop species to be grown. Instruments and reagents are listed in preparing soil—water extracts and for measuring salt content. Two methods are provided in measuring salt content, by weight and by electrical conductivity.

3.1 Background

The measurement of soil salt content is very important for plant salt tolerance studies. The most commonly used method is a simple field test. The characteristics of saline soil areas include microtopography, complicated soil types and significant differences in local soil conditions. In order to reduce testing errors caused by differences in local soil conditions, numerous samples are required and repeat sampling needs to be performed. The soil samples should be collected from different soil layers at different depths based on the plant species root growth. For deep-rooted plants, sample soil layers from 0 to 5 cm, 5 to 10 cm, 10 to 20 cm, 20 to 40 cm, 40 to 60 cm and so on are required to a depth of at least 1 m. The samples from different layers should be mixed uniformly. For plants with shallow rooting systems, soil layers should be sampled to a depth of about 60 cm. The salt content in saline soils is dynamic and changes over time and is heterogeneous from location to location. It also changes with the year and the month and even during a day. Considering seasonal and climatic conditions, the sampling times should include spring and summer when salts tend to accumulate and autumn and winter when rains tend to leach salts from the soil. The growing season, time of sowing, seedling establishment, flowering time and harvest should also be considered as some growth stages may be more sensitive to salt damage than others, depending on the crop. Data collected over the years is also useful in assessing trends in salinisation.

Saline soils possess excessive water-soluble salts. Measuring water-soluble salts has two main steps: (1) the preparation of the sample solution according to the

specific water/soil ratio and (2) the analysis of the soil salt concentration and ionic components in the soil sample. In general, studies on dynamic changes of water and salt content in the soil use a water/soil ratio of 5:1, whereas a water/soil ratio of 1:1 is generally used for the analysis of alkaline soils. The method of saturated soil extract is rarely used because the execution of this method is tedious, and it is difficult to determine the correct saturation point. The sample solution in the following tests refers to 5:1 water/soil extract.

3.2 Instruments and Reagents

Instruments: reciprocating shaker, 1/100 balance, Buchner funnel, vacuum pump, centrifuge (4000 r/min), gas extraction bottle.

Reagent: 0.1 % NaPO₃.

3.3 Preparation of 5:1 Water/Soil Extract

Weigh 100 g air-dried soil sampled from the field that passes through a 1 mm sieve. Put the soil sample in an Erlenmeyer flask. Add 500 ml $\rm CO_2$ -free distilled water (based on a water/soil ratio of 5:1). Seal the flask mouth with a rubber stopper and place the Erlenmeyer flask in a reciprocating shaker and shake for 3 min. Immediately, after shaking, perform an air pump filtration with a Buchner funnel. Collect the clear liquid in a 500 ml Erlenmeyer flask. Add 1 drop of 0.1 % NaPO $_3$ for every 25 ml.

3.4 Preparation of 1:1 Water/Soil Extract

Weigh an air-dried soil sample that passes through a 1 mm sieve. Put the soil sample in an Erlenmeyer flask. Add CO₂-free distilled water based on a water/soil ratio of 1:1. The rest of the operation is the same as above.

3.5 Important Considerations

When extracting with the 5:1 water/soil ratio, hygroscopic water of the air-dried soil can be ignored due to the high percentage of water. When extracting with the 1:1 water/soil ratio, hygroscopic water of the air-dried soil must be corrected to avoid test error (compared to the 5:1 water/soil ratio, hygroscopic water in the

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soil may affect the water/soil ratio at 1:1; this needs to be corrected to avoid test errors, and therefore the use of completely air-dried soil is recommended).

- In the process of extraction of water-soluble salts in soil, 3 min shaking is sufficient for the water-soluble chlorides, carbonates and sulphates to dissolve in the water. With an extended shaking time or standing time, the neutral salts and water-insoluble salts will also enter the extract and cause greater errors.
- Both the partial pressure of CO₂ in the air and the dissolved CO₂ in the distilled water will affect the solubility of some salts including CaCO₃, CaSO₄ and MgSO₄. As a result, the salt content in the extract will be affected. Therefore, CO₂-free distilled water must be used in the extraction.
- The standing time of the sample solution should not exceed 1 day.
- Adding a small amount of NaPO₃ solution in the soil extract can prevent the formation of a CaCO₃ precipitate when standing. Although NaPO₃ will slightly increase the Na⁺ concentration in the extract, the error caused by NaPO₃ is much smaller than the error caused by CaCO₃ precipitate.

