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World Soil Information Service (WoSIS) – Procedures for standardizing soil analytical method descriptions

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Contents

Contents	3
Summary	5
1. Introduction	6
2. Quality aspects related to laboratory data	7
2.1 Context	7
2.2 Laboratory error	7
2.3 Standardization of soil analytical method descriptions	9
3. Criteria for standardizing soil analytical method descriptions	12
3.1 Background	12
3.2 Guiding principles	13
3.3 Coding of method options.....	13
3.4 Example of coding procedure	15
4. Description of method options for soil properties standardized in WoSIS	16
4.1 Bulk density	16
Background	16
Method.....	16
4.2 Calcium carbonate equivalent	17
Background	17
Method.....	17
4.3 Cation exchange capacity.....	18
Background	18
Method.....	18
4.4 Coarse fragments.....	21
Background	21
Method.....	21
4.5 Electrical conductivity.....	22
Background	22
Method.....	22
4.6 Organic carbon.....	22
Background	23
Method.....	23
4.7 Organic matter	24
Background	24
Method.....	24
4.8 Soil pH	25
Background	25
Method.....	25
4.9 Soil texture	26
Background	26
Method.....	27
4.10 Total carbon	29
Background	29
Method.....	29
4.11 Total Nitrogen	30
Background	30
Method.....	30
4.12 Phosphorus	31
Background	31
Method.....	31
4.13 Water retention	32
Background	32

Method.....	33
5. Concluding remarks.....	35
Acknowledgements	35
Appendix – Examples of option tables for describing soil analytical methods.....	36
Bibliography.....	40

List of Tables

Table 1. List of soil properties with standardized analytical method descriptions.	12
Table 2. Procedure for coding standardized analytical methods using pH as an example.	15
Table 3. Procedure for coding cation exchange capacity (Example).	36
Table 4. Procedure for coding Electrical conductivity (Example).	38

List of Figures

Figure 1. Particle size limits used in European countries, Australia and the United States of America.	14
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Summary

This report describes general principles for standardizing soil properties/observations that are served from WoSIS (WorldSoil Information Service) as '[wosis_latest](#)'. Data for nominally similar soil properties, collated from disparate sources, are grouped according to major features of the corresponding analytical methods; that is, the properties are grouped according to 'operational definitions'. For example, major features for defining soil pH, for example, are the sample pretreatment, extractant solution (water or salt solution), and in case of salt solutions the salt concentration (molarity), as well as the soil/solution ratio. A further descriptive element may be the type of instrument used for the actual laboratory measurement.

Currently, the following list of chemical and physical properties is standardized:

- a) Chemical properties: organic carbon, total carbon, total carbonate equivalent, total nitrogen, phosphorus (extractable P, total P and P retention), soil pH, cation exchange capacity, and electrical conductivity.
- b) Physical properties: soil texture (sand, silt, and clay), bulk density, coarse fragments, and water retention.

Full data harmonization in a global context, the ultimate objective of WoSIS, will first become feasible once results of extensive proficiency testing programmes become freely available and a common set of 'international' reference methods has been accepted as the common standard by the international soil community; such would provide the basis for method validation between laboratories with their actual/adjusted method.

1. Introduction

The World Soil Information Service, or WoSIS in short, is a server database for handling and managing multiple soil datasets in an integrated manner, subsequent to proper data screening and cleansing, standardization, and ultimately full harmonization. A key element is that the system allows for inclusion of soil data shared by third parties, while keeping track of the data lineage (provenance) and possible restrictions for use (licenses). The terms of these licenses will determine which set of quality-assessed and standardized data (known as ‘*wosis_latest*’¹) can be served to the international community. These data in combination with spatial environmental co-variate layers can then be used, for example, to generate soil property maps using digital soil mapping (e.g., Poggio *et al.* 2021; Turek *et al.* 2023). Subsequently, these maps can be used to address global challenges such as climate change, food security, and the degradation of land and water resources at continental and (sub)regional scale.

This reports builds upon Appendix C and D of the preceding procedures manual (Ribeiro *et al.* 2020). Section 2 focusses on quality aspects related to soil laboratory data. Section 3 provides criteria for standardizing disparate soil analytical method descriptions (as described in the corresponding standard operation procedures). Section 4 presents the corresponding method options as implemented in the new WoSIS data model. Concluding remarks on the way forward are made in Section 5. The Appendix gives examples of method option tables for CEC and electrical conductivity as examples.

¹ https://data.isric.org/geonetwork/srv/eng/catalog.search#/search?any=wosis_latest

2. Quality aspects related to laboratory data

2.1 Context

WoSIS is being populated using data produced for diverse types of studies; the corresponding data were sampled and analyzed in a range of laboratories according to a wide range of methods. By implication, the quality of the standardized/harmonized data in WoSIS will be determined by the quality of all preceding steps of data processing. Typically, a quality management system comprises measures necessary to arrive at a predefined and constant quality at agreed costs (based on user specific requirements for use). For instance, (certified) laboratories develop/use protocols for each sub-process, use validated methods for laboratory investigations, and participate in round robin tests to monitor their performance over time with respect to certified or consensus reference materials (van Reeuwijk and Houba 1998; WHO 2011; US-EPA 2015; GLOSOLAN 2023).

ISRIC, for example, published reference methods for soil analysis as a step towards standardization of analytical methods in soil laboratories (van Reeuwijk 2002). These methods cover the range of analytical methods required for soil characterization according to the Revised FAO Legend (FAO 1988) and the World Reference Base (IUSS Working Group WRB 2022). Similarly, the Natural Resources Conservation Service of the United States Department of Agriculture publishes a Soil Survey Laboratory Methods Manual (Soil Survey Staff 2022), which is the reference source for the National Cooperative Soil Survey Soil Characterization Database (USDA-NCSS 2021) and widely referred to internationally as reference. Alternatively, Australia has its own set of reference laboratory tests (Rayment and Lyons 2011) and, for example, uses different particle size class limits for defining the 'silt' fraction. More recently, within the framework of the Global Soil Partnership (GSP), GLOSOLAN (2022) are developing a new set of standard operation procedures for worldwide application that may provide the basis for future data harmonisation.

Although adoption of uniform reference methods at different laboratories contributes to a common quality level, it does not rule out that the quality of individual data held in compiled datasets such as WoSIS, which draws on a wide range of source data, may differ in quality as discussed below.

2.2 Laboratory error

Important quality characteristics for any measured data are the random and systematic error (Magnusson and Örnemark 2014). Random errors in experimental measurements are caused by unknown and unpredictable changes in the experiment; such changes may occur in the measuring instruments or in the environmental conditions. Systematic errors in experimental observations usually come from the measuring instruments themselves. Both error components will contribute to a varying extent to the total error. In practice, however, in reports and publications these essential laboratory error characteristics generally are not presented along with the actual data

produced. In such cases, error characteristics can only be extracted afterwards from quality management systems or estimated in special experimental designs. Laboratories participating in inter-laboratory studies such as ring tests or round robin tests receive feedback on their quality performance with the particular methods by comparing their results with those from other participants. Examples are WEPAL (2022), the Wageningen Evaluating Programme for Analytical Laboratories, the Australasia inter-laboratory proficiency programme (ASPAC) (Rayment and Peverill 2002), the North American Proficiency Testing Programme (NATP 2015) and GLOSOLAN (2023). These programmes often are certified according to ISO/IEC 17043. However, they do not consider the influence of differences in sampling procedure and pretreatment at individual laboratories as these programmes use pretreated and homogenized materials. Further, the reference materials need to be relevant/representative for the soil types analyzed at a given laboratory. Ross *et al.* (2015), for example, in studying the inter-laboratory variation in the chemical analysis of acidic forest soil reference samples from eastern North America, stressed the importance of using sample materials representative for the (types of) samples in the batches processed. When a new, or revised, analytical method is introduced, laboratories should do a validation study to compare the quality performance with other (similar) methods, previous versions of the method and materials with reference and consensus results (e.g., Bispo *et al.* 2021; ICP Forests 2021).

An extended guide to the validation of methods, consistent with international standards such as ISO/IEC 17025, is given by EURACHEM (2014). It includes validation and verification methods as well as a number of performance characteristics including random and systematic error, limits of detection, and limit of quantification. For laboratory methods, the latter two characteristics are used to indicate the limit below which the detection of an analyte becomes problematic, respectively the lowest level of analyte that can be determined with acceptable performance. Unfortunately, many laboratories do not include these measures in their quality statements with the data they distribute even though detailed validation reports may be available. These aspects complicate the processing of soil information obtained from different data providers in databases such as WoSIS, hence sometimes necessitating adoption of pragmatic solutions when processing the source data.

Adequate quality management in a laboratory is a prerequisite for reliable results and ‘data fit for intended use.’ However, it should be noted that the contribution of laboratory error is not necessarily the major component of the total error in derived interpretations; spatial variability can contribute even more (Goodchild and Gopal 1989; Goodchild 1994; Heuvelink 2014; Wadoux and Heuvelink 2023). An indication for the presence of other error sources can be found in the difference between the nugget in a variogram and the smaller values for lab error from validation and comparable experiments (Heuvelink 1996).

