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Determination of Particle Size Distribution of Soils in Forensic Science Using Classical and Modern Instrumental Methods

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ABSTRACT: Wet sieving, laser diffraction, Coulter counting, and microscopical image analysis are compared as particle size distribution methods for the characterization of soil samples. The influence of the sample size on the distribution and the problem of choosing representative samples from small amounts of soil are highlighted.

KEYWORDS: forensic science, soils, sieves, lasers, microscopy, soil sieves, laser diffraction, Coulter counter, microscopical image analysis, particle size distribution

Soil may be described as the natural accumulation of unconsolidated mineral particles and organic matter that covers much of the earth's surface and forms the supporting medium for plant growth. The natural abundance of soil makes it an important transference material for the forensic scientist, and methods for discriminating between samples from different sources have been reported by several authors [*1-3*].

Many of the methods described measure the size distribution of soil particles, in particular, sieving. This method dates back to the days of the Egyptians [*4*]. Sieves are relatively cheap, cover a much wider size range than any other known particle sizing method (2 to 125 000 μm), and are simple to use. The application of sieves in forensic science studies have been reported by several authors [*1-7*].

However, our preliminary experiments have shown that in a majority of cases where soil transference to the clothing or shoes occurs, the predominant fraction usually falls below 63 μm[4] (unpublished results). Furthermore, the size of case samples recovered usually lies much below 500 mg. Thus, the sub 63-μm fraction reflects situations that are more likely to be encountered in operational work. This fraction can be examined using modern techniques such as Coulter counting, automated imaging, and laser diffraction. Since these methods measure different physical properties, the size distributions obtained even within

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the same soil will differ. Strictly speaking, what is compared is not the size distribution but the "repeatability" and ultimately the "discriminatory power" obtained using each method.

The Coulter counter has been in existence since 1949 and its operational principle is well known [*8,9*]. The technique permits size analysis in the range 0.4 to 800 μm and its application to soil analysis has been reported by Walker and Hutka [*10*] and by Dudley [*11*].

Microscopy is an old and absolute method of particle size analysis since it permits the observation and measurement of individual particles. The operation of the microscope for reliable size analysis requires skill and can be both tedious and time-consuming. The development of sophisticated image analyzers may assist microscopic examination by giving an objective and automated analysis of microscopically observable particles. Various types of operational principles are used, but basically, they consist of two parts: a unit for the conversion of the optical image into electrical pulses and a unit which analyzes the electrical pulses to generate quantitative image information [*12*]. These instruments can analyze particles in the range 0.4 to 150 μm. To date, no literature exists on the use of these instruments in forensic soil analysis.

The laser diffraction instrument was first introduced as a measurement tool in 1977. The operational principle is quite simple. The sample to be analyzed is dispersed in an inert liquid and circulated through a glass cell. The cell is set in the path of a coherent laser light (produced usually from a low power helium-neon unit). When the beam falls on a particle, light is diffracted and the light intensity passes through a set of decaying maxima and minima. This diffraction pattern is imaged onto a silicon multi-element photodetector and its energy can thus be measured. A small particle will produce a diffraction pattern spread over a larger angle than a large one, thus its maxima and minima will occur in different areas of the photodetector. The Fourier transform lens ensures that particles of the same size will always be imaged on the same detector element irrespective of their position and speed. In practice, there are many particles in the path of the beam and the photodetector measures the integral diffraction patterns. By summing the diffraction patterns taken at different times from the same sample, the microprocessor can produce the size distribution of the measured particles [*9,13*]. There are several instruments based on this principle, and measurements in the range 0.6 to 1800 μm are possible. To date, there is little literature on the use of these instruments in forensic soil analysis [*14*]. This publication is an attempt to assess the relative merits of the various methods of particle size analysis as applied to soil samples of forensic science interest.

Materials and Methods

Control Data Set

Wet Sieving—Twenty-six soil samples, C1 to C26, were collected from various locations in the South East of England. These soils are representative of the soil type within each location and their textural classifications range from sandy to clay. From each dried soil sample, three 0.5-g aliquots were treated and wet sieved as described previously [*15*]. The distribution of particle sizes consisted of six class intervals with upper limits of 63, 90, 250, 500, 1000, and 2000 μm. The fraction collected from each class was expressed as a percentage of the sum of fractions.

