Validation of gravimetric method for determination of clay in soil

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Abstract: The requirements for method validation are taken from the method ISO 11277:2020 Soil quality - determination of particle size in soil - by sieving and sedimentation method, Annex C table C.1 [1]. The validation was performed on soil samples and on soil reference material (old PT scheme). Validation includes checking the volume of the pipette i.e., calibration of the pipette, correction for the contribution of the dispersing agent, repeatability (RSDr), reproducibility (RSDR), recovery (R), checking bias from the used lab equipment and the bias from purity of use chemicals. As a final part of the validation, the combined measurement uncertainty (Uc) and expanded measurement uncertainty (U) with a confidence level of 95% were calculated. The pipette calibration error is 2.40%. The obtained results are RSRr = 1.38%, RSDR = 13.21%, Recovery = 100.66%, laboratory equipment bias is 2.66%, bias from used chemicals is 0.17%, combined uncertainty is 5.08% and expanded measurement uncertainty is 10.17% by gravimetric determination of clay content, using the pipette method.

Keywords: soil composition, clay, method validation

1. Introduction

The mechanical (textural, granulometric) composition of the soil is a key parameter in the study of genesis, evolution, properties and systematics. In the processes of pedogenesis by the effect of physical, chemical, physico-chemical and biological decay and decomposition, mechanical elements (primary particles) are created from the parent substrate. Mechanical elements are elementary (primary) soil particles of different sizes that are under the influence of mild force (under a finger or a jet of water) and cannot be divided or crushed. The percentage of the representation of mechanical elements of different dimensions is called the mechanical composition of soil. From an agronomic point of view, the best soils have a ratio of sand-clay powder fractions of 35%-40%, 35%-40% and 20%-30%. In the processes of connecting primary particles - mechanical elements (sand, powder and clay) create larger secondary particles – micro and macro structural aggregates.

The clay fraction is created by synthesis from the products of chemical decomposition of primary ones, as aluminosilicate or is inherited from the parent substrate, and it is colloidal in nature and has a huge specific surface with which all the essential properties of the soil are connected. With the increased content of the clay fraction, the share of capillary pores and the height of the capillary rise of water increase in the soil.

The mechanical composition has a great influence on other physical, water-physical, physical-mechanical, chemical, biological and ecological-production properties of soil. The water, air, heat and nutrition regime of the soil depend on the mechanical composition. The choice of mechanization for its cultivation depends on the mechanical composition of the soil and on the use of the land.

The clay content contributes significantly to the compaction of the soil, for this reason it is very important to determine as accurately as possible the proportion of clay in the mechanical composition of the soil. Different methods are used to determine the mechanical composition of the soil: fractionation using a series of sieves (for the skeleton and larger fractions) and the pipette method (sedimentation in still water). Large fractions are removed with a set of sieves, while the clay content is determined by dispersion and pipetting.

2. Instructions

Air-dried soil or soil dried in a dryer at 105°C is divided into smaller portions using a Johnson divider, homogenized, and sieved through an ISO sieve with a diameter of 2 mm. Depending on the clay content, 10-30g of soil sample is weighed. The measured mass of the sample is transferred to a vessel with a volume of 500 ml, moistened with 20ml of water, and 30% H_2O_2 is added in portions to remove organic matter. After the reaction is complete, 0.1M HCl is added to remove carbonate interference. After the reaction is finished, add water up to a volume of 500ml and 1-2 drops of aluminum sulfate (may or may not). The next day, the clear part above the solid phase is pumped out with a vacuum system and 25 ml of dispersant and 225 ml of water are added over the solid phase. The suspension is brought to a boil and allowed to boil for 5 minutes and cooled at room temperature. Place a set of sieves with openings of 2 mm, 0.63 mm, 0.063 mm and sift the sand fractions by wet sieving, while collecting the suspension in the sedimentation vessel, taking care that the total volume does not exceed 500 ml. When the 500ml beaker, i.e. the sedimentation vessel, is filled up to the line, stir the suspension. Keep the sedimentation vessel in the middle with a constant temperature and monitor the dependence of the particle size on the temperature so that it is known at what time fractions of dust and clay are pipetted at a depth of 10 cm. After a certain time, the pipetted aliquots are quantitatively transferred into fixed Al containers, and the composition is evaporated to a smaller volume and dried in an oven at 105°C to a constant mass. The working temperature was 25°C where the clay pipetting was done after 6 hours 52 minutes and 37 seconds.

Precision in terms of repeatability and reproducibility is also expressed through the relative standard deviation (RSD, %). The calculation of the combined measurement uncertainty (Uc) includes the contribution from the impurity of chemicals, the error of measuring devices and laboratory equipment, RSDr, RSDR, Recovery ie bias according to equation (1) [2-6]:

$$Uc\,(\%) = \sqrt{(Uimpurity^2 + bias^2 + RSDr^2 + RSDR^2 + + Usampling^2)} \quad (1)$$

The summarized results are given in Table 1.

Table 1. Evaluation of obtained validation results

Validation parameter	Experimental results [%]	ISO 11277:2020 Annex C, Table C.1 and AOAC [1, 2] [%]
RSDr	1.38	5.23
RSDR	13.21	25.93
Recovery	100.66	98-102
U impurity	0.17	/
bias	2.66	/
Uc	5.08	/
U	10.17	/

3. Conclusions

Based on the obtained validation results of the method, it can be concluded that this procedure satisfies the given criteria for validation in determining the clay content by the gravimetric method with sedimentation. The obtained validation parameters meet the criteria according to the AOAC specifications for accuracy, repeatability and reproducibility.

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