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Optical methods in the grain-size analysis of fine-grained sediments

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Optical methods now dominate the grain-size analysis of fine-grained sediments, because of their rapidity. The growing application of optical methods leads to statistical comparison of their results against those from sedimentation methods. Here, the Kołmogorow-Smirnow non-parametric test is demonstrated. Most agreement exists in the 50–20 and 20–6 μm fraction. In order to compare the optical and the combined methods, correlation ratios were calculated for all commonly used grain-size fractions. Regression equations were established for the 50–20 μm fraction.

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INTRODUCTION

Grain-size analysis of fine-grained sediments has been conducted traditionally mainly by sedimentation methods (Rühle, 1973), as only these have allowed determination of the grain-size of fine-grained sediments. For the grain-size analysis of silts Casagrande's areometric method modified by Prószyński, has mainly been used, or the pipette method. However, these methods are time-consuming and laborious, and so other methods of grain-size analysis of fine-grained sediments have been developed (Kasza, 1992; Issmer, 1992, 1994; Nicieja, 1996; Konert and Vandenberghe, 1997; McTainsh *et al.*, 1997; Beuselinck *et al.*, 1998).

These new methods include optical ones, in particular the diffraction method, on the basis of which devices such as the *Analysette 22-E* Laser Particle Sizer manufactured by *Fritsch GmbH* operate. Some authors (Kasza, 1992; Nicieja, 1996; Konert and Vandenberghe, 1997) are imprecise in calling the diffraction method "the laser method". These devices which measure the size of very small particles commonly use Fraunhofer diffraction, which requires the application of a beam of parallel light rays, a laser being used as the light source. Another device operating on the basis of the optical method is the *Analysette 20* Scanning Photo-Sedimentograph manufactured by *Fritsch GmbH.*

OPTICAL DIFFRACTOMETRY METHOD

Light diffraction is a deviation from the linear dispersion of light in the vicinity of non-transparent bodies. On a screen behind the obstruction bending the light, instead of observing a distinct barrier between the light and the shadow, a pattern of diffraction striae, i.e. a series of dark, light or coloured lines with the same intensity of light can be observed. Light diffraction is also observed when a light ray passes through small openings or near the barriers of transparent or non-transparent bodies.

Fraunhofer diffraction occurs when a beam of parallel rays, i.e. a beam from a source at infinite distance hits a curved surface. The result of the curvature is analysed on a screen, also at an infinite distance. In practice, the Fraunhofer diffraction boils down to analysing the curvature of parallel beams obtained by means of converging lenses, when a light source is placed in the lens' focal length. Similarly, bent parallel beams are concentrated in the converging lens' focal area (Skorko, 1979). In the case of *Analysette 22-E*, this is a beam of rays emitted by a helium-neon laser. In the case of Fraunhofer diffraction, the difference in optical paths from the opening to the observation points does not depend on the distance from the diaphragm to the observation point, but on the direction at which the opening is observed. Therefore, the distribution of light intensity does not change with distance.

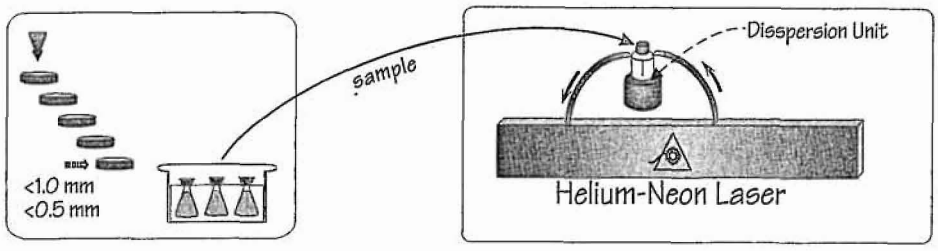

Fig. 1. Mode of operation of the *Analysette 22-E* Laser Particle Sizer

$$R \gg d^2 l^{-1}$$

in which: R — the distance from the opening to the observation point (viewing point), d — opening-specific dimension, e.g., width of slit or diameter of circular opening, l — length of light wave.

