Laboratory Methods for Classification of United Arab Emirates Soils

6

Abstract

Laboratory analyses of soil samples are necessary to verify field soil taxonomic observation. The standard laboratory methods (physical, chemical, engineering, mineralogical, and micromorphological) that form the basis of the operational definitions of the second edition of Soil Taxonomy are described in this chapter, and these methods have been used to support the United Arab Emirates Keys to Soil Taxonomy. Some modifications have been made to fit the hot desert conditions in the United Arab Emirates. The procedures of various determinations such as soil texture, fragments in the soil, moisture content, total pretreatment loss (TPL), loss on acid treatment, carbonate equivalents, gypsum, anhydrite, extractable cations, cation-exchange capacity, exchangeable sodium percentage, saturation percentage, saturation extract analysis, electrical conductivity of the saturated extract, soil reaction or hydrogen ion activity (pH), sodium adsorption ratio, osmotic potential, engineering data (Atterberg limits), AASHTO group classification, Unified Soil Classification System (USCS), percent passing sieves, water retention, bulk density, particle density, porosity, organic carbon, soil mineralogy, clay mineralogy, x-ray fluorescence, thin section study (soil micromorphology) are described in this chapter.

Keywords

Laboratory procedures • Physical • Chemical • Mineralogical • Engineering • Micromorphological

S.A. Shahid et al., *United Arab Emirates Keys to Soil Taxonomy*,
DOI 10.1007/978-94-007-7420-9_6, © Springer Science+Business Media Dordrecht
(Outside the USA) 2014

6.1 Introduction

Laboratory analyses of soil samples are necessary to verify field observation data, to determine properties and characteristics that cannot be estimated accurately by field observations, to help characterize typical profiles, and to properly classify soils. Laboratory procedures are routinely checked and closely monitored to ensure quality control. The standard laboratory methods that form the basis of the operational definitions of the second edition of *Soil Taxonomy* (Soil Survey Staff 1999) are described in the *Soil Survey Laboratory Methods Manual* (Burt 2004) and have been used to support the *United Arab Emirates Keys to Soil Taxonomy*. Some modifications have been made to fit the hot desert conditions in the United Arab Emirates. The following analyses (physical, chemical, engineering, mineralogical, and micromorphological) were carried out. Method codes cited are taken from Burt (2004).

6.1.1 Soil Texture

The texture of a soil is defined according to percentages of sand, silt, and clay in the fraction of the soil that is less than 2 mm in diameter (fine-earth fraction). After pretreatments to remove organic matter and gypsum and to disperse the soil:water suspension, the soil texture was assessed by determining the particle-size distribution. Very coarse (1–2 mm), coarse (0.5–1 mm), medium (0.25–0.5 mm), fine (0.1–0.25 mm), and very fine (0.05–0.1 mm) sands were determined by wet sieving and oven drying the sand fractions retained on each sieve. Coarse silt (0.02–0.05 mm), fine silt (0.002–0.02 mm), and clay (<0.002 mm) were determined from an oven-dried aliquot of suspended soil collected by pipette at specified timings of sedimentation based on Stoke's Law. Results are presented as weight percentages of the fraction less than 2 mm on an oven-dry soil basis, including carbonates and excluding gypsum. Using the proportions of the sand, silt, and clay fractions, the soil texture class was determined using USDA specifications (Soil Survey Division Staff 1993).

6.1.2 Fragments in the Soil

The fraction which is 2 mm or larger in diameter are fragments in the soil. These fragments include rock fragments (i.e., geologic origin), shells, and carbonate nodules, but exclude gypsum crystals, and were determined by a dry sieving procedure. The weights of the material that would not pass sieves with apertures of 5, 20, and 75 mm were recorded.

6.1.3 Moisture Content

A known quantity of air-dried sample was weighed, dried to a constant weight in an oven at 110 ± 5°C, and reweighed. Moisture content of gypsiferous soils that are reported on an oven-dry weight basis was adjusted to remove the weight of the crystal water (methods 2D1-3 and 3D1-3).