While cost-efficient and cost-effective procedures for field sampling are often well described (De Grijter *et al.* 2006; Louis *et al.* 2014), less attention is paid to quality requirements for laboratory investigations. They are often copied from previous and similar studies by applying the same methods. If for practical reasons alternative methods have to be selected, it should be

remembered that numerous soil properties are based on ‘operational definitions’ (Soil Survey Staff 2022) and may apply only for specific user groups. That is, the property is best described by the details of the applied laboratory method. An example is the ‘pH of the soil,’ which needs information on sample pretreatment, soil/solution ratio and description of solution (e.g., water, KCl 1M) to be fully understood. Similarly, in WoSIS soil properties are defined by the analytical methods and the terminology used, based on customary practice in soil science. As noted before, if highest laboratory accuracy is important it should be included in the selection criteria as well.

Two other examples where the description of soil analytical methods is particularly important for selection of alternative methods are cation exchange capacity (CEC) and available Phosphorus. The capacity of a soil to adsorb and exchange cations from exchange sites depends importantly on the actual pH and the ionic strength of the solution. However, the need for a sufficiently detailed description of analytical methods is particularly reflected in the case of so-called plant ‘available phosphate’, where the choice of the appropriate laboratory methods is largely determined by soil pH as a proxy for soil mineralogy and soil type (Elrashidi 2010). Hence, ‘vague’ descriptions for available-P methods are essentially useless, unless used in a specific context such as a (local) fertilizer recommendation scheme. Correlation studies have shown that only in specific cases (i.e., soils and intended use) useful region-specific conversions can be made for available-P values determined according to different analytical methods, such as P-Olsen, P-Bray, modified P-Morgan and Mehlich III (Mallarino 1995; Grandt *et al.* 2010; Breure *et al.* 2022), making international harmonization of results of such methods cumbersome or possibly at best ‘broad brush’. Examples of such efforts include the work of Ciampalini *et al.* (2013) in Tunisia and those of Maire *et al.* (2015) at the global level.

According to GlobalSoilMap (2015) there is generally no universal equation for converting from one analytical method to another in all situations. Within the framework of the Global Soil Partnership (Baritz *et al.* 2014), for example, this would imply that each regional node would need to develop and apply node-specific conversions (towards the adopted standard methods and soil types), building on comparative analyses of say archived samples (Jankauskas *et al.* 2006; Sadoski and Ivanova 2020; Hu *et al.* 2021). The ongoing work of the Global Soil Laboratory Network (GLOSOLAN), established by the Global Soil Partnership in 2017, deserves particular attention in this respect (see Batjes 2023).

2.3 Standardization of soil analytical method descriptions

Lacking detailed quantitative information on the quality of the soil analytical data held in the diverse source databases shared for use in WoSIS, it was necessary to develop a qualitative way to describe the analytical methods in a flexible, yet comprehensive and consistent way. For all source data, as indicated earlier, it is assumed that the quality requirements of the (first) user are met, and basic quality checks and screening have taken place and soil-relevant options in the method are applied in the source laboratory. This allows users of WoSIS to make their own judgement on the quality of individual data, for instance by the assumption that selected data have comparable quality characteristics or an acceptable (inferred) quality compared to their

requirements.

For practical reasons, the options selected for describing laboratory methods in WoSIS are assigned on the basis of the descriptions provided in the respective (database) sources. This implies that information interpreted from the original report (source materials) is used here. At a later stage, in principle, some refinements may be possible if the original data can be consulted again; realistically, such would be the task of the original data providers. Such a mechanism would be realizable once an inter-operable, federated system is in place as foreseen for GLOSI, within the framework of the Global Soil Partnership (de Sousa *et al.* 2021).

The WoSIS approach for the qualitative description of analytical methods can be seen as complementary to method descriptions used in reports from proficiency tests. In these cases, results from participants are coded to provide details of the methods applied within a particular grouping, such as the determination of 'soil organic carbon' content (WEPAL 2022). As explained above, the spread of the results within a given grouping can provide an indication for the spread in a compiled database.

Soil property 'pH KCl' will be used as example here. The selected 'method options' are sample pretreatment, the soil/solution ratio, the molarity of the KCl solution, and the measurement technique. It is assumed that each laboratory, for the particular soils investigated, uses a shaking method and an equilibrium time long enough for the measurement to get a stable reading. These conditions may differ per soil type and (pair of) electrode(s) used, but these are considered of minor importance for differentiating methods in the WoSIS database. Once an option is identifiable, based on the available (source) information, the appropriate option/value can be added (i.e., 0.1, 0.5, 1 M). Such a grouping allows users of the database to select subsets that are analyzed according to comparable analytical methods, and thus may be considered as having equal quality for a specific use. If necessary, values/options not yet considered in the 'method options' table can be added; a web interface for making such insertions is under development).

In addition to the method description according to the standardized coding system, values can be allocated for the inferred confidence in the conversion; this qualitative assessment is based solely on the information embedded in the 'summarized' method descriptions as provided in the various source databases. As indicated, these descriptions were often generalized by the data providers themselves from a more detailed source, such as 'their' laboratory manual. Importantly, the present confidence flags should not be seen as a measure for the quality of a particular laboratory. The criteria for coding 'standardized analytical methods' in WoSIS, as developed so far, are presented in Section 4.

The present approach also allows to accommodate data sets that are derived from proximal sensing methods. Soil sensing methods rely strongly on traditional laboratory methods for their calibration as expressed in region-specific soil spectral libraries (Viscarra Rossel *et al.* 2016; Shepherd *et al.* 2022) with the accompanying statistical models. The resulting, derived data can be described in WoSIS using the prefix '_spectral' with reference to the corresponding analytical method (e.g., 'CEC_pH7_spectral'.) Ideally, the underpinning spectral library and statistical

prediction model applied should be succinctly characterized in the WoSIS method option tables, in view of the associated uncertainties. Overall, data derived from traditional laboratory analysis are more reliable, though more costly, than those derived from spectrometry (Shepherd *et al.* 2022).

3. Criteria for standardizing soil analytical method descriptions

3.1 Background

This section explains WoSIS conventions for describing and coding soil analytical method descriptions. First, the guiding principles are explained in Section 3.2, then the methodology in Section 3.3, and subsequently the approach for describing possible method options (Section 3.4). A concise description of the approach adopted for each analytical method under consideration here (Table 2) is provided in Section 4.

All measurement values in WoSIS are expressed using SI units or non-SI units accepted for use with the International Systems of Units.

Table 1. List of soil properties with standardized analytical method descriptions.

Soil property	Standard units ^a
Bulk density	kg/dm ³
Calcium carbonate equivalent	g/kg
CEC	cmol(c)/kg
Coarse fragments	100 cm ³ /cm ³
Electrical conductivity	dS/m
Organic carbon	g/kg
pH	Unitless
Phosphorus	mg/kg
Sand, silt, and clay fractions	g/100g
Total carbon	mg/kg
Total Nitrogen	mg/kg
Water retention gravimetric (at given tension) ^b	g/100g
Water retention volumetric (at given tension) ^b	cm ³ /100cm ³

^a Conversions: g kg⁻¹ or permille (1 = 0.1%); vol% is equivalent to 100 x cm³ cm⁻³; wt % is equivalent to 100 x g g⁻¹; kg dm⁻³ is equivalent to g cm⁻³ or Mg m⁻³; dS m⁻¹ is equivalent to mS cm⁻¹, originally mmho cm⁻¹, at 25°C; cmol(c) kg⁻¹ is equivalent to meq 100g⁻¹. Layer depth (top resp. bottom) expressed in cm, measured from the surface, including organic layers and mineral covers (see Batjes *et al.* 2020).

^b Water holding capacity may be calculated as the amount (volume) of water held between 1/3 bar and 15 bar according to USDA conventions (Soil Survey Staff 2022).

3.2 Guiding principles

In WoSIS, the description of analytical results considers key aspects of methods that are widely used in soil laboratories for the particular soil property. These are tabled along with available method options with a concise description for each option, as further described in Section 4.

Laboratory results in publications and reports are generally labeled with information that can be found in laboratory manuals, yet the full information is seldom provided in the databases themselves. It is assumed here that all source data (as shared for consideration in WoSIS) have passed routine quality control procedures at the source laboratory and a rigorous check by the first user. If during that process the decision was taken to include or exclude a special treatment, the assumption in WoSIS is that the original decision was made correctly (i.e., that the given option was deliberately left out from the selected criteria). Examples for this are washing out of water-soluble salts in the method for the determination of the CEC, and use of non-dried materials in case of samples with volcanic ash. It is also assumed that when a laboratory method has characteristics that restrict its application to specific soil types, these have been duly pondered upon by the source laboratory. In principle, such checks form an integral part of quality management procedures in a laboratory (van Reeuwijk and Houba 1998).