3.6 Measurements

The main methods of measuring total water-soluble salts in a soil sample are the (1) weight method and (2) conductivity method. The data obtained from the weight method are reliable, but the operation is tedious and time-consuming. The conductivity method is simple.

3.6.1 Weight Method

This method is based on a water extract from a soil sample. The extract is evaporated to dryness and then dried at 105-110 °C to constant weight. The total dried residue contains both water-soluble salts and water-soluble organic matter. H_2O_2 is used to remove the organic matter in the residue. What remains are the total water-soluble salts from the soil.

3.6.1.1 Instruments and Reagents

Instruments: evaporating dish, water bath, dryer, electrothermal drying oven, analytical balance.

Reagents: $15 \% \text{ H}_2\text{O}_2$ and $2 \% \text{ Na}_2\text{CO}_3$.

3.6.1.2 Method

Draw 50.0 ml of solution from a soil sample of known weight (w), place in an evaporating dish and weigh (w_0) . Evaporate to dryness in a water bath and then dry in an electrothermal drying oven at $105-110\,^{\circ}\text{C}$ for 4 h. Remove from the oven and place in a dryer for 30 min, then weigh using an analytical balance. Return sample to the electrothermal drying oven for 2 more hours, cool down and reweigh. Repeat these steps until a constant weight (w_1) is obtained; the weight difference between the two times should not be more than 1 mg. Calculate the weight of the dried residue.

Add 15 % H_2O_2 in drops to wet the residue. Evaporate to dryness in a water bath. Repeat this treatment until the entire residue turns white. Dry the white residue to constant weight (w_2) according to the method described above. Calculate the content of the total water-soluble salts in the soil.

3.6.1.3 Calculation of Total Water-Soluble Salts

Total dried residue =
$$(w_1 - w_0)/w \times 100 \%$$

where w is the weight of the soil sample (g) that the drawn extract is equivalent to.

3.6.1.4 Important Considerations

- The volume of the soil extract to draw is determined by the soil salt content. When the soil salt content is higher than 0.5 %, draw 25 ml; when the soil content is lower than 0.5 %, draw 50 or 100 ml. Make sure that the measured total salt content is 0.02–0.2 g.
- If the residue has high contents of CaSO₄·2H₂O and MgSO₄·7H₂O, drying at 105–110 °C cannot completely remove the water of crystallisation in these hydrated salts. As a result, the constant weight is difficult to obtain. In such cases, the drying temperature should be increased to 180 °C.
- If there are high amounts of $CaCl_2 \cdot 6H_2O$ and $MgCl_2 \cdot 6H_2O$ in the soil, it is difficult to get a satisfactory result even with drying at 180 °C as these salts are very hydroscopic (readily absorbed water). In such cases, first add 10 ml of 2 % Na_2CO_3 . This will generate NaCl, Na_2SO_4 and $MgCO_3$ salts when evaporating to dryness. The amount of Na_2CO_3 added should then be deducted from the result of the total salt calculation.
- Since many salts absorb water from the air, the conditions for cooling and weighing should be the same.
- When using H₂O₂ to remove the organic matter, the residue only needs to be wet.
 Too much H₂O₂ will generate excessive foam as H₂O₂ decomposes the organic

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matter. This may cause splashing and loss of salt. Repeated treatments with small amounts of H_2O_2 are recommended.

3.6.2 Conductivity Method

Water-soluble salts in the soil act as strong electrolytes. As a consequence, the soil solution has conductivity that can be measured. The electrical conductivity reflects the conductive capacity of the soil solution, and within a certain concentration range, the salt content in the soil is positively related to the electrical conductivity. But it cannot reflect the components of the mixed salt composition. If the ratios of the different salts in the soil solution are relatively constant, the salt concentration determined by electrical conductivity is very accurate. The conductivity method is a rapid and accurate method to measure soil salt content. The present tendency is to use the electrical conductivity to represent the total salt content in the soil directly. The SI unit of electrical conductivity is siemens per metre (S/m).

3.6.2.1 Instruments

Conductivity meter, thermometer ranging from 1 to 60 °C.

3.6.2.2 Method

Draw 20–30 ml of sample solution from a known amount of soil and place in a beaker. Adjust the conductivity meter according to the user's manual. Read the value of the electrical conductivity (mS) after the pointer is stable. Measure the temperature of the sample solution every 10 min.

3.6.2.3 Calculation

The electrical conductivity of the soil extract at 25 $^{\circ}$ C (EC₂₅) is used to reflect the soil salt content. It is calculated as follows:

$$EC_{25} = EC_t \times ft$$

where EC₂₅ is the electrical conductivity of the soil extract at 25 °C, EC_t is the measured electrical conductivity of the soil extract at t °C and ft is the corrected value of electrical conductivity at t (see Table 3.1).