Laser Diffraction Analysis—The sub 63-μm fractions collected from the wet-sieved soil Samples C1 to C26 were used for these analyses. Sample sizes of 400, 300, 200, 100, and 50 mg giving a corresponding concentration of 1.00, 0.75, 0.50, 0.25, and 0.125 mg/mL were used. This applied to both Cilas and Malvern instruments. Using the small sample cell holder designed for use with the Malvern instrument, smaller sample sizes of 15, 10, and 5 mg with corresponding concentrations of 1.00, 0.67, and 0.33 mg/mL were examined.

Both the Cilas and Malvern instruments use the same principle but show minor variations in geometry and data presentation.

Coulter Counter Analysis—From each sub 63-μm fraction of soils C1 to C26, a suitable quantity (approximately 10 mg) was withdrawn after thorough mixing. The subsample was then dispersed by gentle manipulation with a spatula after mixing it with a Coulter dispersant. This was followed by a low power ultrasound after suspension in 5% w/v trisodium orthophosphate solution. Finally, the suspension was stirred and determined. Investigation into the effect of sampling on particle size distribution was carried out using one soil sample. This soil sample was repeatedly analyzed in the laboratory by the same operator using the same equipment over a period of three days. The weight of soil used was 3.5 mg per analysis and three analyses were performed per day. All subsamples used were withdrawn from the same gross sample.

Automated Image Analysis—The basic System III comprises a central processing unit, a camera, and a monitor. The central processing unit contains circuits which permit measurements of various functions such as areas, chords, and so forth. Measured data are available in the form of light-emitting diode (LED) or they may be fed to a peripheral computer or printer.

The scanner is a high resolution unit fitted with a vidicon tube. This provides low light performance and good signal-to-noise ratio. The scanner may be attached to any microscope provided with a phototube, and all optical techniques such as phase contrast, dark-ground, incident light, transmitted light, and fluorescence may be used. The monitor is a 12-in. (30.5-cm) green (p31) phosphor tube. Using the sub 63-μm fraction obtained from each soil Sample C1 to C26, 10 mg were subsampled, dispersed by spatulation, and sonicated. Following sonication, the concentration was adjusted to 2 mg/mL using distilled water. The suspension was then stirred and aliquots withdrawn for slide preparation. The slides were scanned using the VIDS III high resolution semi-automatic image analyzer system.

Prediction Data Set

Similar laboratory analyses were performed on 14 blind soil Samples B1 to B14. On completion of this study, the authors were informed that the soil Samples B1 to B10 originated from the control set while B11 to B14 had no association with the control set. Following wet sieving, the sub 63-μm fractions collected were used for laser diffraction, Coulter counter, and automated image analyses as described above.

Statistical Methods

Analysis was undertaken on percentage of particles (weight or volume) per class interval and percentage of organic matter. A one-way analysis of variance provided an estimate of the population variance for each class interval and organic matter content.

Identification of Unknown Samples

Using the two-sample Z statistic, the mean $\bar{b}$, of the percentage particles weight or volume of the class interval(s) (or organic matter content) of each blind sample was compared with $\bar{c}_i$, the mean of the percentage particles weight or volume of the class interval(s) (or organic matter content) for each of the 26 control soil samples, that is,

$$Z_i = \left| \frac{\bar{c}_i - \bar{b}}{\sigma \sqrt{\frac{1}{n} + \frac{1}{m}}} \right|$$

where

i = 1 to 26,
σ = population variance estimated from control soil samples,
n = number of determinations for control soil sample, and
m = number of determinations for blind soil sample.

Using the above procedure, prediction was based on the largest value of the multiplied probabilities of all the class intervals (and organic matter content where applicable). This method had been described previously in detail [*3,14,15*].

Computational Methods

The data were stored on a Digital VAX 11/782 mainframe and analysis was performed using the Minitab Statistical Package version 82.1 [*16*].

Results

Wet Sieving

Typical results obtained from the wet sieving of one control soil sample are presented in Table 1.

A corresponding result for one of the blind soil samples is presented in Table 2.

A comparison of the mean size distributions and organic matter content within Tables 1 and 2 reveals that these soils are very different and their computed similarity probability should tend towards zero. This, in fact, was the case.

Table 3 shows the similarity probabilities based on the combination of five intervals and percentage organic matter for each blind sample to the predicted origin.

