CHANGING A LABORATORY METHOD: AN EXAMPLE CONCERNING PARTICLE SIZE ANALYSIS

M.J. ROWELL

The Agricultural Laboratory, Ministry of Agriculture, Water and Rural Development, Private Bag 13184, Windhoek, Namibia

ABSTRACT

A micro-pipette method to determine the distribution of mineral particles fractions in soil is described. A comparison with a hydrometer method currently in use in our laboratory indicated that the new procedure gave better results for sand and clay content but that variability in silt analysis was about the same. Performance was evaluated by comparing the results with data from an inter-laboratory testing program in which both hydrometer and conventional pipette methods were used. The micro-pipette method was rated as satisfactory to excellent overall with 98% of sand, 90% of silt and 98% of clay analyses falling within 2 standard deviation units of the pooled robust mean. It was concluded that a change to the micro-pipette method was justified in terms of improved accuracy, sample turnover and more efficient use of chemicals and technical time.

INTRODUCTION

Particle size analysis (PSA) into sand, silt and clay fractions and its description as texture is one of the most important soil measurements. Texture is closely linked to water holding, drainage, aeration and mechanical characteristics concerning cultivation and load bearing. Particle size distribution along with organic matter, pH and salinity are the main factors determining soil productivity.

The performance rating for PSA by the Agriculture Laboratory (MAWRD) in a program run by the Agricultural Laboratory Association of Southern Africa (ALASA) was erratic and particularly poor for that of clay content. The method we used was based on the hydrometer method of Bouyoucos (1957) which is conceded to be a compromise between high sample volume and accuracy. The pipette method, which is the standard method of choice, is more time consuming but produces more reliable results which can be defended on a more theoretical basis (Day, 1965).

In this study I modified a micro-pipette procedure that had been used to measure total and water dispersible clay (Miller and Miller, 1987; Burt et al., 1993). The method was tested by comparing the ALASA inter-laboratory data from the last three years with the re-analysis of the same samples using the new procedure.

MATERIALS AND METHODS

1. Background

Both the pipette and the hydrometer methods rely on the same basic principle. A soil sample is shaken with an alkaline sodium hexametaphosphate solution to break up aggregates and disperse the mineral particles. The dispersed soil is allowed to settle on a vibration-free surface. If the individual mineral particles are assumed to be spherical and of uniform density then their settling rate conforms to the principles of Stoke's Law. This may be expressed as the equation:

$$V = \frac{d^2 g (Ps - Pw)}{18\eta}$$

where:

- V = Settling velocity of the soil particle through water (cm/sec)
- Ps = Density of the soil particle, g/cm^3 (generally taken as 2.65 g/cm^3)
- Pw = Density of water (g/cm^3)
- d = Diameter of the soil particle (cm)
- g = Acceleration due to gravity (cm/s^2)
- η = Viscosity of water (poise)

If the temperature remains constant the equation may be resolved to determine the time taken for different sizes of particles to settle and be cleared entirely from a particular depth in the sedimentation container. In the hydrometer method a special soil hydrometer is used to determine the density of the soil suspension while with the pipette method a small sample is removed to determine the solids content. The pipette procedure may use commercially manufactured equipment in the form of a constant temperature water bath, special sedimentation cylinders and a rack holding a 20 ml pipette that can be lowered precisely into the suspension by reference to a graduated scale. The amount of clay (<2m diameter) and silt (2-53 m) are measured as above while the distribution of sand sized particles (53-200 m) is determined by wet sieving. The procedure can be refined to measure sub-fractions of the clay, silt and sand ranges. Pre-treatments are often carried out to remove organic matter, cementing agents or soluble salts.