6.1.4 Total Pretreatment Loss (TPL)

Carbonates were dissolved by heating the soils to approximately 90 °C in a NaOAc solution (buffered to pH 5). The undissolved soil was then separated from the NaOAc solution, and the soil sample treated with H_2O_2 for oxidation of organic matter. Distilled water was then added to remove soluble gypsum. The residual sample was oven-dried overnight. The loss in sample weight (due to removal of carbonates, organic matter, and gypsum) is calculated and reported as a percentage on a <2 mm basis. This method is based on method 3A1a1a (steps 7.5–7.10) and method 3A1a2a

6.1.5 Loss on Acid Treatment (LAT)

A known quantity of sample was treated with HCl to destroy carbonates. The loss in weight is expressed as calcium carbonate on an oven-dry basis, <2 mm basis (based on method 6E1c). The data are a useful check on the accuracy of the carbonate equivalents analysis.

6.1.6 Calcium Carbonate Equivalents

The quantity of calcium carbonate equivalent is required in soil taxonomy to identify a calcic diagnostic horizon and to determine anhydritic, carbonatic, and gypsic mineralogy classes. The amount of carbonate in the soil was measured by treating the samples with HCl. The evolved CO_2 was measured manometrically using a calcimeter. The amount of carbonate was then calculated as percent $CaCO_3$ equivalent regardless of the form of carbonates (dolomite, sodium carbonate, magnesium carbonate, etc.) in the sample (method 4E1a1a1a1).

6.1.7 Gypsum

Gypsum ($CaSO_4.2H_2O$) content of a soil is a criterion for gypsic and petrogypsic horizons and for particle-size and mineralogical classes at the family level (Soil Survey Staff 1999). Gypsum was measured by reacting a soil extract with acetone, the precipitated calcium sulfate was dissolved in water and EC was measured enabling gypsum percentage to be derived from a calibration curve (method 4E2a1a1a1).

6.1.8 Anhydrite

Anhydrite ($CaSO_4$) is quantified by the difference in two analytical procedures. Anhydrite and gypsum are both extracted and measured by the acetone procedure (method 4E2a1a1a1). Gypsum (but not anhydrite) is quantified by thermal gravimetric analysis, a method that measures the weight loss of a sample by heating it from 20 °C to 200 °C at a rate of 2 °C per minute. The weight of water lost between 75 °C and 115 °C is used to quantify the gypsum based on a theoretical weight loss of 20.9 % (Karathanasis and Harris 1994). Finally, the percent anhydrite is equal to the difference between the acetone and thermal procedures.

6.1.9 Extractable Cations

The soil sample was reacted with 1 N NH_4OAc to replace exchangeable cations. The solution was then separated through centrifugation, and the extracted Na, K, Ca, and Mg were determined using Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) and expressed as meq/100 g (Thomas 1982). For SI conversion the units can be considered as 1 meq/100 g = 1 cmol(+)/kg.

6.1.10 Cation-Exchange Capacity (CEC)

Cation-exchange capacity is a measure of the ability of a soil to hold and exchange cations in a plant available form (e.g., Ca, Mg, Na, NH_4, and K). It is one of the most important chemical properties of soil. This method was based on saturation of cation-exchange sites with Na by “equilibration” of the soil with 1 N NaOAc solution buffered at pH 8.2 and washing with 95 % ethanol. The extraction of sodium was carried out with 1 N ammonium acetate solution (pH 7). The extracted Na was then determined using ICP-AEC and expressed as the CEC in meq/100 g

(USDA 1954). For SI conversion the units can be considered as 1 meq/100 g = 1 cmol(+)/kg.

6.1.11 Exchangeable Sodium Percentage (ESP)

Exchangeable sodium percentage was calculated using the relationship given by the U.S. Salinity Laboratory Staff (USDA 1954) for estimating ESP of saturation extract from SAR for soils with CEC less than 50 meq/100 g: $[100 \times (-0.0126 + 0.01475 \times \mathrm{SAR})/(1 + (-0.0126 + 0.01475 \times \mathrm{SAR})]$.

6.1.12 Saturation Percentage (SP)

The saturation percentage provides indication of available water contents in soil and is also a prerequisite for converting soil solution chemistry data (in water) to soil weight basis. A 250 g air-dried soil sample was saturated with water, taking care to ensure that the resulting soil/water mix met the criteria for a saturated paste. The volume of water and weight of soil were used to calculate the saturation percentage (SP) (method 4 F2).