The *Analysette 22-E* Laser Particle Sizer is based on Fraunhofer optical diffraction. A parallel beam of light, emitted by a helium-neon laser, scans a special measuring cell, to which a sample in the form of a suspension has been inserted. The sample is inserted into a measuring cell directly from the suspension dispersing unit, in which it is maintained in suspension. Analysed particles of the sediment sample are surrounded by diffraction rings, which record changes in optical state of the test sample in suspension. Further, the change is recorded by a sensor and transmitted to a computer. Thanks to special software, it is possible to obtain the grain size value in volumetric percentages for the specified 31 and 68 fractions within the range of 1250 to 0.16 μm.

The sample should be dried first at a temperature of 105°C and passed through a sieve with 1 mm mesh size. The grain-size of the remaining part of the sample, diameter of which exceeds 1 mm, can be determined by traditional methods, e.g. by seiving. Next, the sample is mixed with dispersion liquid and/or the sample is dispersed in an ultrasonic bath. In the case of the samples analysed, the dispersion liquid used was 0.25% tetrasodium pyrophosphate ($Na_4P_2O_7$). The volume of dispersion liquid required for performing a control reading and measurements ranges between 90 and 100 ml.

First, we perform a control reading, used for apparatus calibration, and then the measurement range. Next, we pour in the sample to the sampler, depending on the chosen analysis version (Issmer, 1994), which is present in the dispersion unit. The amount of the sample required for performing the analysis is determined automatically, as the beam obscuration ranges between 7–15%. Each measurement lasts from 3 to 15 minutes.

GRAIN-SIZE ANALYSIS BY SCANNING PHOTO-SEDIMENTOGRAPH

The *Analysette 20* Scanning Photo-Sedimentograph operates on the basis of optical measurement of the sedimentation process, which complies with Stokes' law. A beam of monochromatic light passes through a cubic glass sedimentation tank (cuvette). Changes in the suspension's optical state are registered photometrically. The detector is placed at the other side of the cuvette to the light source. It records changes in the light intensity, as part of the light radiation is absorbed by the suspension. In order to shorten the time of sedimentation of the smallest particles, which according to the Stokes' law take the longest to settle, movement of the photometer (light source and detector) that measures the settlement of the smallest particles in relation to the cuvette, is computer controlled.

