Grain size distribution for silt and clay by means of laser diffraction

L'analyze granulometrique des agiles et des silts par la diffraction des rayonnes du laser

O.Muri, J.V.Holm & L.Hamre – *NOTEBY AS, Oslo, Norway*

ABSTRACT: Grain size distribution of soils in the silt and clay fractions has traditionally been tested by hydrometer for geotechnical purposes at the NOTEBY laboratory. This laboratory was some years ago fitted with a laser diffraction instrument of type Beckman Coulter that is used for determination of the particle dimensions of silt and clay soils. This paper gives the results of parallel testing between testing of soil materials in the silt and clay fractions by means of laser diffraction and hydrometer testing. The comparability between the test methods is in general good, and the laser diffraction method is recommended for use in grain size analyses.

RÉSUMÉ: La courbe granulometrique est traditionnellement determineé dans les laboratoirs de NOTEBY par la méthode de hydromètre. Les laboratoires ont eté recemment équipés avec un diffractomètre du laser, de type Beckman Coulter, qui permets la determination des dimensions des partiqules des sols argileux et silteus. L'article present les resultats de l'analyse granulometrique sur echantillons des sols coherents avec la méthode de diffractomètre du laser et avec la méthode de hydromètre. Les resultats obtenues par les deux méthodes montrent une bonne concordance et la méthode à diffractomètre du laser est recommandeé pour l'analyse granulometrique.

1 INTRODUCTION

Grain size distribution of soils in the silt and clay fractions has traditionally been tested by hydrometer for geotechnical purposes at the NOTEBY laboratory and nationwide. For the special purpose of testing cement grouts at a major tunnel project at site, a field laboratory was fitted with a laser diffraction instrument. Now, as the project has ended the instrument is used also for testing silt and clay soils for geotechnical purposes.

By taking a new method into use for deciding the 2 µm limit of the clay content, it was necessary to establish experience with the comparability between the two methods. An internal project was established to gain experience and to document correct input parameters and preparation procedures.

This paper gives the results of parallel testing between testing of soil materials in the silt and clay fractions by means of laser diffraction and hydrometer testing.

2 INSTRUMENT AND PRINCIPLES

A Beckman Coulter LS230 Particle Size Analyzer (1997) fitted with a large volume module was found to comply with our needs with respect to flexibility in the software and practical performance. It allowed us to measure particle sizes in the size range of 0,04 µm to 2000 µm in one single scan. The principle of operation of the LS230 is as follows:

The soil sample (normally < 125 µm) is ultrasonically dispersed in water and flows through a detection cell where the coherent beam of light passes through the water suspended sample. The laser beam scattering is related to the size of the particles – the smaller particles cause a greater amount of scattering.

A series of 132 optical detectors are used to register the diffraction of the laser light. The method uses a patented technology to carry out and calculate the diffraction tests. This technology, Polarization Intensity Differential Scattering (PIDS) uses three wavelengths of light, filtered for polarization in the vertical and the horizontal planes. A total of 42 measurements are made at six scattering angles and three wavelengths, each at two polarizations. The combination of multiple wavelengths and two polarizations provides information that differentiates between sub-micron particle sizes and dramatically increases resolution.

The performance of the Beckman Coulter LS230 Particle Size Analyser relative to traceable standards indicates that the instrument can be used with confidence, and that the particle size distributions it produces accurately reflect the sample material.

NOTEBY is participating in a Laser Diffraction Proficiency Testing Scheme (LDPTS). The Beckman Coulter UK Ltd. administrates it, with participants from users of Coulter and Malvern instruments in Europe, UK and USA. It enables us to compare our performance with other laboratories and to demonstrate to customers and regulatory bodies, on an international basis, the validity of our results. We have so far obtained satisfactory results. Questionable results have occurred, but has mainly been possible to trace and been a source to improvement of routines. It is important to be aware that the results produced may differ, depending on the type of instrument used. It is therefore necessary to inform users of the data produced, the type and manufacturer of the instruments and bare this in mind when comparing the results.

The comparison between the laser diffraction analyses and the traditional hydrometer analyses has been carried out on a total of 37 tests. The tests are from 15 different locations, and the classification according to the hydrometer analyses are as follows:

Table 1. Database

Description	Clay Content	No. of tests
Silt	< 5 %	5
Silt, clayey	5 – 15 %	11
Clay, silty	15 – 30 %	10
Clay	>30 %	11
Total		37

The most important issues for these tests are the preparation of the soil specimens before testing, and the selection of optical model for use in the laser diffraction analyzer.

The hydrometer analyses were carried out as described by Lambe (1969) and State Roads and Highways Authority (Statens Vegvesen 1996), and the laser diffraction analyses were carried out according to ISO 13320-1 (1999) and Stark (1993).