TABLE 1—*Particle size distribution shown as percentage for one clay soil sample along with percentage organic matter.*

Samples	<63 μm	63 to <90 μm	90 to <250 μm	250 to <500 μm	500 to <1000 μm	1000 to <2000 μm	Organic Matter
1	94.9	1.4	1.4	1.3	1.0	0.0	5.6
2	93.8	1.4	1.1	1.6	1.0	1.1	5.6
3	92.9	1.2	1.2	1.7	1.3	1.7	5.4
Mean $\bar{a}$	93.9	1.3	1.2	1.5	1.1	1.0	5.5

TABLE 2—*Particle size distribution shown as a percentage for a sandy soil sample along with percentage organic matter.*

Sample	<63 μm	63 to <90 μm	90 to <250 μm	250 to <500 μm	500 to <1000 μm	1000 to <2000 μm	Organic Matter
1	27.8	10.6	50.6	7.5	3.5	0.0	1.6
2	28.8	10.0	50.1	7.1	3.5	0.5	2.0
3	26.8	11.4	51.1	6.5	3.1	1.1	1.9
Mean $\bar{b}$	27.8	10.7	50.6	7.0	3.4	0.5	1.8

TABLE 3—*Predicted identity of each blind soil sample origin. The five class intervals used had upper limits of 1000, 500, 250, 90, and 63 μm.*

Blind Sample	Calculated Similarity Probability	Predicted Origin from Control Soil Samples	Correct Identity of Blind Sample Origin
B1	0.95	C18	C18
B2	0.83	C13	C13
B3	0.90	C2	C2
B4	0.95	C22	C22
B5	0.96	C22	C22
B6	0.95	C16	C16
B7	0.94	C22	C22
B8	0.91	C21	C21
B9	0.82	C18	C18
B10	0.93	C4	C4
B11	0.90	C8	2 m from C8
B12	0.34	C8	11 m from C8
B13	0.90	C26	no association with controls
B14	0.51	C1	no association with controls

Laser Diffraction Analysis

A typical result obtained for a laser diffraction analysis using a sample size of 400 mg (1 mg/mL) with the Cilas instrument is presented in Table 4. A corresponding result on the Malvern Instrument using 15 mg (1 mg/mL) is presented in Table 5. In both cases, the same soil was used.

For analytical purposes, the number of class intervals was kept small by combining several

TABLE 4—*A typical % volume fraction result from the laser Granulometer 715 showing three consecutive measurements of one soil sample.*

	% Volume Fractions		
Size in μm	I	II	III
<1	11.1	10.9	10.5
1-1.5	2.9	2.9	2.9
1.5-2.0	7.2	7.2	7.7
2.0-3.0	11.7	11.4	11.8
3.0-4.0	9.8	9.5	9.7
4.0-6.0	14.3	13.4	13.9
6.0-8.0	9.7	9.4	9.8
8.0-12.0	12.3	12.5	12.0
12.0-16.0	7.4	8.0	8.3
16.0-24.0	8.6	7.9	7.7
24.0-32.0	3.5	4.3	3.5
32.0-48.0	0.9	2.0	1.7
48.0-64.0	0	0	0
64.0-96.0	0	0	0
96.0-128.0	0	0	0
128.0-192.0	0	0	0

TABLE 5—*A typical % volume fraction result from the Malvern 3600E type laser analyzer showing three consecutive measurements of the same soil sample as illustrated in Table 4.*

	% Volume Fractions		
Size in μm	I	II	III
<1.2	5.2	5.2	4.8
1.2-1.5	0.7	0.7	0.7
1.5-1.9	4.2	4.2	4.3
1.9-2.4	6.1	6.1	6.3
2.4-3.0	5.8	6.0	6.2
3.0-3.9	10.3	11.0	11.2
3.9-5.0	11.1	11.4	11.4
5.0-6.4	12.1	11.6	11.7
6.4-8.2	6.8	6.7	6.8
8.2-10.5	8.1	8.0	8.1
10.5-13.6	10.9	11.0	10.9
13.6-17.7	6.4	6.2	6.0
17.7-23.7	5.0	4.6	4.9
23.7-33.7	4.4	4.8	4.8
33.7-54.9	2.8	2.4	1.9
54.9-118.4	0.1	0.1	0.0

classes. Thus the number of classes was reduced from 16 to 4. In the case of results from the Cilas instrument, the upper limits for these classes were 6, 16, and 192 μm while those of Malvern were 6.4, 17.7, and 118.4 μm. The combined classes were used for prediction of origin. The results obtained indicate that greater variability exists in the uppermost interval, and for this reason, the first interval was omitted in the calculation of similarity probability (see Tables 6 and 7).