6.1.13 Saturation Extract Analysis

The solution in the saturated paste was separated from the soil by vacuum extraction and filtration and analyzed for the following characteristics (methods 4F2c1a1 to 4): soluble Ca^{2+}, Mg^{2+}, Na^+, and K^+ were measured by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES). Soluble HCO_3^- by titration with 0.011 N H_2SO_4 in the presence of methyl orange indicator, CO_3^{2-} by titration with 0.011 N H_2SO_4 in the presence of phenolphthalein indicator, and soluble Cl^-, NO_3^-, PO_4^{3-}, and SO_4^{2-} by ion chromatography.

6.1.14 Electrical Conductivity of the Saturated Extract (ECe)

The ECe is a standard representation of an indirect measurement of soil salinity (soluble salts commonly composed of contain Ca^{2+}, Mg^{2+}, Na^+, and K^+, HCO_3^-, CO_3^{2-}, Cl^-, and SO_4^{2-}). The ECe is measured with a standard EC meter and reported as deci Siemens/meter (dS m^{-1}).

6.1.15 Soil Reaction or Hydrogen Ion Activity (pH)

The soil reaction is an expression of the degree of acidity or alkalinity of a soil and is expressed as pH. The pH of saturated soil paste (pHs) is measured with a standard pH meter calibrated using buffer solutions of pH 4.0, 7.0, and 10.0.

6.1.16 Sodium Adsorption Ratio (SAR)

The SAR is a measure of the activities of sodium relative to calcium and magnesium in the soil saturation extract. It is an indirect measure of the equilibrium relation between sodium in the salt solution and exchangeable sodium adsorbed on the soil exchange complex. The SAR was calculated by standard formula (method 4F3b): $\mathrm{SAR} = \mathrm{Na}/[(\mathrm{Ca} + \mathrm{Mg})/2]^{0.5}$, using inputs for the water soluble cations expressed as milliequivalents per liter (meq L^{-1}), and SAR expressed as (mmoles $\mathrm{L}^{-1})^{0.5}$.

6.1.17 Osmotic Potential (OP)

Osmotic potential (atmospheres) provides a guide to the energy a plant must expend to extract water from soil solution; a more saline solution requires more energy. The ECe value was used to estimate the osmotic potential in atmospheres of a solution (USDA 1954) as $\mathrm{OP} \approx 0.36 \times \mathrm{ECe}$ where ECe is expressed as dS m^{-1}.

6.1.18 Engineering Data

The soils can be analyzed for their Atterberg limits and classified for engineering purposes according to the Unified Soil Classification System (USCS) and the system adopted by the American Association of State Highway and Transportation Officials (AASHTO).

6.1.19 Atterberg Limits

Atterberg limits are determined on the fraction less than 0.4 mm in size. Atterberg limits are soil water contents that define the upper and lower boundaries of the plastic state for a given soil. They provide basic measures of the engineering behavior of fine-grained soil and include the plastic limit (Wp) and liquid limit (Wl) and plasticity index (Ip), which is the difference in water content between the liquid limit and plastic limit. The plastic limit is determined by rolling a soil sample into 3.2 mm diameter thread. This thread is broken into pieces, squeezed together again, and rolled until its water content is reduced to a point at which the thread crumbles and can no longer to be pressed and rolled. At this point the water content of soil is reported as the plastic limit. Liquid limit is determined by performing observations in which a portion of sample is spread in a brass cup, divided in two by a grooving tool, and then allowed to flow together by repeatedly dropping the cup in a uniform manner in the standard mechanical liquid limit device. The multipoint liquid limit procedure requires the performance of three or more observations over a range of water contents and the plotting of data from the trials to obtain the relationship from which the liquid limit is determined (ASTM D 4318–2004).

6.1.20 AASHTO Group Classification

The AASHTO group classification is based upon particle-size distribution and Atterberg limits. The system is used for geotechnical engineering purposes to make general interpretations relating to the engineering performance of soils for construction and load bearing purposes.