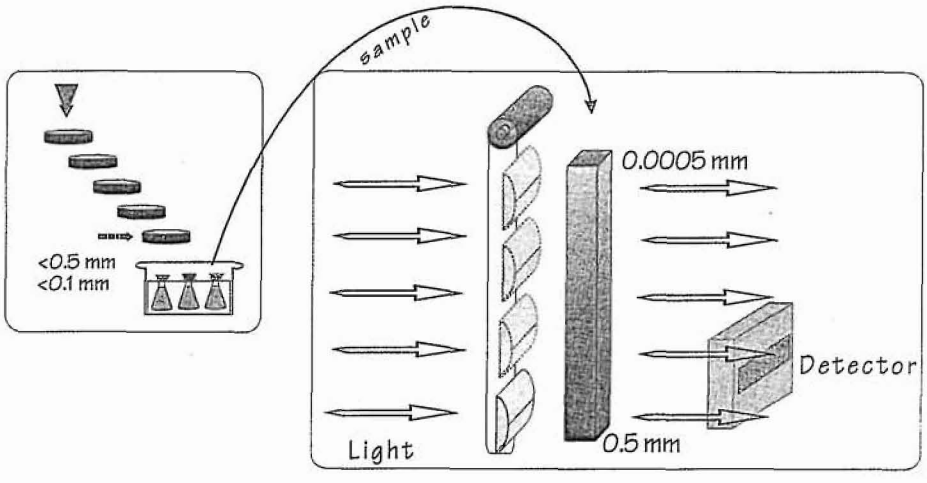

Fig. 2. Mode of operation of the *Analysette 20* Scanning Photo-Sedimentograph

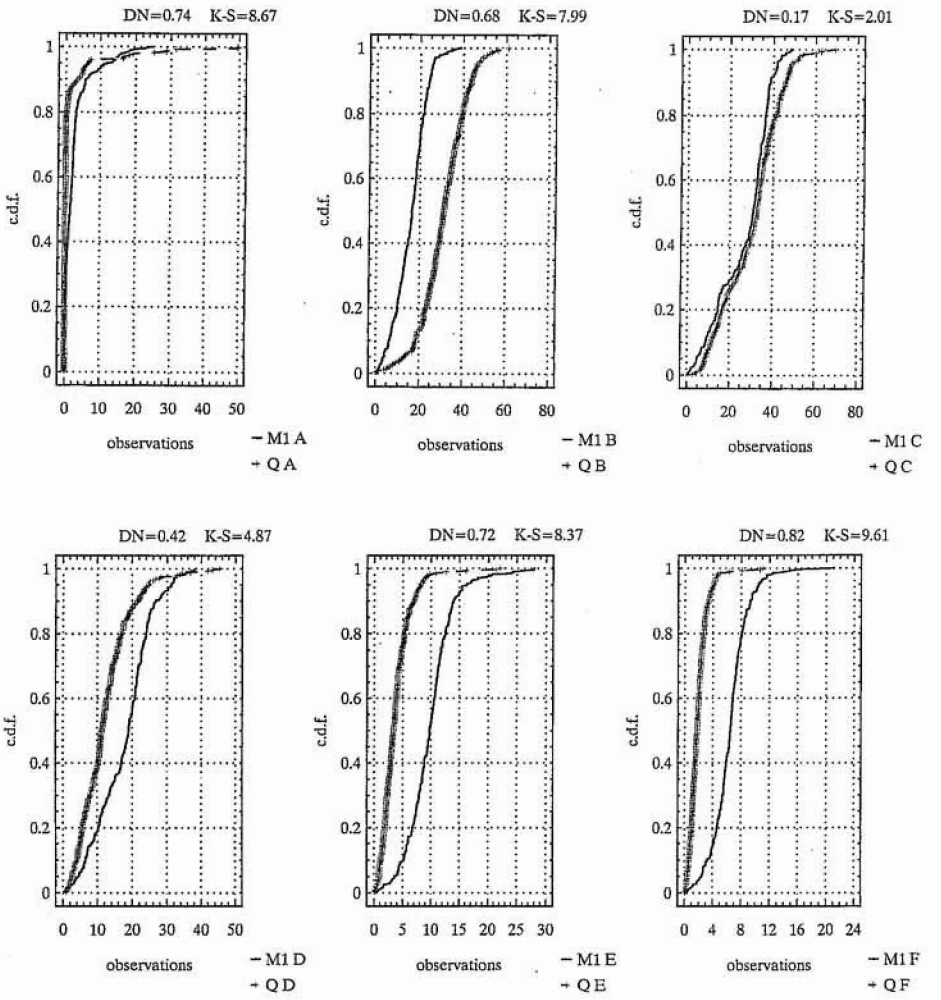

Fig. 3. Kołmogorow-Smirnow test for the *quick* (Q) and diffraction (M1) methods

c.d.f. — cumulative density function, DN — estimated overall statistics, K-S — two-sided large sample Kołmogorow-Smirnow statistics, A — >100 μm, B — 100–50 μm, C — 50–20 μm, D — 20–6 μm, E — 6–2 μm, F — < 2 μm

Changes in the intensity of the light radiation resulting from the sedimentation of the sediment are registered via the detector. Next, they are processed into electric impulses, and as such are received by the computer. Next, using suitable software, the volume percentage of particular 31 and 68 fractions in the measurement range from 500 to 0.5 μm is determined.

The sample for analysis is prepared similarly to grain-size analysis using the *Analysette 22-E* Laser Particle Sizer. The sample, dried at a temperature of 105°C, should be passed through a sieve with 0.5 mm mesh. The grain-size of the remaining part of the sample can be determined by means of the sieve method; however, the results obtained should not be combined, as the ones from optical methods are expressed in volumetric percentages, whereas those from sieve and gravity methods are in weight percentages. Next, the sample is mixed with the dispersion liquid and dispersed in an ultrasonic bath. The volume of the dispersion liquid, required for performing the control reading and the measurements ranges between 50 and 100 ml. In order to perform the measurement, we must consider the relative density of the sample and the dispersion liquid. For this purpose, 0.25% tetrasodium pyrophosphate ($Na_4P_2O_7$) is used. The basic materials and liquid types have an enclosed database detailing relative density, and there is also a possibility of entering new data.