Coulter Counter Analysis

A typical result obtained using the Coulter counter is presented in Table 8. The upper limits for the combined classes were 10.08, 25.4, and 80.63 μm. The prediction result is presented in Table 9.

The size distributions obtained for the 18 analyses carried out in the study of the effect of sampling were examined using the cumulative size distribution. In all cases the cumulative % volumes in 2 classes (>16 and >2 μm) were analyzed.

Since the three analyses per day were performed at different times (morning, lunchtime, and afternoon), a two-way analysis of variance was performed on each variable. The results obtained are presented in Table 10.

Significant daytime lack of reproducibility indicates that the pattern of response seen at the different times in each day is different from day to day. This means that consistent results cannot be generated from day to day.

Automated Image Analysis

A typical result obtained using the automated image analyzer is presented in Table 11.

The upper limits for the combined classes were 2.0 and >30 μm. The major problem encountered and one which could lead to a biased result, is a condition termed "streaming." This condition is related to the state of the prepared slide in which the particles under obser-

TABLE 6—*Using the Cilas Granulometer 715 the origin of blind soil samples based on the combination of three class intervals only were predicted. The three class intervals used had upper limits of 6, 16, and 192 μm.*[a]

Blind Sample	Calculated Similarity Probability	Predicted Origin from Control Samples	Correct Identity of Origin
B1	0.26	C26	C18
B2	0.47	C2	C13
B3	0.70	C2	C2
B4	0.87	C22	C22
B5	0.92	C22	C22
B6	0.71	C12	C16
B7	0.97	C22	C22
B8	0.49	C9	C21
B9	0.73	C18	C18
B10	0.79	C4	C4
B11	0.76	C8	2 m from C8
B12	0.15	C8	11 m from C8
B13	0.82	C24	no association with control
B14	0.36	C5	no association with control

[a]Sample size used = 400 mg (1 mg/mL).

TABLE 7—*Using the Malvern 3600E type, the origin of blind soil samples based on the combination of three class intervals only were predicted. The three class intervals used had upper limits of 6.4, 17.7, and 118.4 μm.*[a]

Blind Sample	Calculated Similarity Probability	Predicted Origin from Control Samples	Correct Identity of Origin
B1	0.62	C26	C18
B2	0.74	C22	C13
B3	0.46	C23	C2
B4	0.74	C22	C22
B5	0.74	C22	C22
B6	0.55	C21	C16
B7	0.81	C15	C22
B8	0.64	C15	C21
B9	0.63	C14	C18
B10	0.70	C6	C4
B11	0.74	C8	2 m from C8
B12	0.79	C8	11 m from C8
B13	0.46	C17	no association with control
B14	0.50	C12	no association with control

[a]Sample size used = 15 mg (1 mg/mL).

TABLE 8—*A typical % volume result from the Coulter counter model TA II/PCA/Accucomp™ system for three consecutive measurements of one soil subsample.*[a]

	% Volume Fractions		
Size in μm	I	II	III
<2.52	0.0	0.0	0.0
2.52-3.17	1.3	1.4	1.4
3.17-4.00	1.8	1.9	1.9
4.00-5.04	2.2	2.2	2.3
5.04-6.35	3.0	3.2	3.0
6.35-8.00	3.7	4.0	3.8
8.00-10.08	4.5	4.7	4.4
10.08-12.70	4.9	5.2	5.1
12.70-16.00	6.0	5.8	5.8
16.00-20.16	7.4	6.7	7.0
20.16-25.40	9.7	9.6	9.0
25.40-32.00	14.8	15.3	14.6
32.00-40.32	24.0	20.2	24.8
40.32-50.80	15.4	18.5	16.3
50.80-64.00	1.3	1.1	0.7
64.00-80.63	0.0	0.0	0.0

[a]Sample size used is approximately 10 mg (0.05 mg/mL).