3 PREPARATION OF THE SOIL SPECIMENS

3.1 *General*

Parallel testing with LS230 were carried out on samples from soil investigation programs where hydrometer analyses were carried out. The sample specimens, approximately 200 g, were homogenized by stirring before quartered to suitable sizes for the analyses. In a few cases, dried material was used. In those cases, the specimens were crushed and homogenized in a porcelain mortar in accordance with traditional preparation for hydrometer analyses. The tests were carried out at least twice on each sample.

Further preparation followed different procedures, depending on weather the material was characterized as silt or clay.

3.2 *Preparation of Clay*

For clay materials, the laser diffraction analyses require a very small amount of material. For this material, it was found appropriate to prepare a larger specimen, and extract part specimens from this. The procedure was as follows:

1 g homogenized specimen, 10 drops dispersion solution and 10 ml water was measured into a beaker. The beaker was exposed to ultra sound for approximately 2 minutes, and thereafter continuously stirred by hand. Small specimens of the rotating suspension were extracted from the solution by a pipette, and the whole solution was then added to the sample module. Several extractions were made, until indications on the instrument showed sufficient admixture.

3.3 *Preparation of Silt*

The measurement of particles of silt size, requires approximately 0.25 g material. 5 drops dispersion solution and 5 ml water was measured into a beaker and exposed to ultra sound for approximately 2 minutes, and thereafter continuously stirred by hand. For silt materials it was, due to danger of separation of the particles, found necessary to add the total of the prepared specimen to the sample module.

4 OPTICAL MODEL

The use of laser diffraction for grain size distribution measurements demands an optical model for the given material when using Coulter instruments. The model demand input values for obscurance and the refraction index. The parameters for known minerals and chemical compositions are available. However, natural clays may consist of different minerals with different optical properties. A suitable model must therefore be based on an "average composition". The model used for the material tested in this test program has been an optical model called "Clay", with refraction index 1,57 and obscurance (imaginary part) 0,01.

This model corresponds with the clay material's mineral composition and the optical characteristics of these minerals. Results of the comparison between the hydrometer test and laser diffraction with optical model "Clay" are shown for two typical grain size compositions in Figure 2.

From this comparison it is seen that the laser diffraction analyzes gives somewhat lover clay content than the traditional hydrometer test. Other alternative optical models were also investigated during this test program. A better correlation between the two methods for measuring clay content was found to be a model with a refraction index of 1.51 and an imaginary part of 0,01.

However, as uncertainties connected to the traditional grain size test methods perhaps are larger than for the laser diffraction due to for example variation in particle shape and density, and phenomena such as flocculation the theoretical model valid for the actual mineral composition of typical clay materials was chosen in this documentation.

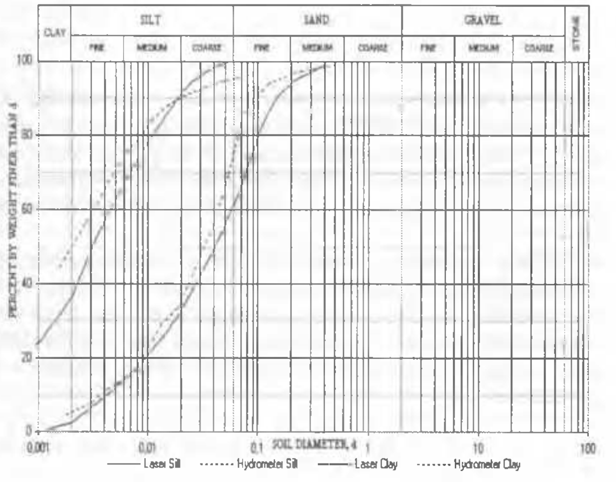

Figure 2. Comparison between hydrometer test and laser diffraction

5 MAXIMUM GRAIN SIZE

Determination of grain size on soil materials can be made by several methods. For particle sizes larger than 125μm the most common method is sieve analysis. Tests on fractions between 125 μm and 2,0 mm showed that the laser diffraction gives larger diameters than the sieve analysis. This was expected, as the laser measures average of all diameters, while the sieve analysis measures only diameters normal on the longest axis.

Considering the widespread use of sieve analyses, and the relative large discrepancy between the two methods for the given grain size range, the upper bound grain size was set to 125 μm in this study.

6 INFLUENCE OF SALT CONTENT

In order to investigate the effect of salt content on the grain size measurements with laser diffraction, several analyses were carried out, increasing the salt content to 500 times the salt content for a clay sample with 60% water content and water with salt content as in seawater. The results of the 6 tests are shown in Figure 3, and the conclusion is clearly that salt content does not influence the laser diffraction test results.