After turning on the apparatus, a control measurement should first be performed and the parameters of the sample and dispersion liquid determined, including the suspension's relative density and temperature. The control measurement is performed when the developing dish contains only the dispersion liquid. Next, depending on the method of analysis chosen, we pour the sample into the developing dish. Sample volume should be established individually for a given sediment; the range of the volume required to perform the measurement is given each time by the computer and described as the degree of shading, ranging from 50–70%. Individual measurements depend on the grain-size of a given sample and last from a few minutes to up to an hour in the case of a full range measurement. The finer the material, the longer the measurement time.

After each measurement, the results obtained with a *Analysette 22-E* Laser Particle Sizer and with a *Analysette 20* Scanning Photo-Sedimentograph should be stored on a hard disk or on a floppy disk in a commonly used format. Data can be

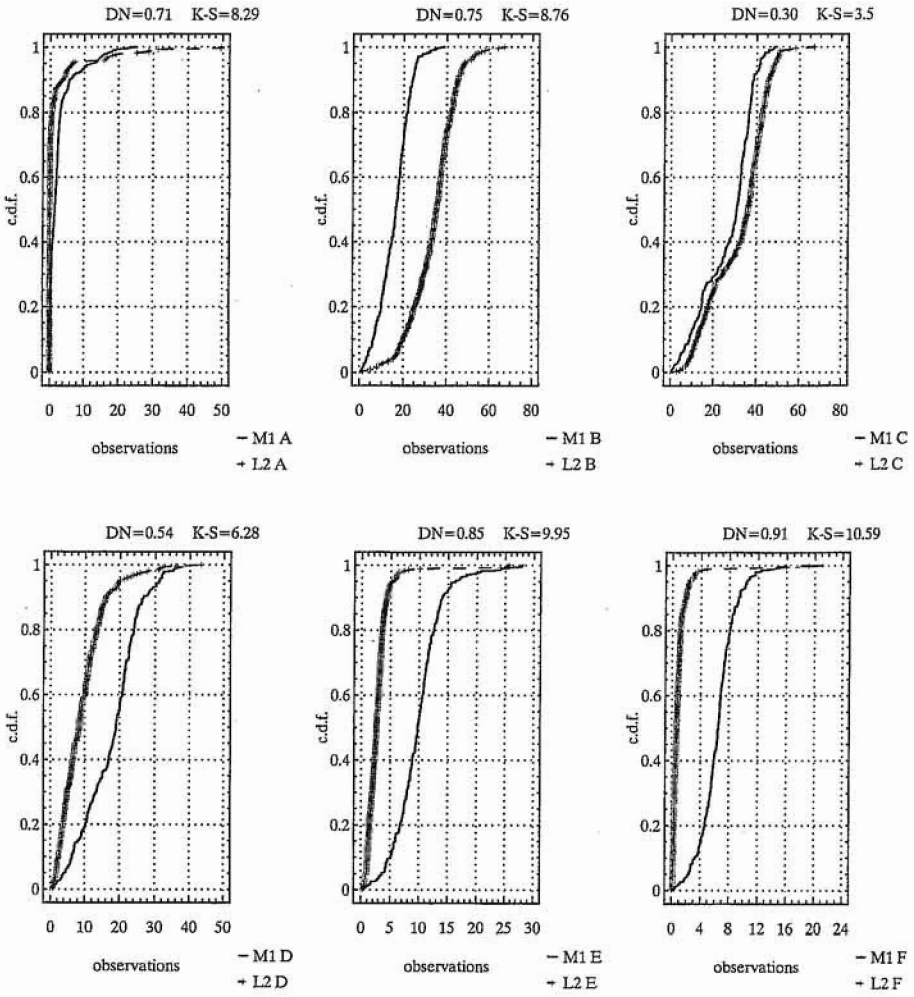

Fig. 4. Kołmogorow-Smirnow test for the *lower 2* (L2) and diffraction (M1) methods; for explanation see Fig. 3

stored as Lotus 1-2-3 files, and diagrams as MsChart files. Measurement results are presented in the form of reports, as tables specifying figures for fraction 31 and 68 in the set measurement interval, cumulation curves, histograms and statistical indices.