TABLE 9—*Using the Coulter counter the origin of blind soil samples based on the combination of three class intervals only were predicted. The three class intervals used had upper limits of 10.08, 25.4, and 80.63 μm.*

Blind Sample	Calculated Similarity Probability	Predicted Origin from Control Samples	Correct Identity of Origin
B1	0.22	C13	C18
B2	0.52	C23	C13
B3	0.16	C2	C2
B4	0.23	C12	C22
B5	0.46	C13	C22
B6	0.22	C16	C16
B7	0.55	C13	C22
B8	0.81	C11	C21
B9	0.31	C16	C18
B10	0.27	C5	C4
B11	0.11	C8	2 m from C8
B12	0.21	C8	11 m from C8
B13	0.45	C5	no association with control
B14	0.29	C13	no association with control

TABLE 10—*Analysis of variance on soil fractions >16 and >2 μm on observations obtained using the Coulter counter.*

	DF	SS	MS	*F* Ratio
		(a) >16-μm SOURCE		
Days	2	0.004 405	0.002 203	14.4934
Time[a]	2	0.003 675	0.001 837	12.0855
Days × time	4	0.016 224	0.004 056	26.6842 Sp < 0.01
Error	9	0.001 372	0.000 152	
Total	17	0.025 676		
		(b) >2-μm SOURCE		
Days	2	0.000 582 3	0.000 291 2	10.6667
Time	2	0.001 714 3	0.000 857 2	31.3993
Days × time	4	0.001 899 3	0.000 474 8	17.3919 Sp < 0.01
Error	9	0.000 246 0	0.000 027 3	
Total	17	0.004 442 0		

[a]Time represents morning, lunchtime, and afternoon.

TABLE 11—*A typical result from the VIDS III high resolution semi-automatic image analysis system showing three consecutive measurements of one soil sample.*

	Accumulated Count %		
Size in μm	I	II	III
<1.0	57.57	57.42	57.38
1.0-2.0	32.23	32.54	32.33
2.0-3.0	4.95	4.88	4.90
3.0-4.0	1.92	2.01	2.07
4.0-5.0	1.37	1.32	1.42
5.0-10.0	1.10	1.09	1.14
10.0-20.0	0.55	0.48	0.46
20.0-30.0	0.27	0.24	0.28
>30.0	0.04	0.02	0.02

vation are seen to be flowing from one position to another. Since measurements were performed over the entire slide, "streaming" could lead to counting of the same particles several times as they change positions. Sizing was performed in the chord mode but it is probable that area sizing would yield better results. The results of prediction are presented in Table 12.

In Table 13, the results from all methods are summarized.

Discussion

The wet sieving of the soil samples permitted determination of organic matter content and size distributions. Although the size distributions obtained were satisfactory for sandy soils (Table 2), a major proportion of the bulk sample fell below 63 μm for clay samples (Table 1). This prevented the use of median particle size for comparative purposes. Predictions based

TABLE 12—*Using the image analyzer, the origin of blind soil samples based on the combination of two class intervals only were predicted. The two class intervals used had upper limits of 2.0 and >30 μm.*

Blind Sample	Calculated Similarity Probability	Predicted Origin from Control Samples	Correct Identity of Origin
B1	0.52	C14	C18
B2	0.47	C12	C13
B3	0.39	C5	C2
B4	0.50	C18	C22
B5	0.78	C4	C22
B6	0.32	C11	C16
B7	0.16	C12	C22
B8	0.25	C12	C21
B9	0.50	C25	C18
B10	0.23	C20	C4
B11	0.87	C26	2 m from C8
B12	0.69	C12	11 m from C8
B13	0.48	C5	no association with control
B14	0.63	C10	no association with control

TABLE 13—*A summary of correct predictions from the control data set using all methods studied.*

Method	Weight of Sample Used, mg	Number of Correct Predictions Out of Ten
Wet sieving	500	10[a]
Laser diffraction	400 (Cilas)	6[b]
	15 (Malvern)	2[c]
Coulter counter	10 approximately	2
Automated image analyzer	10[d]	0

[a]Of the ten correct predictions of blind sample origin, three samples originated from C22 and two from C18—see Table 3.

[b]Of the six correct predictions of blind sample origin, three samples originated from C22—see Table 6.

[c]The two correct predictions originated from the Sample C22—see Table 7.

