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Soil quality — Determination of redox potential — Field method

Qualité du sol — Détermination du potentiel d'oxydoréduction — Méthode de terrain

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Page

Contents

Forew	vord	iv
Introduction		v
1	Scope	1
2	Normative references	1
3	Terms and definitions	1
4	Principle	1
5	Apparatus	
6	Reagents	
7	Site selection and sampling	
8	Procedure8.1Care, cleaning and testing of the redox electrode system8.2Preparation of site and measurement of redox potential	3
9	Evaluation	
10	Expression of results	
11	Test report	5
Annex	A (informative) Description of the construction of redox electrodes, the salt bridge, and their arrangement during measurement	6
Annex	B (informative) Potentials of platinum electrodes in different solutions	10
Annex C (informative) Potentials of reference electrodes		11
	x D (informative) Soil moisture status 11271	
Biblio	s://styndards.iteh.ai/catalog/standards/sist/19729597-d486-401b-a7cb-a4b383e0a3c7/iso- graphy	13

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 190, *Soil quality*, Subcommittee SC 3, *Chemical and physical characterization*.

This second edition cancels and replaces the first edition (ISO 11271:2002), of which it constitutes a minor revision. The changes are as follows:

- update of normative references;
- update of bibliography;
- editorially revised.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at <u>www.iso.org/members.html</u>.

Introduction

The redox potential is a physico-chemical parameter used to characterize soil aeration status in a global way. Under field conditions, it gives information on the condition of oxidation or reduction of those compounds which, according to cases, play an important part in plant nutrition, can induce toxicity phenomena or intervene in gas transfers to the atmosphere (greenhouse effect). It can also be used to a certain extent to follow soil performances in case of sludge disposal or composting, and to adjust applications accordingly. Under laboratory conditions it can be used in order to study oxygen diffusion phenomena to aggregate level.

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Soil quality — Determination of redox potential — Field method

1 Scope

This document specifies a field method for the determination of soil redox potential $(E_{\rm h})$.

NOTE The electrochemical measurement of redox potential described in this document is possible only if the relevant soil horizon has a moisture status defined as fresh or wetter according to the classes presented in <u>Annex D</u>.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1

redox potential (E_b) ai/catalog/standards/sist/19729597-d486-401b-a7cb-a4b383e0a3c7/iso-

electrochemical potential reflecting the oxidation-reduction status of a liquid chemical system (in this case of the soil solution)

4 Principle

Redox potential is an electrochemical equivalent of the free energy of redox reactions, and for an equilibrated single redox system of the general form:

$$A_{\rm ox} + n e^- + m H^+ \ge A_{\rm red} \tag{1}$$

is given by the Nernst equation:

$$E_h = E^0 + \frac{RT}{nF} \ln \frac{A_{\text{ox}}}{A_{\text{Red}}} - \frac{2,303 \, mRT}{nF} \, pH \tag{2}$$

where

 A_{ox} and A_{Red} are the activities of the oxidized and reduced forms of the element, respectively;

- e⁻ refers to the electron(s) involved in the reaction;
- H⁺ refers to the proton(s) involved in the reaction;

n and *m* are the numbers of electrons and protons involved in the reaction, respectively;

- E^0 is the standard value of the potential in volt (V) i.e. when $A_{ox} = A_{Red}$ and pH = 0;
- *R* is the universal gas constant (8,314 1 J mol⁻¹ \cdot K⁻¹);
- *T* is the absolute temperature in kelvin (K);
- F is the Faraday constant (96 500 C \cdot mol⁻¹);
- 2,303 is the natural log of 10.

Redox potential is related to electron activity (e⁻) in the system as follows:

$$E_{\rm h} = \frac{RT}{F} \ln\left(e^{-}\right) \tag{3}$$

Users of this document unfamiliar with these electrochemical concepts should consult appropriate texts or seek professional advice.