STATISTICAL VERIFICATION OF GRAIN-SIZE ANALYSIS

In order to compare results of the grain-size analysis using a combined method (Casagrande's aerometric method modified by Prószyński, and sieving) and optical methods, 274 reference samples of loess deposits were selected and analysed exclusively by optical methods, and 225 samples were analysed by optical methods and additionally with the combined method. The grain-size distribution for 274 loess sediment samples analysed by different optical methods indicated some regularity, which led to their verification with statistical methods.

Samples for analysis with optical methods were prepared as follows: The sample was dried first at temperature 105°C, then sieved through a sieve with 0.5 mm (500 μm) mesh size, and then covered with 0.25% tetrasodium pyrophosphate and dispersed for 10 minutes in an ultrasound tank. The grain-size of samples in suspension was then determined using the

Table 1

Coefficients of correlation between grain-size fractions for the combined and optical methods; bolded are statistically significant values

Fraction [μm]		Optical methods		
		quick	*lower 2*	diffraction
Combined method	> 100	0.019	0.018	0.618
	100–50	0.510	0.530	0.401
	50–20	**0.879**	**0.915**	**0.911**
	20–6	**0.808**	**0.821**	**0.848**
	6–2	0.420	0.443	0.538
	< 2	0.652	0.507	0.493