As indicated, WoSIS is populated with data coming from many sources that consider different laboratory standards and approaches. Therefore, the number of method options has been limited to a practical level keeping in mind the intended use of the standardized data served from WoSIS (i.e., mainly for broad scale applications, see Poggio *et al.* (2021); for more detailed analyses at (sub)national level the 'local or national' databases should be consulted).

3.3 Coding of method options

Soil analytical methods are described using a sequential approach. First, the soil property matching the label of the source data to be entered in WoSIS needs to be selected. This must be done with utmost care: some soil property 'labels' in the source database can include information on the laboratory method applied; these are 'operational' definitions, for example 'CEC-NH₄OAc'.

Once the appropriate soil property (e.g., electrical conductivity) is identified, method characterization is according to the corresponding option table (see Appendix for examples). Next, the option(s) that best describe(s) the particular feature(s) of the laboratory method is(are) identified and coded. This step may be 'tricky' when incomplete information has been provided in the source metadata; in such situations it may be necessary to contact the original data provider for additional information². Alternatively, for some datasets it may be unrealistic to aim for a full characterization of the analytical methods. In such cases, a lower confidence level can be assigned based on an evaluation of all method options (from high (1) to low (3)). This step, however, is not

² In case a low confidence rating is specified in the standardized data sets, national data providers are encouraged to help ISRIC WDC-Soils updating the corresponding descriptions as they may have access to more detailed sources, such as the actual laboratory manuals.

shown in the option tables themselves (i.e., only visible to the database manager.)

Results of soil laboratory analyses are expressed with reference to a base, known as ‘fine earth fraction’ of a soil sample. This fraction is prepared during sample pretreatment at the source laboratory upon air drying. Next, part of the air-dried material is sieved. The actual sieve size determines both the upper limit for the fine earth fraction and the lower limit for the coarse fraction. Most countries use 2 mm as the upper limit for the fine earth fraction (Soil Survey Staff 2022). Alternatively, in the former Soviet Union and satellite countries, laboratories used 1 mm for this (Katschinski 1956). Analytical results expressed with respect to the <1 mm and <2 mm fractions as base are essentially different and difficult to harmonize unless large data sets are available for the comparisons (Sadovski and Ivanova 2020). Such aspects need special attention when international datasets are to be fully harmonized, for example using transformation or pedotransfer functions (Rousseva 1997; Nemes *et al.* 1999; Esmaeelnejad *et al.* 2016; Makó *et al.* 2017; Takahashi *et al.* 2020), and should be considered in international laboratory intercomparison programmes such as GLOSOLAN.

Sub-divisions (i.e., name and class size limits) within the defined coarse and fine earth fractions can vary markedly between countries as shown in Figure 1 (Nemes *et al.* 1999; GlobalSoilMap 2015). Further, as observed by Loveland (2016), interchangeability of sub fractions is restricted by differences in quality of say the pipette-method, hydrometer method, and the grainsize distribution determined using diffraction (e.g., Elfaki *et al.* 2016; Faé *et al.* 2019). Harmonizing such disparate textural data to a common standard remains an important challenge (e.g., GlobalSoilMap 2015; Moeys 2018; Sadovski and Ivanova 2020).

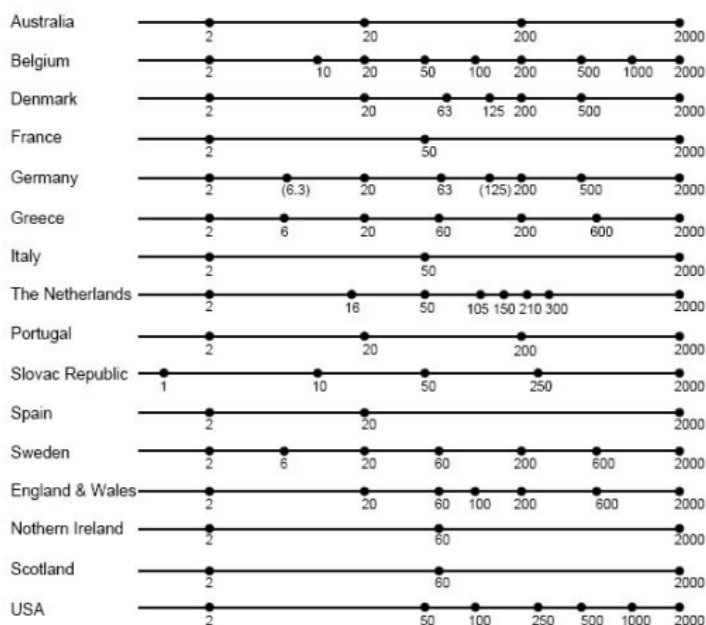


Figure 1. Particle size limits used in European countries, Australia, and the United States of America.

3.4 Example of coding procedure

The versatility of the WoSIS approach for coding soil analytical method descriptions, in a global context, is illustrated in Table 2. Five different reference methods for pH KCl, namely ISO 10390:2005 (ISO 2021), USDA (Soil Survey Staff 2022), ISRIC (van Reeuwijk 2002), WEPAL (2022) and GLOSOLAN (2021) are described by their main features.

Table 2. Procedure for coding standardized analytical methods using pH as an example.

<i>Key</i>	<i>Method</i>				
	<i>ISO</i> ⁵	<i>ISRIC</i> ⁶	<i>USDA</i> ⁷	<i>WEPAL</i> ⁸	<i>GLOSOLAN</i>
Pretreatment	<2 mm	<2 mm	<2 mm	<2 mm	<2 mm
Solution	KCl	KCl	KCl	KCl	KCl
Concentration	1 M	1 M	1 M	1 M	1 M
Ratio	1 : 5	1 : 2.5	1 : 1	1 : 5	1 : 5
Ratio base	v/v	w/v	w/v	v/v	w/v
Instrument	Electrode	Electrode	Electrode	Electrode	Electrode

4. Description of method options for soil properties standardized in WoSIS

This section describes method options considered in the '2023' WoSIS standardization workflow. In future, the list will be expanded when additional soil properties are standardized.

4.1 Bulk density

Background

Bulk density provides a measure for soil compaction. It is defined as the ratio of the mass of oven dried solids to the total or bulk volume. In contrast to particle size density, soil bulk density includes both the volume of solids and pore space. Pore space depends on the soil's extensibility and so the volume of soil changes almost dynamically with soil water content. Bulk density is needed to convert data from weight to a volume basis and *vice versa*.

Method

Sampling techniques for bulk density are determined by the natural setting of the solid soil components and the pores. Therefore, the moisture condition has to be defined at the time the bulk density is estimated. Measurements can be based on weight and volume.

Methods for determining bulk density are described using four method options:

- *Sample type*: In one group of methods, a sample is taken by pressing cores with known volume (rings) into the soil. Other methods use clods taken in the field; after sealing, the volume of the coated clod is measured by submerging into water (nowadays, this can also be done using 3D scanning). Alternatively, in some cases, clods are reconstituted from soil material <2 mm (Soil Survey Staff 2022). If soil coherence is too weak to prepare a clod or take a soil core, the volume of excavated soil is measured instead.
- *Measurement condition*: The moisture condition of the sample determines whether swelling or shrinking has taken place (e.g., air dried, rewetted).
- *Base*: The bulk density of a soil sample is the ratio of the mass of solids to the total or bulk volume. The total volume includes both solids and pore space. Bulk density is usually reported for the '<2 mm' soil fabric, the mass and volume of rock fragments are subtracted from the total mass and volume (Soil Survey Staff 2022).
- *Corrections*: Bulk density is calculated for soil materials <2 mm in g cm^{-3} (i.e., fine earth fraction). Mineral materials larger than 2 mm and vegetal materials are removed from the sample. If the density of rock fragments is known or can be approximated, corrections can be included in the calculations of the bulk density (Soil Survey Staff 2014). Sometimes

bulk density is expressed on the basis of the whole soil. Such measurements refer to all particle-size fractions, including boulders with maximum horizontal dimensions less than those of the pedon (Grossman and Reinsch 2002; Soil Survey Staff 2022).

- *Calculation*: When not actually measured, bulk density can be based on expert knowledge. In such cases, 'calculation' is flagged as 'unknown.' (Such values are not served with 'wosis_latest')

4.2 Calcium carbonate equivalent

Background

Carbon in soils can be subdivided into organic carbon and inorganic carbon. Inorganic carbon is present in various mineralogical materials such as carbonates (e.g., CaCO_3 , MgCO_3 , $\text{CaMg}(\text{CO}_3)_2$). A convenient solution in the laboratory is to express results for inorganic carbon forms as if originating from calcium carbonate, i.e., as 'Total Calcium Carbonate Equivalent.' Most soils with a pH less than 7 do not contain any significant amounts of carbonates.