The system identifies two general groups:

- Granular materials having 35 % or less, by weight, particles smaller than 0.074 mm in diameter; and
- Silty-clay materials having more than 35 %, by weight, particles smaller than 0.074 mm in diameter.

These two groups are further subdivided into seven main subgroup classifications. The group and subgroup classifications are based on estimated or measured grain-size distribution and on liquid limit and plasticity index values derived from measured values of samples taken from typical profiles.

The AASHTO group and subgroup classifications may be further modified by the addition of a group index value. The empirical group index formula was devised for approximate within-group evaluation of "clayey granular materials" and "silty-clay" materials. The dominant AASHTO group classifications for UAE soils and their inherent characteristics are:

A-3: Silty sand with a maximum of 10 % silt. Typically nonplastic. These soils are suitable for structural and general backfill material.

A-2-4: Silty sand with a maximum silt content of 35 %. The material is either nonplastic or has a low plasticity. The high sand content allows the soils to be classified as structural fill material and hence used for general engineering purposes (herein defined as construction and earthworks purposes).

A-1-b: These soils contain stone fragments and up to 25 % fines (very fine sand, silt, and clay). They are either nonplastic or slightly plastic. They are suitable as general backfill for engineering purposes.

A-1-a: These soils are similar to those classified as A-1-b but are limited by the high percentage of stone fragments.

6.1.21 Unified Soil Classification System (USCS)

The USCS is a system for classifying mineral and organic soils for engineering purposes based on particle-size characteristics, the amounts of various sizes, and the characteristics of the very fine particles assessed by the liquid limit and plasticity index.

The USCS identifies three major soil divisions:

(i) coarse-grained soils having less than 50 %, by weight, particles smaller than 0.074 mm in diameter;
(ii) fine-grained soils having 50 % or more, by weight, particles smaller than 0.074 mm in diameter; and
(iii) highly organic soils that demonstrate certain organic characteristics.

These divisions are further subdivided into 15 basic soil groups. The major soil divisions and basic soil groups are determined on the basis of estimated or measured values for grain-size distribution and Atterberg limits. The various groupings of the USCS classification have been devised to correlate with the engineering behavior of soils. This correlation provides a useful first step in any field or laboratory investigation of soils for engineering purposes. The classification and its interpretation are applicable in the design of foundations for a range of structures. The dominant USCS classifications for UAE soils and their inherent characteristics are:

SM: Silty sand with a maximum of 30 % fine material that is nonplastic. These soils contain various cementing agents, such as calcium carbonate, gypsum, clay, and silt. The degree of cementation can range from noncemented to strongly cemented in both the horizontal and vertical directions (note that within the soil profile various horizons of SM material can exhibit differing degrees of cementation). From an engineering point of view, the soil would classify as SM.

SP-SM: Silty sand with fine material limited to between 5 % and 12 %. These soils contain various cementing agents, such as calcium carbonate, gypsum, clay, and silt.

The soils are relatively permeable due to poor gradation. These soils are suitable for general engineering purposes.

SP: Poorly graded or gap graded fine to medium sand with little or no fines; not recommended for engineering purposes; difficult to compact; suitable for highly permeable fill material. This is the most common category for UAE soils.

SW: Well graded siliceous sand with little or no fine material; very good material for construction and general engineering purposes. Rarely found in the study area.

CL: Inorganic clay. Typically mixed with sands with low plasticity and Wl <50 %. These soils are not common in the UAE. This material is considered unsuitable for general engineering purposes.

MH: Inorganic silts with high plasticity. Very rare in the UAE. This material is considered unsuitable for general engineering purposes.

CH: Inorganic clays with high plasticity. Very rare in the UAE. This material is considered unsuitable for general engineering purposes.

6.1.22 Percent Passing Sieves

An air-dried soil subsample can be sieved through a nest of sieves, mesh numbers 4 (4.75 mm), 10 (2 mm), 40 (0.42 mm), and 200 (0.074 mm), and shaken mechanically. The material retained on each sieve can be weighed, and calculations can be made using a standard formula (USDA-NRCS 1995) to represent percent soil passing the respective sieves.