7 RESULTS

The correlation of the results between the hydrometer analyses, and the laser diffraction analysis is illustrated by the figures 4 to 7. The figures illustrate the correlation between the methods for grain sizes divided into four categories: < 2 μm, < 6 μm < 20 μm and < 60 μm.

The basis for the test program is 37 tests carried out on soil samples with varying grain size compositions. Material with grain size larger than 125 μm was extracted before the tests were

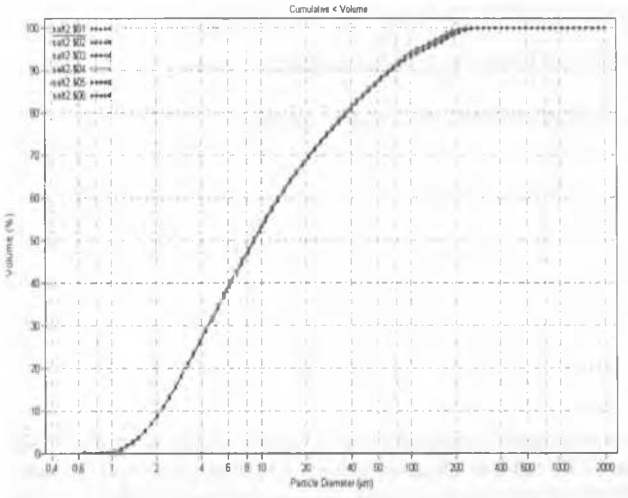

Figure 3. Effect of salt on grain size measurement

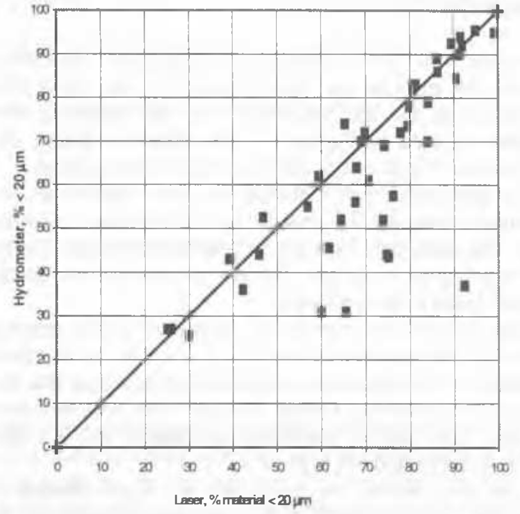

Figure 6. Material < 20 µm

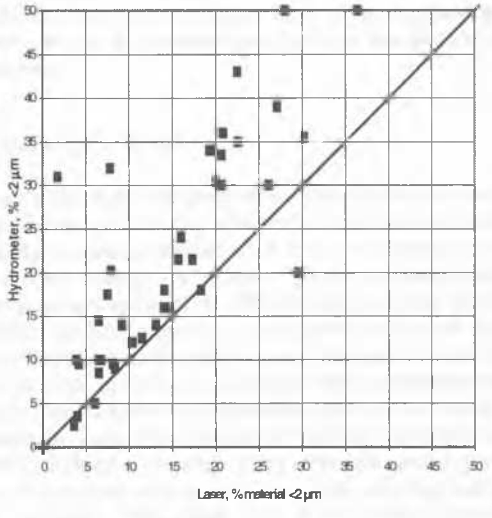

Figure 4. Material < 2 µm

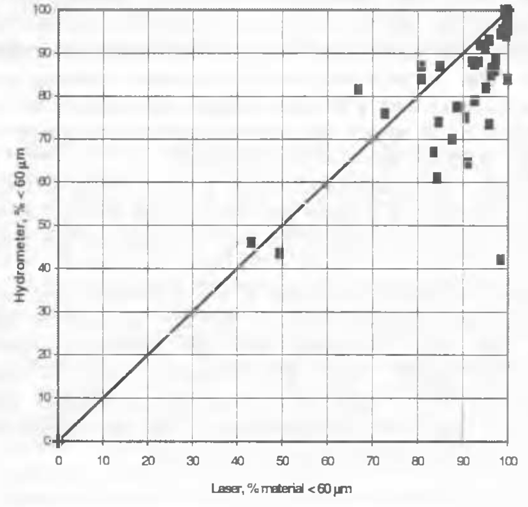

Figure 7. Material < 60 µm

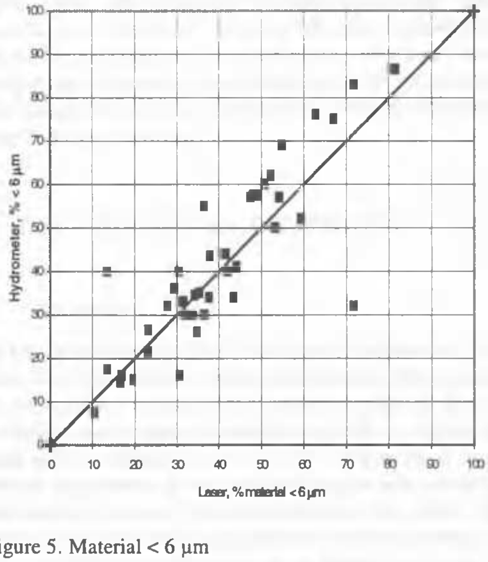

Figure 5. Material < 6 µm

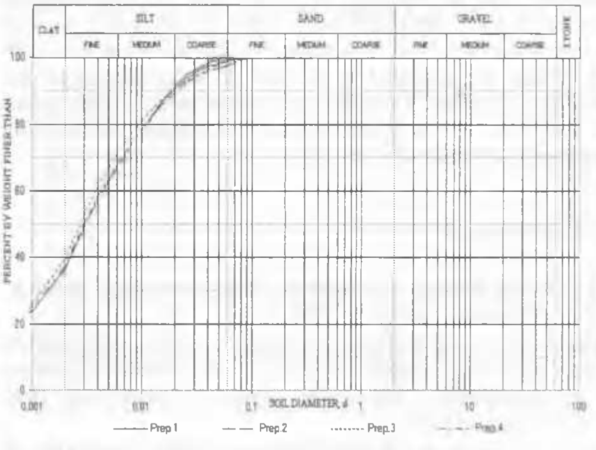

Figure 8. Test repetition, four tests

carried out. The preparation of the tests was carried out according to the procedures described previously, and the optical mode "Clay" was used as basis for the laser diffraction analyses.

8 EVALUATION

The experience with laser diffraction as a method for measuring grain size on silt and clay materials is generally very good. The test method is fast and the repetition of the test results is very high as demonstrated in Figure 8. The effect of drying the material does not influence the grain size distribution curves.

There is good agreement between the laser diffraction test and hydrometer tests for the coarser part of the testes material (<125μm). The agreement between the methods for more clayey materials is not quite so good. The reasons for this are many, and some are listed in the following.

The shape of the clay particles is dependent on the mineral composition of the soil and can be flaky (silicates) or spherical (round, quartz). The hydrometer test procedure presumes that the single grains are spherical. Grains that are flaky will sediment slower, hence they will be registered as smaller, and the clay content will be registered too high.

Using the hydrometer test procedure, the grain density is usually set to 2.65 tonnes/m^3 in the calculation procedure. In clays containing organic material, this material will sediment slower, as the grain density is lower. Hence the clay content will be registered too high in the hydrometer analyses.