Inorganic carbon can be quantified by dissolution of carbonates in acid and titration of residual acid, or measurement of evolved CO_2 by volume, pressure, or absorption on a solid or precipitation in a solution. Instrumental methods use sensors for CO_2 in purified streams of evolved gases with acid treatment or burning at elevated temperatures. An alternative is to measure the weight loss with burning at selected temperatures. For a detailed description of available options see (Soil Survey Staff 2022).

Method

Analytical methods to determine calcium carbonate equivalent in soils are described using the following method options.

- *Sample pretreatment* : Analyses are for the fine earth fraction using the limits defined by the source laboratory, which is <1 mm or <2 mm.
- *Reaction*: Quantification of carbonates often is based on dissolution in acids. The choice of the acid depends amongst others on the expected amount of carbonates, and the subsequent detection technique. The concentrations should be strong enough to get dissolution in a reasonable time but exclude the risk of and error by acid fumes.
- *Temperature*: At higher temperature dissociation of acids and so dissolution of carbonates increases. Possible options are described in the feature table, for example 'dissolution of carbonates by sulphuric acid [H_2SO_4]'.
'
- *Detection*: Where substantial amounts of carbonates are present, titrimetric methods for the excess of acid after dissolution or the volume of CO_2 evolved at dissolution can be

used. Evolved CO₂ can also be quantified by increased pressure in a fixed volume. Alternatives are gravimetric methods with precipitation of CO₂ or weight loss from the sample. If the evolved stream of gases is purified, sensors sensitive to carbon forms can be applied as well. These instrumental methods (element analyzers) usually are applied only with smaller amounts of (inorganic) carbon / carbonates in the sample (Soil Survey Staff 2022).

- *Calculation:* Many laboratories for soil analyses do not have methods that can discriminate inorganic C that originates from various sources. Most methods are based on acid consumption with dissolution of carbonates, or quantification of CO₂ evolved. An indirect method is the subtraction of total organic carbon (TOC) from total carbon (TC). These forms are for instance determined by element analyzers with and without prior acid treatment for removal of carbonates. In all cases the quantified amounts have to be converted to the same molecular amounts of CaCO₃.

4.3 Cation exchange capacity

Background

Micro- and macro-nutrients and heavy metals are held and released into the soil solution from exchange sites associated with clay and organic matter fractions; cations are held by the negative charges at the exchange sites. The electric charge that can develop at these sites varies with clay mineralogy, type of organic matter and the pH (Dixon *et al.* 1977). The cation exchange capacity (CEC) is an estimate of the total amount of cations held at these exchange sites, at a pre-defined pH (e.g., 'pH7 buffer'). The 'effective CEC' is the CEC determined at the pH of the soil.

Salts present in the soil are considered a separate resource of ions and have to be removed by washing prior to analyses of the saturation status of the exchange complexes. If present in considerable amounts, sodium and calcium from carbonates, and gypsum can dominate the saturation of the exchange sites. Part of these soil components may dissolve in the solutions used (pH 8.2 to 8.5) preventing complete saturation of exchange sites in the lab methods as described below.

Usually, before the determination of the CEC, the amount and type of exchangeable bases are determined first. These 'exchangeable bases' are Calcium, Magnesium, Sodium and Potassium. From these, the bases saturation can be calculated as the ratio of exchangeable bases and the cation exchange capacity (at pH 7).

Method

Various methods are used to determine CEC. In practice, these methods are selected considering available lab facilities, soil types to be investigated and specific (research) requirements. CEC

methods are described here according to their major features; detailed information about specific methods, including their limitations, may be found in the underpinning laboratory manuals. In the standardization process for WoSIS, CEC methods are first subdivided on the basis of the technique applied (i.e., direct and approximate (indirect) methods).

- *Sample pretreatment* : This feature indicates whether data are reported for a fine earth fraction defined as having an upper limit of 1 mm (Katschinski 1956) or 2 mm (most common).
- *Technique*: As a preceding step to the determination of the total amount of cations that can be held in soil often the actual saturation of the exchange sites with basic cations is determined. This amount is known as ‘exchangeable bases.’ Typically, these cations are exchanged by a (buffered) solution with a cation occurring in the soil (e.g., NH_4^+ which is not considered a ‘base cation’). If exchangeable acidity (sum of exchangeable Hydrogen and Alumina) is also needed, the actual saturation of the exchange complex with cations can be estimated (indirect method), although minor fractions of manganese and iron are ignored in the method. These techniques with summation (of groups of) exchangeable cations are considered here as indirect methods for the determination of the CEC.
- *Reported pH*: The release of cations from and the saturation of the exchange sites depend on the charge of the exchange surfaces, as determined by clay mineralogy and type of organic matter. These charges will vary with the pH of the exchange solution (e.g., buffer) as applied in the laboratory method. This effect is particularly for soils with variable charge components (e.g., volcanic materials) and high organic matter content. Conversely, soils with a permanent charge show only small differences in CEC with increasing lab pH (Dixon et al. 1977; Gillman 2007). The ‘CEC’ is often determined at pH 7 that is with use of solutions buffered at pH 7; this CEC value then is considered representative for most agricultural soils. If the CEC at field pH is needed, then unbuffered solutions are used for saturation.
- *Exchange solution*: 1 M KCl and 1 M NH_4Cl are considered un-buffered solutions; they can be used for the determination of exchangeable acidity in soils with pH less than 5.5. Alternatively, the potentially available acidity is determined by extraction with a solution buffered at pH 8.2 such as BaCl_2 ; the change in acid ‘consumption by the buffer’ is measured by back titration of the solution. This amount (i.e., potential acidity) is generally higher than the amount of field exchangeable acidity.

The total amount of cations that can be held in soil can also be calculated in a *direct* way: the soil sample is saturated by applying an excess of a suitable index cation with subsequent determination of the excess or replacement of the index cation by another cation (see *direct methods* below).

- *Replacement solution*: Once the exchange sites are saturated, the excess of the solution used for the exchange is washed out. In a next step the index cation is brought into solution by a replacement solution (buffered) at the particular pH. This is used for instance

in the well-known CEC methods with BaCl₂ – Triethanolamine (TEA) for (Ba²⁺) buffered at pH 8.2, and NH₄-acetate/Na-acetate at pH 7 and similar methods. This (washing and) replacement step can be omitted if the amount of index cation removed from the (buffered) index cation solution is estimated. For instance, if solutions with Ag-Thiourea (+) unbuffered or buffered from 4.0 to 8.0 are used.

- *Index cation:* The index cation criterion further specifies the method for the determination of the CEC. The index cation can be a cation from a buffer solution, or a cation added to the buffer solution. The latter group of cations are not common in soils or cations with a high selectivity. Their selection may depend on the clay minerals and type of organic matter present in a given sample. For some of these cations sophisticated instruments like flame atomic absorption and inductively coupled plasma spectrometers are needed for quantification.

CEC can be determined using *direct* and *indirect* methods as described below:

- *Direct methods:* These are used to achieve complete saturation of exchange sites at a particular pH, buffered solutions that contain a cation with high selectivity are used to saturate all exchange sites. The cation can be regarded as an index cation if the cation is used to quantify the exchange sites. The index cation can be a cation from a buffer component (i.e., Na⁺ or NH₄⁺). Cations that can be considered absent in the soil (i.e., Ba²⁺, Ag-thiourea, and Li⁺) or have been exchanged in previous treatments (i.e., Na⁺) can also be index cations. To improve exchange often solutions with a high concentration of the index cation and replacing cation have to be used. The selection of an index cation is not only based on the selectivity of the ions at the exchange sites, but also on the instrumentation available for quantification.

To enhance the saturation with an index cation, samples can be pre-leached with a buffered solution as well. The small amount of buffer solution and cation(s) left improves the selectivity of the analysis.

Further information may be found elsewhere (Baize 1993; Pansu and Gautheyrou 2006; Soil Survey Staff 2022).

- *Indirect method (CEC by approximation):* Cations that may be present on the exchange sites can be subdivided into three groups. Through summation of the cations in these three groups, the cation exchange capacity can be approximated in an indirect way. In most agricultural soils, the exchangeable bases Calcium, Magnesium, Sodium and Potassium dominate. In case of soil with a pH <5 part of the exchange sites will be saturated with Alumina and Hydrogen. Iron and Manganese are often the most prominent in the 'rest group.' The cation exchange capacity can be approximated by summation of these groups of cations (Pansu and Gautheyrou 2006; Soil Survey Staff 2022).

Exchangeable bases and exchangeable acidity can be determined in buffered and unbuffered solutions. If determined in unbuffered solutions the CEC at (field) soil pH is approximated. It should be noted that the CEC based on summation of individual cations may be less accurate than values for CEC determined using a *direct* determination.

4.4 Coarse fragments

Background

As indicated, the solid part of the soil mass is subdivided by sieving into a fraction smaller and a fraction larger than 2 mm; these fractions are often denoted as the fine earth and coarse earth fraction. Data sets from the former Soviet Union and its satellites, however, often use 1 mm as upper limit (Katschinski 1956) for the fine earth fraction.