6.1.23 Water Retention (WR)

Water retention is defined as the soil water content at a given soil water suction. By varying the soil suction and recording the changes in soil water content, a water retention function or curve is determined. A pressure membrane apparatus method (3C1ae) is used to determine water retention (percent) at 1/10, 1/3, and 15 bars, respectively, for sieved, <2 mm, air-dry soil samples. The following are SI conversions:

15 bar = 1,500 kPa
1/3 bar = 33 kPa
1/10 bar = 10 kPa

6.1.24 Water Retention Difference (WRD)

The calculation of the WRD is considered the initial step in the approximation of the available water capacity. The WRD can be calculated as the difference in water retentions at 33 and1,500 kPa, converted to units of mm by multiplying by bulk density and to a whole soil basis by multiplying by the proportion of soil that passes a 2 mm sieve (method 3D5a).

6.1.25 Bulk Density (BD)

Bulk density is the weight of soil (oven dry) per unit bulk volume of soil (volume occupied by soil solids and pore spaces). The bulk density of a soil indicates the pore space available for water and roots. For example, a bulk density of 1.6 g/cm^3 and higher can restrict water storage and root penetration. Bulk density samples can be collected in standard steel cores of known volume and then oven dried. The loss in weight on drying is recorded and bulk density calculated by standard formula (method 4A3a). Where bulk density is not measured but is required for calculating other properties, an estimated value (BDcalc) is derived from the sand content (TOTALSAND%) based on the following equation derived from UAE soil surveys data: $\text{BDcalc} = 0.0098 \times \text{TOTALSAND\%} + 0.657$

6.1.26 Particle Density (PD)

The particle density provides an understanding of the basic component particles of the soil. The method requires using a pycnometer to estimate the volume of water or an inert gas (e.g., He) displaced by a known weight of soil, ensuring that all air is removed from the mixture (method 3G). Where particle density has not been measured, a value of 2.65 g/cm^3 has been assumed as the basis of other calculations.

6.1.27 Porosity

The percent volume of bulk soil that is occupied by pore spaces (the space filled by air or water) is called porosity. Porosity is calculated by using a standard formula of $100\ (1-\text{BD}/\text{PD})$, where BD and PD represent the bulk density and particle density, respectively.

6.1.28 Organic Carbon (OC)

Organic carbon was determined by oxidizing the sample by flash combustion followed by reduction of gases produced. The product gases formed by reduction are separated chromatographically on a column using helium as carrier gas and detected using thermal conductivity detector (TCD). The percent organic carbon is converted to percent organic matter by multiplying by a factor of 1.724 (Burt 2004).

6.1.29 Soil Mineralogy (XRD Analysis)

The x-ray diffraction (XRD) is by far the most powerful technique to identify mineral species in soils. Whole soil mineralogy from the control sections of the typical soil profiles of identified soil families was determined by using a standard x-ray diffraction technique. The soil material was ground with an agate pestle and mortar and lightly pressed in an aluminum sample holder.

X-ray patterns were recorded using Cu-Kα radiation, using variable divergent, receiving and scattering slits. The step size was 1°2θ/min. The procedure is similar to method 7A2i (Burt 2004). A Philips x-ray diffraction model PW/1840, with Ni filter, Cu-Kα radiation ($\lambda = 0.154$ nm) at 40 kV, 55 mA, and scanning speed 0.02°/s, was used. Diffraction peaks between 2 and 60°2θ were recorded. The corresponding d-spacing and the relative intensities (I/Io) were calculated and compared with standard patterns (ASTM cards).

6.1.30 Clay Mineralogy (CM)

Clay (particles <2 μm) is a reactive (high surface area) soil component that controls physical, chemical, and nutritional behavior (nutrient storage) of soil and therefore affects agricultural potential. The clay-size material was separated from soil after the cementing material (organic matter, gypsum, and soluble salts) was removed for dispersion of clay in a soil suspension. Sedimentation and sampling for clay was based on Stoke's Law.