In addition, when the temperature of the soil extract is 17–35 °C, the electrical conductivity of the soil extract increases or decreases about 2 % for every 1 °C in the difference of the soil extract temperature and the standard temperature (25 °C).

Table 3.1 Corrected values of electrical conductivity at different testing temperatures

| Temperature (°C) | Corrected value |
|------------------|-----------------|------------------|-----------------|------------------|-----------------|------------------|-----------------|
| 3.0 | 1.709 | 20.0 | 1.112 | 25.0 | 1.000 | 30.0 | 0.907 |
| 4.0 | 1.660 | 20.2 | 1.107 | 25.2 | 966.0 | 30.2 | 0.904 |
| 5.0 | 1.613 | 20.4 | 1.102 | 25.4 | 0.992 | 30.4 | 0.901 |
| 6.0 | 1.569 | 20.6 | 1.097 | 25.6 | 886.0 | 30.6 | 0.897 |
| 7.0 | 1.528 | 20.8 | 1.092 | 25.8 | 0.983 | 30.8 | 0.894 |
| 8.0 | 1.488 | 21.0 | 1.087 | 26.0 | 9760 | 31.0 | 0.890 |
| 9.0 | 1.448 | 21.2 | 1.082 | 26.2 | 0.975 | 31.2 | 0.887 |
| 10.0 | 1.411 | 21.4 | 1.078 | 26.4 | 0.971 | 31.4 | 0.884 |
| 11.0 | 1.375 | 21.6 | 1.073 | 26.6 | 1960 | 31.6 | 0.880 |
| 12.0 | 1.341 | 21.8 | 1.068 | 26.8 | 0.964 | 31.8 | 0.877 |
| 13.0 | 1.309 | 22.0 | 1.064 | 27.0 | 096.0 | 32.0 | 0.873 |
| 14.0 | 1.277 | 22.2 | 1.060 | 27.2 | 0.956 | 32.2 | 0.870 |
| 15.0 | 1.247 | 22.4 | 1.055 | 27.4 | 0.953 | 32.4 | 0.867 |
| 16.0 | 1.218 | 22.6 | 1.051 | 27.6 | 0.950 | 32.6 | 0.864 |
| 17.0 | 1.189 | 22.8 | 1.047 | 27.8 | 0.947 | 32.8 | 0.861 |
| 18.0 | 1.163 | 23.0 | 1.043 | 28.0 | 0.943 | 33.0 | 0.858 |
| 18.2 | 1.157 | 23.2 | 1.038 | 28.2 | 0.940 | 34.0 | 0.843 |
| 18.4 | 1.152 | 23.4 | 1.034 | 28.4 | 0.936 | 35.0 | 0.829 |
| 18.6 | 1.147 | 23.6 | 1.029 | 28.6 | 0.932 | 36.0 | 0.815 |
| 18.8 | 1.142 | 23.8 | 1.025 | 28.8 | 0.929 | 37.0 | 0.801 |
| 19.0 | 1.136 | 24.0 | 1.020 | 29.0 | 0.925 | 38.0 | 0.788 |
| 19.2 | 1.131 | 24.2 | 1.016 | 29.2 | 0.921 | 39.0 | 0.775 |
| 19.4 | 1.127 | 24.4 | 1.012 | 29.4 | 0.918 | 40.0 | 0.763 |
| 19.6 | 1.122 | 24.6 | 1.008 | 29.6 | 0.914 | 41.0 | 0.750 |
| 19.8 | 1.117 | 24.8 | 1.004 | 29.8 | 0.911 | | |
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The electrical conductivity of the soil extract at 25 °C can also be calculated according to the following formula when the soil extract temperature is 17–35 °C:

$$EC_{25} = EC_t \times [1 - (t - 25) \times 2\%]$$

where EC₂₅ is the electrical conductivity of the soil extract at 25 °C, EC_t is the measured electrical conductivity of the soil extract at t °C and t is the temperature of the soil extract (°C).

3.6.2.4 Important Considerations

- The measuring time for each sample should be relatively constant after the electrodes are inserted into the solution.
- The solutions used for conductivity measurements should be clear. Do not use liquid suspensions as these will damage the platinum back layer on the platinum electrode and cause test errors.
- Solutions with high conductivity should be diluted before taking measurements. Highly concentrated solutions will polarise the electrode and will decrease the sensitivity of the instrument.