The amount of coarse fragments is needed in calculations that consider the total soil mass as reference. Internationally, different systems are used for naming subclasses within the coarse fraction, using a range of class limits and names (Nemes *et al.* 1999; FAO 2006; ISO/TC 190 2016; ANSIS 2023).

Alternatively, the fine earth fraction is most relevant for soil processes. This fraction provides the base material for most soil physical, chemical, and mineralogical analyses; see section on particle size distribution and analysis (sand, silt, and clay fractions).

Method

Methods for the determination of mass of coarse fragments are described using the following method options:

- *Sample pretreatment* : This feature indicates whether data are reported with reference to an upper limit of 1 mm or 2 mm for the fine earth fraction.
- *Size*: The coarse fraction can be subdivided into several subclasses. Typically, their respective amounts can best be estimated or weighed in the field or laboratory.
- *Type*: The coarse fraction can comprise several types of materials which can be described according to their size and nature, as e.g., determined by their resistance against disintegration in water or sodium-hexametaphosphate. During sample pretreatment, pedogenetic materials have to be carefully removed from the sample and treated as a separate sub fraction. Breaking up of such materials may lead to the release of smaller ferromanganese and calcium carbonates (and barium sulphate or gypsum concentrations) nodules.

- *Estimates*: There are two ways to express the amount of coarse fragments (i.e., volume (V) basis and weight (W) basis). For conversions from V to W the bulk density is required. In the absence of such data, default data for bulk density (1.45 g cm³) and mineral particle size density (2.65 g cm³) are often assumed (Soil Survey Staff 2022).
- *Base*: The amount of respective coarse fractions can be expressed as part of the whole soil or related to e.g., the fine earth fraction or other fractions determined.

4.5 Electrical conductivity

Background

The electrical conductivity (EC) of a soil extract is a measure for the salt content in the solution (salinity of soil). EC is a measurement that correlates with soil properties that affect crop productivity, including soil texture, cation exchange capacity (CEC), drainage conditions, organic matter level, salinity, and subsoil characteristics. The saturation extract (EC_e) is considered to give a better representation of actual soil conditions with respect to plant environments (Soil Survey Staff 2022).

Method

Electrical conductivity is described using four method options:

- *Sample pretreatment* : The analyses apply to fine earth fraction as defined in the source laboratory (i.e. <1 mm (Katschinski 1956) or <2 mm.)
- *Solution*: Electrical conductivity is measured in soil extracts prepared with distilled or demineralized water (i.e., with very low electro conductivity).
- *Ratio*: The amount of salts that can be dissolved depends on the soil / water ratio used (e.g., 1:5). These ratios are often predefined in soil classification systems and soil fertility evaluating schemes. With smaller ratios, EC can easily be determined in the supernatant solution after sedimentation of coarser materials. Saturated paste solutions are close to the soil solution. Often the pH H₂O and EC are measured in the same soil extract (soil/water ratio).
- *Instrument* : EC is measured using an electro conductivity electrode. This can be done in the laboratory and in the field using hand held instruments (Rhoades *et al.* 1999; Soil Survey Staff 2022).

4.6 Organic carbon

Background

Carbon in soils is divided over several fractions and components. Major fractions are organic and inorganic carbon. Inorganic carbon is present in mineralogical materials such as carbonates. Cells of microorganisms, plant and animal residues at various stages of decomposition, stable 'humus' synthesized from residues, and nearly inert and highly carbonized compounds, such as charcoal, graphite, and coal (Nelson and Sommers 1982, p. 347), all contain organic C. Organic materials coarser than 2 mm are considered residues. The 'fine earth' fraction is the basis for the laboratory analyses for soil carbon.

The preferred method for determining 'total organic and inorganic carbon,' or total carbon (see Section 4.10), should consider an element analyzer that allows for controlled temperature and detection based on CO₂ evolution. Other methods often require a correction for lower recovery (Lettens *et al.* 2007).

Method

Methods used for the determination of organic carbon are described using six method options.

- *Sample pretreatment*: This feature indicates how a laboratory has defined the upper limit for the fine earth fraction, as discussed earlier: <1 mm or <2 mm. These values also set the lower limit for the coarse fraction.
- *Pretreatment*: Carbon in the fine earth fraction can be determined by oxidation and quantification of CO₂ released. If the sample also contains carbonates (i.e., inorganic carbon) this fraction can liberate CO₂ as well. This inorganic fraction can be removed by acid treatment (without oxidation power) prior to the actual determination of organic carbon.
- *Reaction*: Organic carbon can be determined by wet or dry oxidation (ISO-10694 1995; van Reeuwijk 2002; Soil Survey Staff 2022). For wet oxidation, mixtures of bichromate and sulphuric acid are frequently used. Determination can be based on excess bichromate or CO₂ development. Methods based on CO₂ detection need sample pretreatment to remove carbonates. CO₂ is also liberated with dry oxidation and quantified in element analyzers.
- *Temperature*: In both wet and dry oxidation methods the completeness of the reaction depends on the temperature. The heat of reaction during wet oxidation should be sufficient to induce substantial oxidation of organic materials (i.e., a high recovery fraction for organic carbon, see GLOSOLAN 2019)) Thermal decomposition of bichromate may occur when external heating cannot be limited and controlled during wet oxidation. Dry oxidation requires a furnace with controlled temperature of over 900 °C.
- *Detection*: Excess of bichromate can be determined by back-titrating with Fe³⁺. Alternatively, the organic carbon content can be calculated from the amount of chromic ions (Cr³⁺) formed using colorimetric techniques. Wet oxidation methods with detection based on excess bichromate are hampered by interferences from charcoal, Fe²⁺, Mn²⁺ where present.

In a furnace, with extra oxygen and catalysts added to complete oxidation to CO₂ and purification of the gas stream evolved, total C (organic C and inorganic C) can be detected. Gas chromatography, coulometric methods and/or infrared-based methods can be used to quantify CO₂ evolution. Inorganic carbon should preferably be removed by prior acid treatment.

- *Calculation:*

Dry oxidation: Total carbon (TC) is defined as the sum of total organic carbon (TOC) and total inorganic carbon (TIC), where TIC is derived from the calcium carbonate equivalent or from a direct determination (as measured for basic soils). Methods to assess TIC by prior treatment with acid or separate determination are covered in Section 4.10.

Wet oxidation: Methods based on the original method of Walkley and Black (1934) often involve incomplete oxidation (Lettens *et al.* 2007; Soil Survey Staff 2022) (and consider a recovery factor. This factor is based on the assumption that 77% of the total carbon present is determined during the analyses. However, in practice, the actual factor will depend on the type of soil organic matter present in the sample and soil type (Grewal *et al.* 1991; Lettens *et al.* 2007). Use of a correction factor that is not representative for the soil material under consideration will introduce a systematic error. Only few methods with wet-oxidation, with or without external heating, claim 100% recovery (for all soil types); for a critical review see (Pribyl 2010).

4.7 Organic matter

Background

Soil organic matter has been defined as the organic fraction of the soil exclusive of undecayed plant and animal residues. It has been used synonymously with “humus.” For laboratory analyses, however, the soil organic matter generally includes only those organic materials that accompany soil particles in the fine earth fraction (Soil Survey Staff 2022). Organic matter content can be determined directly by ignition (400 °C) after which the loss in weight of the soil sample is taken as a measure of the organic matter content as well as *indirectly*.

Method

- *Sample pretreatment* : This feature indicates how a laboratory has defined the upper limit for the fine earth fraction, as discussed earlier: <1 mm or <2 mm. These values also set the lower limit for the coarse fraction.
- *Detection*: Weight loss on ignition (LOI), which involves ignition at 400 °C in a furnace, is used as a measure for the content of organic matter present in the fine earth fraction.
- *Calculation*: No conversion factor is applied for soil organic matter (i.e., LOI values are reported as is). According to Soil Survey Staff (2022), the percent organic matter lost on

ignition (400 °C) can be used in place of *indirect* organic matter estimates of SOC loss by the Walkley-Black organic C method. x

Note: Generally, it is assumed that soil organic matter contains 58% C hence the commonly used conversion factor of 1.724 times the amount of soil organic carbon (van Bemmelen 1890). The actual conversion factor, however, may be up to 2.0 for topsoil's and even 2.5 for subsoils depending on the type of soil organic matter (Pribyl 2010; Minasny et al. 2020; Shamrikova et al. 2022). An appropriate factor should be used to convert soil organic matter (SOM) content to content of soil organic carbon (SOC): $\text{SOC (g/kg)} = 1/\text{factor} * \text{SOM (g/kg)}$; this factor should be indicated in the corresponding method option table, when possible.

4.8 Soil pH

Background

The soil solution reflects the composition of the soil. Therefore, the pH and electro-conductivity of a standardized soil water mixture are often used for the first discrimination of soils and soil layers. The difference in pH measured in a soil/water extract and extract with 1 M KCl (pH delta value) for instance is an indication for the potential acidity. This 'delta pH' should be measured with equal conditions for both measurements. For some analytical methods, the pH determines the sample pretreatment for that method (i.e., presence of carbonates) or is a criterion for additional analyses. pH in saturated paste of soil, for example, is used in cross-checking salinity data (Rhoades *et al.* 1999).