The electrometric determination of redox potential is analogous to the determination of pH. The determination of E_h follows the principle of measuring potential differences between an inert measuring electrode (usually a platinum electrode) i.e. an electrode not reacting with the solution per se, with respect to the standard hydrogen electrode used as the reference electrode. Many redox systems are involved in the soil solution, and the resulting potential is a mixed potential depending on the existing electroactive redox couples. For practical reasons, a silver-silver chloride electrode is usually used as the reference electrode, the potential of which is added to the measured potential difference (see Annex C) in order to obtain the values expressed on the basis of the standard hydrogen electrode.

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5 Apparatus

The following apparatus shall be used.

<u>ISO 11271</u>

- https://standards.itch.ai/catalog/standards/sist/19729597-d486-401b-a7cb-a4b383e0a3c7/iso-5.1 Millivoltmeter, with an input resistance not less than 10 GΩ and sensitivity of 1 mV.
- **5.2** A set of redox-electrodes, constructed as described in A.1.

All electrodes should be sufficiently robust for field use (see <u>Annex A</u>).

5.3 Reference electrode: silver/silver chloride reference electrode in 1 mol/l or 3 mol/l potassium chloride solution.

Other reference electrodes such as the calomel electrode can also be used but are not recommended because of the health hazard connected with the use of mercury. The potential of such reference electrodes with respect to the standard hydrogen electrode is given in <u>Annex B</u>. Reference electrodes should be stored in a potassium chloride solution (see <u>6.4</u>) of the same concentration as that present in the electrode, or directly in the salt bridge (see <u>5.5</u>) containing the same concentration of potassium chloride. It should be noted that lower concentrations of potassium chloride will reduce contamination of the soil with this salt.

5.4 Rigid rod, (stainless steel has been found suitable), 20 cm to 100 cm in length with the diameter 2 mm greater than that of the redox electrodes (see <u>5.2</u>).

The rod shall have a length which allows the redox electrodes to be inserted to the desired depth in the soil.

- **5.5 Salt bridge,** to connect the reference electrode with the soil (see <u>A.2</u>).
- **5.6 Hand auger,** with a diameter 3 mm to 5 mm greater than that of the salt bridge.

5.7 Electrode cleaning materials: finest grade of steel wool, scouring powder and some cotton cloth have been found suitable.

5.8 Thermometer, to measure the temperature at the location of the reference electrode (see <u>Clause 8</u>) with an accuracy of 1 °C.

6 Reagents

6.1 Redox buffer solution, to calibrate the redox electrodes.

Use either buffered quinhydrone solution (prepared by adding quinhydrone to a pH buffer to obtain a suspension), or an equimolar solution of potassium hexacyanoferrate(III) and potassium hexacyanoferrate(III) (see <u>Annex B</u>).

6.2 Water, with an electrical conductivity less than 0,1 mS m⁻¹ (equivalent to resistivity greater than 0,01 M Ω m at 25 °C).

6.3 Agar, $\rho = 0.5$ %, in a potassium chloride solution of the same concentration as that in the reference electrode.

6.4 Potassium chloride solution, of the same concentration as that chosen in <u>5.3</u>.

NOTE This solution is used to store the reference electrode and to add to the salt bridge, as needed.

7 Site selection and sampling dards.iteh.ai)

The selection and description of the place of measurement, and of samples for laboratory measurement, should follow the guidelines given in the ISO 18400 series and ISO 11464.

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8 Procedure

112/1

8.1 Care, cleaning and testing of the redox electrode system

The platinum electrodes shall be stored in air and kept clean. They shall be inspected for damage and/or contamination at intervals of not more than one year, and every time they are used. Oils, fats and waxes, and other chemicals likely to adhere are particularly damaging to electrode performance. If contaminated with soil material, they shall be cleaned gently with a cotton cloth and rinsed with distilled water. In cases of severe contamination, e.g. with oils, an appropriate solvent followed by scouring material (see 5.7) shall be used.

The reference electrode(s) shall also be inspected at the same time as the platinum electrode(s). Any visible change in the colour or transparency of the potassium chloride electrolyte solution indicates probable trouble. In such cases the reference electrode shall not be used. Crystals of potassium chloride are, however, a normal feature of saturated potassium chloride solutions and are no reason to reject the electrode in question.