[d]The actual analysis was carried out on approximately 0.5 mL of suspension which should contain approximately 1 mg of sample.

on a combination of five intervals only were the same as for the combination of five intervals and percentage of organic matter. Various interval combinations were examined. The organic matter content is included in the results because it can be determined on sample sizes as small as 500 mg and gives additional confidence to the results obtained. Of the 14 blind samples examined, 10 were selected from the control set and all were correctly predicted (Table 3). Of the 10 samples, 3 were taken from Sample C22 and 2 were taken from Sample C18. If the replicates are considered as 1 sample each, then only 7 out of the 26 control samples were represented in the blind samples. The remaining 4 (B11, B12, B13, and B14)

were outside the controls. Of these, B11 and B12 were collected at distances of 2 and 11 m from the control soil Sample C8. It is therefore not surprising that both B11 and B12 were predicted to be C8. The other two blind samples, B13 and B14, had no association with the controls but their high similarity probabilities to some of the control soil samples indicate that soils from other sources can display size distributions similar to those of the controls. These soils may be excluded on the basis of other parameters such as color, pH, and so forth, but in this study, only their size distributions were considered (Table 3).

The rest of the discussion will concentrate exclusively on blind samples (B1 to B10) originating from the control group. The triplicate results obtained using the various modern instruments show that all are capable of achieving consistent repeatable results (Tables 4, 5, 8, and 11). The differences in results obtained for the same soil sample (Tables 4 and 5) could be due to several factors. Some of these include the use of different soil weights, differences in dispersion of the determined soil samples (these were actually determined in different laboratories at different times), or differences in data acquisition and presentation of these instruments. The last of these could be tested if access to Cilas and Malvern instruments were possible at the same time using the same sample. However, as long as both controls and blinds are analyzed using the same instrument, such differences become unimportant.

The two major factors that emerge from this study relate to sampling and sample preparation. Although the instruments can achieve consistent results at the weight levels examined, it appears that operators cannot prepare representative subsamples at these levels. The results obtained from the sampling examination of one soil sample using the Coulter counter clearly show that there are major differences between the size distribution obtained from different subsamples (Table 10). This limitation is not restricted to the Coulter instrument and is applicable to both the laser diffraction instruments and the automated image analyzer. The other limitation is the achievement of repeatable dispersion. For some unexplained reason, very good dispersion can be obtained within an hour for some samples using low power ultrasound. However, the same samples can take a couple of hours to achieve full dispersion at other times. It is therefore important that dispersed samples should be microscopically examined under low magnification before determination on all instruments.

The number of correct predictions (Table 13) for the laser diffraction analysis would suggest that what matters is not sample concentration but sample weights. Although the sample concentrations on both instruments were 1 mg/mL, it is more difficult to achieve a representative subsample of 15 mg as opposed to 400 mg. It is therefore not too surprising to see that the number of correct predictions had fallen from six to two. The same condition prevailed for the Coulter analysis, but the automated image analysis was taken one step further. In this case, the original subsample of 10 mg was again subsampled after dispersion to deposit approximately 1 mg of material on the slide. The final analysis was therefore performed on approximately 1 mg of sample while all other methods used at least 10 mg of sample. On this occasion, no correct predictions were made.

From the results of this study and our previously published work it is clear that wet sieving is a very useful and reproducible technique for the analysis of the larger soil sample. Standardization of operating parameters is however crucial in obtaining the maximum information from the sample. The technique is inexpensive, readily available, and certainly can have an important role in a protocol for soil examination and comparison. Our studies of soils have focused on methods for particle sizing, but we clearly recognize and do not underestimate the value of many other parameters in the analysis of soil. Although our results show more sophisticated instruments to be inferior (in prediction analysis) to wet sieving, it should be borne in mind that one of us (S. W.) has had extensive experience with this technique and relatively less experience in the use of other methods employed. It may be that long-term use of one of these methods would yield the consistency required to give reproducible results with minute samples. The prize to be gained lies in the smaller sample size required compared to wet sieving.

Conclusion

Particle size distribution analysis may not be a suitable method for the comparative analysis of minute quantity of soils in which all the particle sizes fall below 63 μm. The results obtained from this study suggest that a sample size of 100 mg could be the limit of repeatable sampling for an experienced operator. Furthermore, the results suggest that the inherent limitations in particle size distribution analysis are not a function of instrumental error but of sampling ability and a detailed study of sampling procedures of small quantities of soil is required.

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