Method

Methods for the determination of the pH of soil samples are characterized using seven method options:

- *Sample pretreatment* : Refers here to the upper size limit adopted by a laboratory to define the 'fine earth fraction.' Two physical pretreatments are widely used internationally for this: a) sieve crushed, dried soil material through a 1 mm sieve, and b) idem, but using a 2 mm sieve. By implication, these values will determine the corresponding lower limit for the 'coarse fraction.'
- *Solution*: To determine the pH of a soil sample, H⁺ ions need to be brought into solution. Distilled water and solutions with low ionic strength are used to stimulate this process. 'Stronger' salt solutions are needed to force exchange of i.e., Al³⁺. Measurements in a saturated paste are aimed to represent the natural environmental conditions Pansu and Gautheyrou (2006).
- *Ratio*: pH methods consider different 'soil / solution' ratios. The specific ratio determines the composition of the supernatant solution, leading to different pH values. More ions

can dissolve in a larger volume until maximum solubility is reached for the particular combination of exchanged and dissolved ions. Agitation time and method of shaking, as well as place of measurement (i.e., 'in the rest' or 'actively stirred' portion) have to be standardized in a laboratory to establish consistent measurement conditions. To obtain reliable measurements for pH H₂O in soils with high organic matter content, usually a higher water: soil ratio is used (Pansu and Gautheyrou 2006). Measurements in which electrodes are in contact with the sediment, may show a 'suspension' effect; this effect can modify results by +/- 1 pH unit.

- *Ratio base*: Results can be expressed on a weight or volume basis; as such, ratios can be expressed as weight / volume (w/v) and volume / volume (v/v).
- *Concentration*: Solutions with a high salt concentration enforce ion exchange processes. For instance, a 1 M KCl solution is used to release hydrogen ions and Al³⁺ ions from the exchange complex. With 1 M NaF, OH⁻ is released in the solution and Al³⁺ complexed; the observed increase in pH is an indication for 'active aluminum' (van Reeuwijk 2002).
- *Instrument* : In the laboratory, usually the pH is measured with a pH-meter. The (combination of) electrode(s) and their maintenance are controlled by the laboratory; they are checked before the measurement starts with calibration of the instrument. As the position of the electrode in the solution is considered most important, the type of electrode used is not considered a key-criterion here. pH test strips and hand-held pH meters are also used to measure soil pH in the field; however, such pH values may differ widely from those recorded under standard laboratory conditions.
- *'Monitoring'*: This term is used here solely with respect to observing (monitoring) changes in oxidizable sulfur compounds over time for which changes in soil pH are measured until equilibrium is reached, providing a measure for 'sulfidic soil materials' (Soil Survey Staff 2022). Such conditions commonly occur in intra-tidal zones adjacent to oceans.

4.9 Soil texture

Background

The particle size distribution (PSD) in soils is important in a wide range of agricultural, geological, and technical and engineering studies. It influences soil properties such as the workability, water holding capacity and drainage, and the capacity to hold (micro) nutrients as a reservoir for plant growth. For most laboratory investigations samples are first passed through a sieve to determine the 'fine earth' fraction (van Reeuwijk 2002; Soil Survey Staff 2022); the upper limit of 2 mm is widely used for this. Alternatively, in the former Soviet Union and its satellite countries, the upper limit for fine earth fraction has been set at 1 mm (Katschinski 1956).

The 'coarse fraction' is always defined with reference to the upper limit of the 'fine earth fraction'

in the corresponding scheme. Whole soil refers to all particle-size fractions, including boulders with maximum horizontal dimensions less than those of the pedon. In general, the term soil texture is used to describe the fine-earth fraction (Soil Survey Staff 2022). At one time, the term rock fragments was differentiated from the term coarse fragments, which excluded stones and boulders with diameters >250 mm (Soil Survey Staff 1975). The rationale for this distinction was that particles <250 mm were generally regarded as part of the 'soil mass' as they affect moisture storage, infiltration, runoff, root growth, and tillage (Soil Survey Staff 1951).

Soil particles in the fine earth fraction can be subdivided into fractions of distinct size classes. In most cases, the following (nominal) classes are defined: 'sand,' 'clay,' and an intermediate class named 'silt.' However, the actual size limits for these classes can vary between countries and laboratories, for example an upper limit for the 'silt' fraction given as 0.050, 0.060 or 0.63 mm (see Figure 2). Such fractions, in turn, can be subdivided to provide additional detail (e.g., fine silt and very fine silt); again, subclasses are defined using different size limits and (local) names, complicating overall standardization and harmonization efforts (Loveland and Whalley 2000).

In view of the above, in WoSIS descriptive criteria for 'sand,' 'silt,' and 'clay' are presented in one single feature table. During laboratory analyses, these fractions are often determined simultaneously using the same sample.

Method

Analytical methods for the determination of the sand, silt, and clay (size) fraction in soil samples are described using three options for the sand and silt size fraction, and four for the clay size fraction. When the exact fraction sizes are not reported in the source metadata, of necessity the inferred class limits are sometimes inferred (with a flag for lower confidence).

- *Sample pretreatment*: Refers here to the upper size limit adopted by a laboratory to define the 'fine earth fraction.' Two physical pretreatments are used internationally: a) sieve crushed, air dry soil material through a 1 mm sieve (Katschinski 1956), and b) idem, but using a 2 mm sieve (Soil Survey Staff 2022). By implication, these values will determine the corresponding lower limit for the 'coarse fraction,' as well as the actual 'base' to which results of soil chemical analyses apply.
- *Size*: Fractions for a given particle size distribution should be distinguished according to their size and not according to their allocated names as used in different (inter)national systems. This practice facilitates logical grouping of size fractions in WoSIS according to the particle size distribution schemes as adopted in the various source databases.
- *Treatment*: Soil particles are often bound into aggregates by cementing materials such as organic matter, calcium carbonate and iron oxides. Such aggregates are broken up during the pretreatment step of the analytical method. For example, hydrogen peroxide (H₂O₂) is used to remove organic matter by oxidation. HCl, HAc (acetic acid) or buffer solutions (Na acetate) are used to remove carbonates in basic pH soils (pH >7). Ultrasonic treatment is needed for samples that contain kaolinite, micas (e.g., illite and muscovite) and

allophanic materials; the energy generated by the vibrations is transferred into the suspension and breaks the cohesion of the aggregates. In some laboratories, sesquioxides are removed in an optional deferration step. Many of these treatments are soil type specific. When such (pre)treatments and dispersion methods are excluded from the laboratory method, measurement will be for 'water dispersible' or 'natural clay' (see below).

- *Dispersion*: During the dispersion of clay size particles, effects of the electric charges at the exchange surfaces have to be controlled carefully (Soil Survey Staff 2022). A clay suspension is most stable when the particles have large electrical double layers which is the case with monovalent cations and diluted solutions (Bolt and Bruggenwert 1978). Chemisorption of phosphate at pH values far from the isoelectric point, can also reduce repulsion forces by charges on the clay surface. Therefore, a mixture of sodium hexametaphosphate buffered at pH 10 by Na_2CO_3 (also known as 'Calgon' type) is frequently used as dispersant. Alternative dispersants include ammonia and soda. Well-dispersed soil solutions remain "turbid" for a longer time than those that have not been submitted to any pretreatment.
- *Instrument*: Subsequent to pretreatment and dispersion of the fine earth fraction, this fraction is divided into so-called sand, silt, and clay fractions (see above discussion about class size limits and naming issues). In the '<2 mm' scheme, the sand fraction is separated from the silt and clay fraction by sieving; for example, using 0.050 or 0.063 mm as the lower limit. Typically, the silt and clay fractions are determined after sedimentation in a cylinder based on Stokes' law, which assumes silt and clay particles to be perfect spheres. The rate of sedimentation depends amongst others on the radius, shape, density of the different particles, density of the liquid used for dispersion, and temperature (Soil Survey Staff 2022). Detailed descriptions of possible error components in results from particle size analyses can be found elsewhere (Loveland 2016).

The main instruments for particle size analysis include:

- *Pipette method*: In the (Robinson) pipette method the relevant (i.e., <0.050 or 0.063 mm) fraction is sampled at a predefined depth and time after sedimentation started. The actual depth is calculated from Stokes' law for a specific settling time interval, size fraction and temperature. The pipette method is considered to be the reference method.
- *Hydrometer*: This method (often referred to as Bouyoucos hydrometer method) is commonly used as an alternative to the 'pipette' method. It is based on the density of the suspension, which is a function of the concentration and kind of particles present (after a certain time of settling). Results are considered to be less reliable than those obtained using the pipette method.
- *Laser diffraction (beam)*: These methods for grain size analyses are based on the measurement of the intensity of diffracted laser beams of near infrared / infrared light

on suspended particles. The scattered light is detected at an angle (often 90°) of the incident beam. The number of detectors used, determines the number of fractions in the measurement range. With a high number of detectors, this results in a 'continuous' spectrum. Wavelength, size, refractive index of surface of particles are principal factors in the calibration of the instrument. Results may differ from those obtained with the pipette method for the same samples (Loveland and Whalley 2000; Pieri *et al.* 2006; Buurman and Van Doesburg 2007).