Another well-known phenomena that can be observed using the hydrometer test, is that the grain size curve becomes flat in the upper part, and that the transition to the sieve analyses results is abrupt. This is observed by the aerometer showing an increasing tendency during the first minutes. This is likely due to the air content in the suspension, and will lead to a measurement of too high content of soil with coarser grains.

Clay particles with diameter less than 1 μm act like colloids (does not sediment). It is uncertain weather this will influence the measurements in the boarder for clay with diameter less than 2 μm.

Based on the above evaluation of the hydrometer test, we consider the optical model used to be relevant for the grain size analyses with laser diffraction with the Beckman Coulter Analyzer. This model, "clay", with refraction index 1.57 and imaginary part 0.01 is valid for a typical clay mineralogy and was therefore used in this documentation of the laser diffraction test, and is also recommended used in the future.

9 CONCLUSION

The conclusion drawn is based on presumptions of the use of instrument and procedures. We will from the test program, conclude that the laser diffraction method compares well with the hydrometer analyses. Discrepancies found for material with D = 2 μm are regarded to be due to uncertainties in the hydrometer analyses. The test repetition is generally very good, and the effect of salt is negligible. Recommended maximum grain size was found to be 125 μm.

10 REFERENCES

ISO 13320-,1 Particle size analysis - Laser diffraction methods, Part 1: General principles, 1999-11-01

Stark, U., Univerität Weimar/Deutschland: Using Coulter LS130 laser diffraction analyses for testing and research work on building materials, Sonderdruck aus ZGK International 1993 Heft 8-seite 458-462.

Lambe T. William and Robert Whitman - 1969, John Wiley & Sons Inc.: Soil testing for engineers, The Massachusets Institute of Technology, Chap. IV, p. 32-39, Hydrometer analysis.

Statens Vegvesen, Laboratoriehåndbok 014, 1996, Vedlegg 1 jordartsklassifisering side 1-2

Laser Diffraction Proficiency Testing Scheme (LDPTS), Beckman Coulter UK Ltd.