The electrodes shall be checked prior to each series of measurements, by determining their response in the redox buffer solution (see <u>6.1</u>). The measured redox potential values shall be as given in <u>Annex B</u>. Any electrode differing by more than 10 mV from the required value shall be cleaned and tested again, and discarded if cleaning fails to correct the problem.

The use of strong oxidising agents e.g. nitric acid, hydrogen peroxide solution, as cleaning agents for platinum electrodes can result in high redox potentials. The use of such agents is, therefore, not recommended.

In practical terms, the reference electrode potentials can be checked against each other. An electrode which gives a reading differing by more than 10 mV from the other(s) is likely to be faulty and should be discarded. This check procedure requires a minimum of 3 reference electrodes, connected in turn.

Reference electrodes can be checked in absolute terms only against a standard hydrogen electrode. This can be done only in specialized laboratories. It is usually more convenient to buy electrodes only from a reputable source.

8.2 Preparation of site and measurement of redox potential

At the place of measurement, drive a hole into the soil using the rigid rod (see <u>5.4</u>) to a depth 2 cm to 3 cm less than the desired measurement depth. Immediately insert the redox electrode into the hole to a depth deeper by 2 cm to 3 cm than that of the hole. At least two electrodes should be installed for each measurement depth. Leave the platinum electrodes in the soil for at least 30 min following the installation before connecting them to the millivoltmeter.

At a distance of 10 cm to 100 cm from the redox electrode, auger a hole down to a fresh (or wetter) soil layer (see Annex D) and install the salt bridge so as to obtain good contact between the ceramic cup of the salt bridge and the soil. Measure the potential difference, $E_{\rm m}$, in millivolts between the platinum electrode and the reference electrode after 1 h, using the millivoltmeter (see 5.1). The measurement period can be shorter (but not <30 min) only if the difference between successive measurements at 10 min intervals is ≤ 2 mV. Measure the temperature (see 5.8) at the location of the reference electrode at the time of measurement of the potential difference, $E_{\rm m}$.

It is recommended that the platinum electrode be disconnected from the millivoltmeter between readings. Potassium chloride can leak from the salt bridge and reach significant amounts after about 2 h. If this is undesirable, the salt bridge should be removed from the soil and re-installed prior to each set of measurements.

Protect the reference electrode from direct heating by the sun. The temperature of the reference electrode and not that of the soil, should be taken into account when deriving the reference electrode potential. https://standards.teh.ai/catalog/standards/sist/19729597-d486-401b-a7cb-a4b383e0a3c7/so-

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9 Evaluation

The measured voltage, $E_{\rm m}$, shall be related to the voltage of the standard hydrogen electrode by adding the potential of the reference electrode, $E_{\rm r}$, (see <u>Annex C</u>) to the reading $E_{\rm m}$ [see <u>Formula (4)</u>]. The voltage obtained is indicated as the redox potential, $E_{\rm h}$.

$$E_{\rm h} = E_m + E_{\rm r}$$

NOTE The redox potential in soils ranges from -400 mV (extremely reduced conditions) to +900 mV (highly oxidized conditions).

10 Expression of results

The value of the redox potential shall be quoted rounded to the nearest 10 mV.

a) Example of calculation:

Measurement of $E_{\rm m}$: +327 mV, measured against a silver/silver chloride electrode in 1 mol potassium chloride solution. The temperature of the location of the reference electrode at the time of measurement is 17 °C, which means its potential with respect to the standard hydrogen electrode is +241 mV (see Annex C). Thus:

 $E_{\rm h}$ = + 327 mV + 241 mV = + 568 mV

b) Example of presentation of results:

(4)

Redox potential ($E_{\rm h}$) = 570 mV.

11 Test report

The test report shall include the following information:

- a) a reference to this document, i.e. ISO 11271:20—;
- b) a complete identification of the test site, including the depth of measurement and the soil moisture status (see Table D.1);
- c) the result of the determination;
- d) any details not specified in this document, which are optional, as well as any factor(s) which can have affected the results.

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<u>ISO 11271</u>

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