- *Field hand estimates*: Trained personnel with field experience in a specific region or with specific soil types may estimate the clay content in a semi quantitative way by 'hand, finger' sensing. Texture classes assigned from field texture and laboratory particle size analyses are not always equivalent, especially where the clay content is considered to be high (Minasny *et al.* 2007; Richer-de-Forges *et al.* 2023).

4.10 Total carbon

Background

Carbon in soils can be divided in an organic and inorganic fraction. Inorganic carbon is present in carbonate minerals such as calcite (CaCO_3) and dolomite (MgCO_3). Organic carbon is found in organic materials such as plant residues, humus, and charcoal. Total carbon (TC) is defined as the sum of total organic carbon (TOC) plus total inorganic carbon (TIC, expressed as Calcium carbonate equivalent). From an analytical point of view, TOC and TIC are defined measurement categories, also referred to as 'sum parameters', whereas each part can contain several substances in varying proportions (Hilke 2015).

Method

Analytical methods for total carbon are described using two options.

- *Sample pretreatment* : Analyses are for the fine earth fraction using the limits defined by the source laboratory. This can be <1 mm or <2 mm, which may be seen as the present international standard for defining the coarse fraction.
- *Calculation*: When small amounts of inorganic carbon are present, total carbon can be estimated by dry combustion at higher temperature using an elemental analyzer (Kuhlbusch *et al.* 2009; Soil Survey Staff 2022). However, in practice inorganic carbon is removed first after which the organic carbon fraction is determined. Larger amounts of inorganic carbon usually are usually determined after dissolution of the mineral fraction with acids and release of CO_2 . Results are expressed as total carbonate equivalent. Total carbon can be calculated using a range of methods for both inorganic and organic carbon.

4.11 Total Nitrogen

Background

Nitrogen (N) in soils is present as NO_3^- and NH_4^{+2} with negligible amounts as NO_2 ; together they form the mineral N-fraction. N in organic matter forms the organic fraction. N_2O and N_2 are present as gases. Due to microbial activity and other soil processes, the size of the different fractions will vary with time and management conditions (including sample pre-treatment). The individual N-fractions present at the time of sampling are estimated by extractions. For the determination of Total Nitrogen, two methods are commonly used: combustion and digestion. A detailed account is provided in soil laboratory manuals (van Reeuwijk 2002; Soil Survey Staff 2022).

Method

Analytical methods to determine Total Nitrogen are divided over four techniques and described using three more method options.

- *Sample pretreatment* : Analyses are for the fine earth fraction as defined by the source laboratory, that is <1mm (Katschinski, 1956) or <2 mm.
- *Technique*: Traditional determinations for Total Nitrogen can be grouped into two categories: combustion of organic matter in a furnace (Dumas) and digestion with acid treatment and heating (Kjeldahl). NIR/MIR spectroscopic methods allow for non-destructive determinations of Nitrogen, yet these methods need to be calibrated against conventional wet-chemistry measurements (Shepherd *et al.* 2022).

- *Digestion*: Since the introduction of the Kjeldahl method for the determination of N, the method has been modified to accelerate the method and include all forms of nitrogen in soils. Salts like Potassium or Sodium-sulphate are added to sulphuric acid to raise the boiling point of the digestion mixture. A catalyst (i.e., CuSO_4) is used to accelerate the reactions. An addition of KMnO_4 or HClO_4 may be needed to complete the digestion.

To include NO_3 and NO_2 , the Kjeldahl digestion is preceded by an oxidation step with i.e., H_2O_2 . After excess H_2O_2 is removed, NO_3 is coupled with salicylic acid in the sulphuric acid medium. Some methods use sodium thiosulfate to reduce the formed nitro-compounds into amino compounds. Fixed NH_4^+ is liberated with HF-treatment and combined into the Kjeldahl digestion.

- *Combustion*: In the Dumas combustion method, Nitrogen is oxidized with copper(I) oxide in a stream of CO_2 or with O_2 , in an elevated temperature furnace at 900 - 1350 °C. The flushed gas stream is purified and after conversion of NO_x to N_2 total Nitrogen is estimated.
- *Detection*: With sodium hydroxide, Nitrogen as NH_3 is released from the Kjeldahl digest and distilled into a solution of boric acid and titrated. For quantification by semi-automatic systems, the digest is diluted, and an aliquot used for the colorimetric

determination of phosphorus by nitroprusside reaction at 660 nm.

Modern Element Analyzers use thermal conductivity for quantification. Other systems measure N₂ manometrically after absorption of CO₂.

4.12 Phosphorus

Background

Phosphorus is one of the major plant nutrients in the soil and one of the most complex. It is a constituent of plant cells, essential for cell division and development of the growing tip of the plant. Therefore, it is vital for seedlings and young plants. Phosphorus quickly binds with iron and aluminum in the soil and becomes unavailable to plants, especially when soil pH CaCl₂ is below 5.0.

Method

Analytical methods to determine total Phosphorus are divided over thirty-three techniques and described using three more method options.

- *Sample pretreatment* : Analyses are for the fine earth fraction as defined by the source laboratory, that is <1mm (Katschinski, 1956) or <2 mm.
- *Technique*: To determine total phosphorus, sample material has to be decomposed with acids and oxidizing reagents and destructed at high temperature. Fractions of this total phosphorus can be determined by selective extractants (e.g., Bray and Kurtz 1945; Olsen *et al.* 1954; Levy and Aschlesinger 1999; Elrashidi 2010). Spectroscopic methods like MIR/NIR may be used, but these non-destructive methods need calibration against conventional laboratory methods to allow for interpretation of the P-measurements. Extractants (for extractable P): Soil-P is one of the most cumbersome properties to measure and interpret (Soil Survey Division, 2014). The pH of a soil is a useful criterion for selecting the appropriate extractant for 'extractable-P' (Elrashidi 2010). Such extractants differ in pH, buffer capacity, and ionic strength which makes them selective for certain P types and fractions. As a result, measures of extractable P for a given soil sample (soil type) as obtained using different methods, such as P-Olsen (for basic soils) and P-Bray (for acid soils), are not interchangeable.

As indicated, the 'chemistry' of the analytical methods is complex due to the different (types) of components in the extractant. Soil legacy data thus can best be grouped first using the source (name) of the extractant (e.g., P-Olsen, P-Bray 2, or Mehlich III), and subsequently considering key features of the extractant as given in the description.

- *Decomposition (for Total P)*: For total P methods, the sample has to be decomposed by

treatments with destructive and oxidizing capacities. Several mixtures of acids can be used. In the same process organic matter is oxidized.

- *Detection:* For quantification of Phosphorus, the colorimetric ‘molybdate blue’ and ‘molybdate yellow’ method are considered standard methods. Organic matter in the extract may give an interference by its colour and so has to be removed. Adsorption to charcoal and oxidation of dissolved P are frequently used. These treatments of the extract and the acids used towards the end of the method may influence the fractionation of phosphate in the extract. Ascorbic acid is often preferred over SnCl_2 as a reductant for colour development in the Molybdate-blue method.

4.13 Water retention

Background

The capacity of a soil to hold water (and air) depends on the amounts and types of organic matter, content of sand, silt, and clay, as well as soil structure or physical arrangement of the particles. Water and air are held in the inter-connected pore spaces between the solid materials. If all these spaces are filled with water, the soil is at its maximum water holding capacity. The corresponding volume of water can be gradually depleted by plant growth, transpiration, evaporation, and drainage. Water is less readily released by and transported through smaller sized pores. In some soil types, such as shrink-swell clays, the water holding capacity can change with desorption or absorption of water (hysteresis effect).

In the laboratory, water holding capacity at predefined suctions is determined by suction or application pressure on a sample. A detailed account is provided in soil physics handbooks (Koorevaar *et al.* 1983; Jury *et al.* 1991) and laboratory manuals (Soil Survey Staff 2022). Currently, members of the SOPHIE network are working on the standardization and harmonisation of soil water retention methods (SOPHIE 2022; Guillaume *et al.* 2023).

The nature of the soil material may preclude some sampling types. For example, water retention for expanding clays is overestimated when sieved samples are used instead of natural soil fabric at tensions of 6, 10 and 33 kPa (Soil Survey Staff 2022). Cores cannot be used when the soil material is not coherent enough, such as in very sandy soils.

When reporting values for water retention, it is important to note whether results are expressed on a w/w basis or w/v basis. Conventionally, with pF curves the moisture content is expressed in volume % (w/v) rather than weight % (w/w); conversion between these units require the availability of bulk density data (Soil Survey Staff 2022).