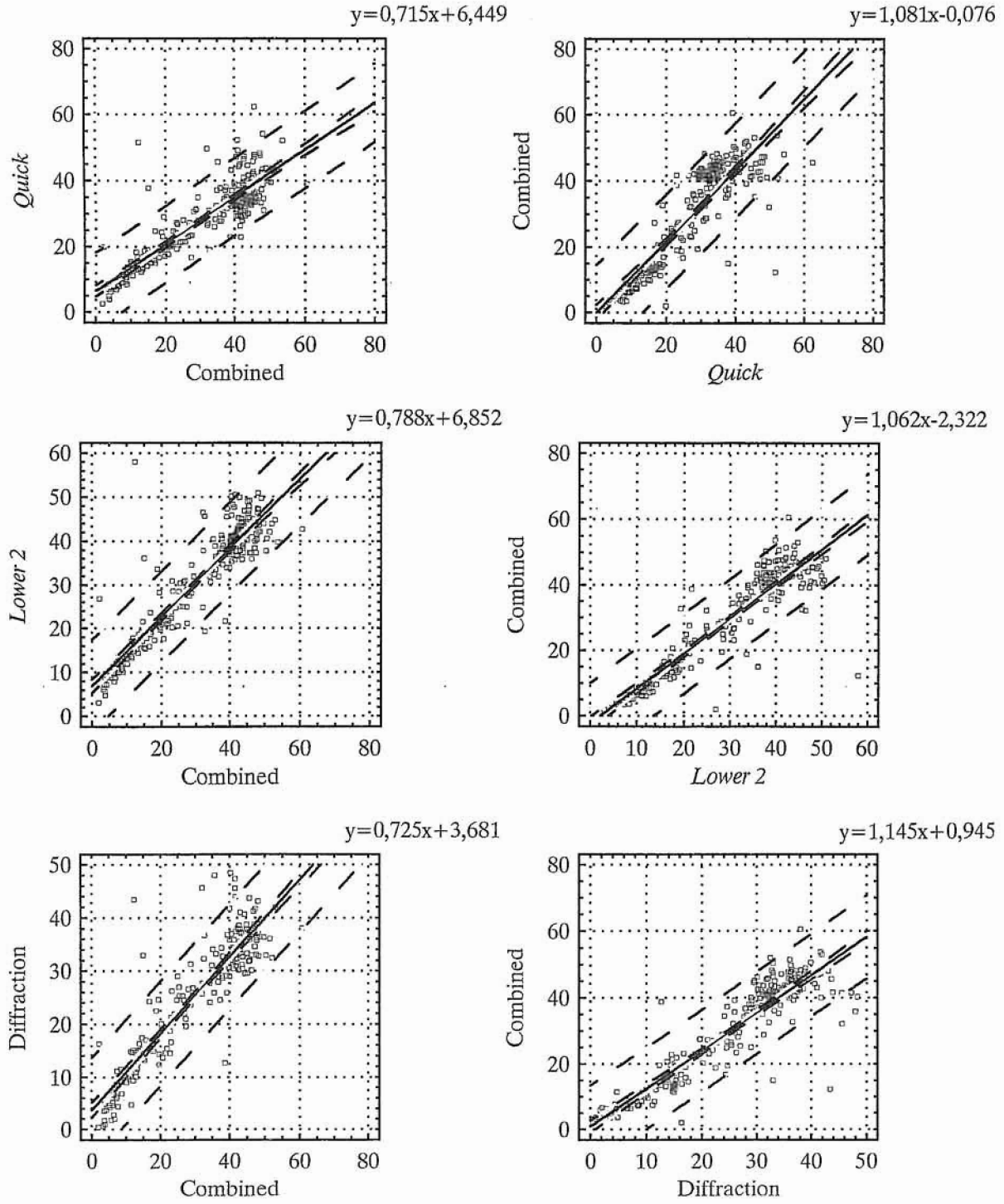

Fig. 5. Diagrams and functions of linear regression comparing the results of the 50–20 μm size fraction (loessic fraction) for optical and combined methods

Analysette 22-E Laser Particle Sizer, and the *Analysette 20* Scanning Photo-Sedimentograph. In the case of analyses performed using the *Analysette 22-E* Laser Particle Sizer, the measurement interval was set at 0.99 to 142.5 μm (0.00099–0.1425 mm). And in the case of the *Analysette 20* Scanning Photo-Sedimentograph, the analyses were performed using two methods, i.e. the *quick* and the *lower 2* ones. The *quick* method encompasses the entire measurement interval of the apparatus, from 0.5 to 500 μm. In the *lower 2* method, measurement is down to 2 μm. Selection of the measurement method was determined by the sediment grain-size, that is by the high homogeneity of sediments which had been analysed earlier with the combined method.

In order to compare the agreement between the results obtained via different optical methods, the Kołmogorow-Smirnow non-parametric test was applied. This test was performed for the following fractions: >100 μm (A), 100–50 μm (B), 50–20 μm (C), 20–6 μm (D), 6–2 μm (E), < 2 μm (F) using the *quick* (Q) and *lower 2* (L2) optical methods, and the diffraction method (M1) (Figs. 3, 4). The Kołmogorow-Smirnow test proved statistically that the optical methods used give the most compliant results in the 50–20 μm fraction, i.e. the loessic fraction.

However, in order to compare the optical methods with the combined one, correlation ratios were calculated for the commonly used grain-size fractions (Table 1) and regression equations were established for the 50–20 μm fraction (the loessic fraction), with high correlation coefficients in comparison to other fractions investigated (Fig. 5). The regression analysis used firstly the grain-size value for the optical methods as the independent variable, and then the grain-size value for the combined method.

CONCLUSIONS

The statistical analysis proved that the results obtained using optical methods are largerly in agreement with the results obtained using traditional methods in the commonly used grain-size fractions. The highest level of agreement between the results is observable in the 50–20 μm fraction (loessic fraction) and the 20–6 μm fraction, and the lowest level of agreement was observed in the coarse fraction (>100 μm). Comparing the optical method alone, high levels of agreement were found for the loessic fraction.

Comparing the results obtained using optical methods, the highest level of agreement was obtained for the *quick* and the *lower 2* methods, using the same apparatus. The lowest level of agreement occurs between the results from the *lower 2* and the diffraction methods.

The close similarity between the results from different methods allowed the projection of results from one method to the other. The equations and regression diagrams for the loessic fraction enabled recalculation of grain-size results from the optical methods into those obtained from traditional methods and *vice versa*. This means that pre-optical and optical data can be directly compared, even though they were originally expressed in different units.

Optical methods permit quick and precise measurements of the grain-sizes of fine-grained sediments in any dispersion centre while maintaining freely selected fractions for small samples (1g each). It must be noted, however, that results from optical methods are expressed in volumetric percentages and not weight percentages. Therefore, the grain-size indices of Folk and Ward (1957) cannot be determined on the basis of the results. Due to its multifunctional industrial application, the apparatus is suitable for granulometric analyses of homogenous sediments but not for heterogeneous sediments such as tills.

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