To resolve the clay mineral species, the clay was treated with K, K-heating at 300 °C and 550 °C, Mg, Mg ethylene glycol, and Mg-glycerol solvation. The treated clay samples were mounted on glass slides, and x-ray diffraction patterns were recorded using XRD equipment as above. The XRD patterns were interpreted for mineral species, comparing with standard reference patterns and measurements (ASTM cards). An additional test was made to confirm the presence of kaolinite using the Lim test (Lim et al. 1980). In this test the 7.2 Å peak of first order kaolinite shifts to 11.2 Å; however, second order chlorite peak remained at 7.00 Å. Based on the peak heights of the minerals, area under the peaks, and sharpness, the amounts of each mineral was estimated semiquantitatively (%).

6.1.31 X-ray Fluorescence (XRF)

A 1 g soil sample was ignited at 1,100 °C and dissolved in 9 g of lithium tetraborate and lithium borate (66:34) and 0.5 g of lanthanum oxide flux to form a disc for x-ray fluorescence analysis. The information is presented as percentages of elemental oxide.

6.1.32 Thin Section Study (Soil Micromorphology)

Undisturbed soil samples were collected in standard Kubiena boxes. Samples were dried and impregnated with a mixture of resin, acetone, and a hardener. On setting, the impregnated blocks were cut into small sections with a diamond saw and lubricant. The face of the section was polished with aloxite grit (6 μm) and then with diamond paste (6 and 3 μm) with Hyprez fluid as lubricant. It was then cleaned ultrasonically with petroleum spirit to remove residue from pores. The polished side was then stuck with adhesive on a clean slide and clamped to allow the adhesive layer to cure. The excess of the block was cut off with a diamond saw. The section was then ground initially to approximately 50 μm thickness on a lapping and polishing machine, then to 30 μm by hand grinding. The quartz grains were viewed in the microscope to observe the onset of gray birefringent color under crossed polars. Final polishing was completed with 6 and 3 μm diamond pastes followed by ultrasonic cleaning. Thin sections were studied on a petrographic microscope fitted with a digital camera, and the different components (groundmass, microstructure, mineral composition, pedofeatures, and pore space) were described using the procedures of Bullock et al. (1985) through point counting. Photographs of important features were captured using a digital camera fitted to the petrological microscope.

References

Bullock, P., Fedoroff, N., Jongerius, A., Stoops, G., Tursina, T., & Babel, U. (1985). *Handbook for soil thin section description*. Albrighton: Waine Research.

Burt, R. (2004). *Soil survey laboratory methods manual*. Soil Survey Investigations Report No. 42, Version 4.0. Nebraska: U.S. Department of Agriculture, Natural Resources Conservation Service, National Soil Survey Center.

Karathanasis, A. D., & Harris, W. G. (1994). Quantitative thermal analysis of soil minerals. In J. E. Amonette & L. W. Zelazny (Eds.), *Quantitative methods in soil mineralogy* (pp. 360–411). Wisconsin: Soil Science Society of America Miscellaneous Publication SSSA.

Lim, C. H., Jackson, M. L., Koons, R. D., & Helmke, P. A. (1980). Kaolins: Sources of differences in cation exchange capacities and cesium retention. *Clays and Clay Minerals, 28*, 223–229.

Soil Survey Division Staff. (1993). *Soil survey manual* (U.S. Department of Agriculture Handbook, No. 18). Washington, DC: U.S. Government Printing Office.

Soil Survey Staff. (1999). *Soil taxonomy: A basic system of soil classification for making and interpreting soil surveys* (U.S. Department of Agriculture Handbook 2nd ed., No. 436). Washington, DC: U.S. Government Printing Office.

Thomas, G. W. (1982). Exchangeable cations. In A. L. Page, et al. (Eds.), *Methods of soil analysis, part 2: Chemical and microbiological properties* (Agronomy monograph No. 9, 2nd ed., pp. 159–166). Madison: American Society of Agronomy, Inc./Soil Science Society of America, Inc.

United States Department of Agriculture, Natural Resources Conservation Service (USDA-NRCS). (1995). *Soil survey laboratory information manual* (Soil survey investigations report, No. 45, Version 1.0). Nebraska: National Soil Survey Center.

USDA. (1954). *Diagnosis and improvement of saline and alkali soils* (U.S. Department of Agriculture Handbook, No. 60). Washington, DC: U.S. Department of Agriculture.

Sandy deserts with some vegetation cover are attraction to grazers