Method

Analytical methods for water retention are described using six options.

- *Tension*: The capacity of a soil to hold water is often expressed as the water content determined after equilibration of samples, from different depth layers, with water at various suction values (kPa). For practical reasons, high suction values are often controlled by tension in pressure pans. Desorbed or absorbed water between these equilibrium points and the initial saturated condition is measured on a mass basis (W). Water content at selected tension points can be expressed in different measurement units, as follows: 1 bar = 100 kPa (kilo Pascal); 1 bar = 1020 cm H₂O ≈ 1000 cm H₂O; 1 bar = 75.01 cm Hg; 1 bar = 0.9869 atm ≈ 1 atm.
- *Sample type*: Water holding capacity can be derived from: air dry soil material sieved (disturbed samples, fine earth fraction as defined in given laboratory e.g., '1 mm' or '<2 mm'); natural clods; reconstituted clods of soil material; and soil cores taken at a particular depth.
- *Treatment*: For desorption methods, irrespective of the initial moisture status, all samples are first saturated on a tension table by capillarity; subsequently, they are equilibrated at pre-defined water retention levels. Methods that involve wetting of oven dry soils may cause irreversible changes in pore size. To study changes resulting from wetting and drying, the gravimetric water content can be determined after a second equilibration.

In the *absorption methods*, the sample is initially very dry and subsequently equilibrated at the pre-defined retentions levels without prior saturation.

To facilitate desorption by tension or pressure in lab experiments samples have to be in close contact with a supporting surface. Silt or kaolin can be used as an intermediate layer to improve the contact of the sample with a ceramic or porous plate or, in a sandbox for multiple samples.

Clods are generally sealed by a plastic lacquer (e.g., 'Saran F 310 resin'). A flat surface is cut to enhance contact on the ceramic plate or to allow clods to get wet on a tension table. At higher tension levels water is removed from the smallest pores by pressure. Here the original arrangement of soil materials is no longer important; bulk material (sieved <2 mm, air dry, e.g., removed from cores), is placed in retainer rings on the ceramic plate.

Desorption method: Equilibrium values for water desorption can be set by a hanging water column or application of 'over pressure.' A hanging water column with a water manometer is used for lower retention levels (<250 kPa). Hanging water columns should not exceed a length of 10 meters unless vacuum conditions are created and controlled by an Hg-manometer at the 'open end' of the column. With the pressure method, multiple samples are placed on a ceramic plate in a 'pressure pan.' In a series of equilibrium points the above methods are often combined; also, there is an overlap in potential use of the respective desorption methods.

- *Device:* Within the range of equilibrium values for the determination of water holding capacity (e.g., from pF 1.5 to pF 4.2) several 'overlapping' devices can be applied. Saturation is achieved on a tension table at 5 cm tension (5 kPa) applied at the base of the sample. Other equilibrium points may be set in a pressure plate extractor, on a porous plate with burette to measure the volume of desorbed water, or in a kaolin bath. Tensiometers are used with the wind evaporation method.
- *Expression:* Water content may be expressed on a volume basis (volume of water per unit volume of moist soil), on a dry mass basis (mass of water per unit mass of soil solids), or on a wet mass basis (mass of water per unit mass of wet soil). Water holding capacity conventionally is expressed in volume % (weight per volume, w/v) rather than in weight % (weight per weight, w/w). To arrive at volume %, weight% has to be multiplied by bulk density. Where present, a correction needs to be applied for coarse fragments (Soil Survey Staff 2022).

5. Concluding remarks

The methods outlined in this document will be gradually expanded when additional soil properties (e.g., exchangeable cations and acidity; extractable nutrients) are considered in the WoSIS data ingestion and standardization workflow.

Optimistically, the long-term goal is to cover the broader range of soil chemical and physical properties as originally considered for the WISE project (Batjes 2016).

New soil properties will first be served with 'wosis_latest' once at least 30,000 observations (e.g., for gypsum content) have been standardized.

Full data harmonization will first become feasible once results of extensive proficiency testing programmes become freely available, and a common set of 'international' reference methods (e.g., ISO methods or methods developed by GLOSOLAN) has been accepted as the common standard by the international soil community. Availability of such PT data sets will provide the basis for method validation between laboratories with their actual/adjusted method.

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Appendix – Examples of option tables for describing soil analytical methods

Method options for the soil properties served with ‘wosis_latest’ can be consulted using a [dashboard](#) (*note*: third party access is restricted so far).

The coding procedure for CEC (cation exchange capacity) and electrical conductivity are given below, as examples of the coding procedure (Table 3 and 4 respectively).

Table 3. Procedure for coding cation exchange capacity (Example).

<i>Key</i>	<i>Code</i>	<i>Value</i>
sample pretreatment	0	not specified
sample pretreatment	1	sieved over 1 mm sieve
sample pretreatment	2	sieved over 2 mm sieve
Technique	0	not specified
Technique	1	determination by lab procedure
Technique	2	approximated by summation exchangeable cations
reported pH	0	not specified
reported pH	1	reported as (no buffer applied); field pH
reported pH	2	buffered at 7.0
reported pH	3	buffered at 8.0
reported pH	4	buffered at 8.1
reported pH	5	buffered at 8.2
reported pH	6	buffered at 8.3
reported pH	7	buffered at 8.4
reported pH	8	buffered at 8.5
exchange solution	0	not specified
exchange solution	1	not applied
exchange solution	2	1 M KCl
exchange solution	3	1 M NH ₄ -acetate
exchange solution	4	1 M NH ₄ -chloride
exchange solution	5	0.5 M Li-acetate
exchange solution	6	0.5 M BaCl ₂ -TEA
exchange solution	7	0.1 M BaCl ₂ -TEA
exchange solution	8	1 M Na-acetate
exchange solution	9	0.01 M Ag-thiourea
exchange solution	10	0.01 M Ag-thiourea + buffer
exchange solution	11	0.25 M NH ₄ Ac
exchange solution	12	0.1 M CaCl ₂
exchange solution	13	0.5 M Li-chloride
exchange solution	14	0.1 M Li-EDTA
exchange solution	15	Ba-acetate
exchange solution	16	BaCl ₂
Replacement	0	not specified
Replacement	1	not applied
Replacement	2	NaOH
Replacement	3	KCl
Replacement	4	NaCl
Replacement	5	KNO ₃ Ca(NO ₃) ₂
Replacement	6	NH ₄ -acetate
Replacement	7	Ca-Acetate

Replacement	8	Mg(NO ₃) ₂
Replacement	9	Mg(SO ₄) ₂
Replacement	10	NH ₄ Cl
Replacement	11	NaNO ₃
Replacement	12	10% NaCl + HCl
Replacement	13	CaCl ₂
Replacement	14	K-EDTA
Replacement	15	Na-acetate
index cation	0	not specified
index cation	1	not applied
index cation	2	NH ₄ ⁺
index cation	3	Na ⁺
index cation	4	0.01 M Ag-Thiourea ⁺
index cation	5	Ba ²⁺
index cation	6	Li ⁺
index cation	7	Mg ²⁺
index cation	8	Ca ²⁺
bases approximation	0	not specified
bases approximation	1	not applied
bases approximation	2	sum exchangeable bases (NH ₄ Cl, unbuffered)
bases approximation	3	sum exchangeable bases (NH ₄ OAc, buffered pH 7)
bases approximation	4	sum exchangeable bases (BaCl ₂ -TEA pH 8.1)
bases approximation	5	sum of extractable Na, K, Ca, Mg (NH ₄ Cl, 0.05M, unbuffered)
acidity approximation	0	not specified
acidity approximation	1	not applied
acidity approximation	2	exchangeable acidity (KCl extract), (sum of) H, Al
acidity approximation	3	extractable / potential acidity (BaCl ₂ - TEA, pH 8.2)
acidity approximation	4	(sum of) extractable H, Al (NH ₄ Cl, 0.05M, unbuffered)
other exchangeable cations	0	not specified
other exchangeable cations	1	not applied
other exchangeable cations	2	(sum of) extractable Fe, Mn (NH ₄ Cl, 0.05M, unbuffered)
Spectral	1	False
Spectral	2	True

Table 4. Procedure for coding Electrical conductivity (Example).

<i>Key</i>	<i>Code</i>	<i>Value</i>
sample pretreatment	0	not specified
sample pretreatment	1	sieved over 1 mm sieve
sample pretreatment	2	sieved over 2 mm sieve
Solution	0	not specified
Solution	1	water [H ₂ O]
Ratio	0	not specified
Ratio	1	1:1
Ratio	2	1:2
Ratio	3	1:2.5
Ratio	4	1:5
Ratio	5	1:10
Ratio	6	1:50
Ratio	7	saturated paste
Ratio	8	slurry
ratio base	0	not specified
ratio base	1	weight / volume
ratio base	2	volume / volume
Instrument	0	not specified
Instrument	1	electrode
Instrument	2	electrode (field measured)
Spectral	1	false
Spectral	2	True

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