2. Micro-pipette method

Some 49 soil samples were selected from those used between 1995 and 1997 in the ALASA testing scheme on the basis of adequate sample weight and wide variation in particle size range. All except 6 had been previously analysed by the laboratory using the hydrometer method. No pre-treatments were carried out. Air-dried soil samples (20 g) were weighed into identical straight-sided 250 ml capacity leak proof polyethylene bottles with inner liners and tight fitting screw caps. To each was added 20 ml of dispersing solution (40 g sodium hexametaphosphate and 10 g of sodium carbonate per litre) and 100 ml water. The bottles were capped and shaken lengthwise for two 30 minute periods on the first day. A blank bottle containing dispersing solution and water but no soil was prepared. After standing over-night the bottles were shaken for an additional 30 minutes, an extra 80 ml of water was added, the caps replaced and the bottles placed on a laboratory bench. The temperature of the fluid in the blank bottle was noted. Tables were consulted to determine the correct sampling times for silt and clay analysis for that particular temperature. The first sample of silt plus clay is taken after about 25 seconds while the second for clay alone is taken after about 4 hours. The procedure for the initial sampling involved shaking the soil suspension for 30 seconds, placing the bottle on the bench, removing the liner and cap, and recovering a 5 ml sample at a depth of 6 cm with a variable volume 1000-5000 ml micro-pipette. To the sampling tip of the micro-pipette was attached a rigid expanded polystyrene collar that formed a template so that when the pipette assembly rested on the neck of the bottle the tip reached to a depth of exactly 6 cm into the suspension. The sample was ejected into a previously weighed aluminium weighing dish and dried at 105° C. Samples were taken at two minute intervals. The second 5 ml sample for clay estimation was recovered after the prescribed clay sampling interval, placed in an aluminium weighing dish and dried and re-weighed as before. Samples were taken from the blank bottle at identical times to take into account the chemicals in the dispersant. Finally, the sand fraction was recovered by tipping the contents of the bottle onto a 53 m sieve and washing the sieve free of fine material with a fine jet of tap water. The sand residue was rinsed into another aluminium dish and oven dried to determine the weight. The amount of sand, silt and clay was calculated from the weight of residue recovered from each sampling event and the sieving. Correction was made for the blank readings. A spread sheet was prepared to simplify the routine calculations and provide results in terms of percentage of sand, silt, clay and total material recovered.

3. Data analysis

The ALASA program provides a statistical summary of data, which includes the robust mean, standard deviation as well as a z-score and rating for the 2 soils analysed each month by the participating laboratories. Over the 3 year period 9 to 12 laboratories have taken part in the performance testing scheme. The majority used the normal pipette method while a few, like us, used the hydrometer method. It is assumed that the ALASA mean and standard deviation reflects the true content and likely variability for sand, silt and clay in these soils.

The z-score returns positive or negative values relative to the difference between the individual result and the mean in terms of the standard deviation as calculated from:

$$z_i = (x_i - m)/s$$

where: x_i is the result from laboratory i
m is the mean
s is the standard deviation.

I also calculated the coefficient of variation (CV) for the ALASA data, which gives a comparison of variability between laboratories for results of varying magnitude. The CV is calculated from:

$$CV\ (\%) = s/m \times 100$$

Separate CVs were determined for between-run and within-run duplicate measurements in our laboratory.

The individual soils in the ALASA data set were compared graphically with laboratory results for hydrometer and micro-pipette methods. Performance was further assessed by comparing deviation and bias about the ALASA mean by examining individual z-scores and defining a mean z-score. It should be realised that the latter is not a true statistic when comparing data that has not contributed to calculation of the ALASA mean.

RESULTS

ALASA data were sorted from low to high values for sand, silt and clay and scatter plots prepared to compare them with our results (Figures 1 to 3). The data indicated that for sand there was generally a good agreement between the ALASA mean and the micro-pipette results but that our hydrometer results included many readings that were too high. Both methods showed equivalent variability in silt analysis though the micro-pipette method tended to give readings that were higher than the ALASA mean. The micro-pipette method gave close agreement with the ALASA mean for clay content while many of our hydrometer results were much too low.

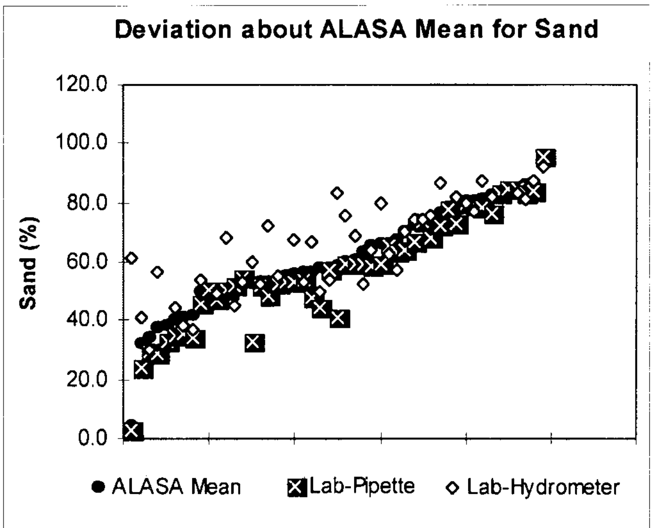


Figure 1. Comparison of laboratory results with ALASA mean for percentage sand.

The performance of each method was analysed in terms of z-scores (see Figures 4 to 6) to compare variability in respect to the standard deviation of the ASALA set. The performance data is summarised in Table 1. Performance of a method is rated as excellent for z-scores of between -1 and +1 and operationally satisfactory with z-scores of -1 to -1.9 or 1 to 1.9. The micro-pipette method gave improved performance in all analytical categories in terms of mean z-scores and a higher percentage of A-ratings. Our sand results using the hydrometer

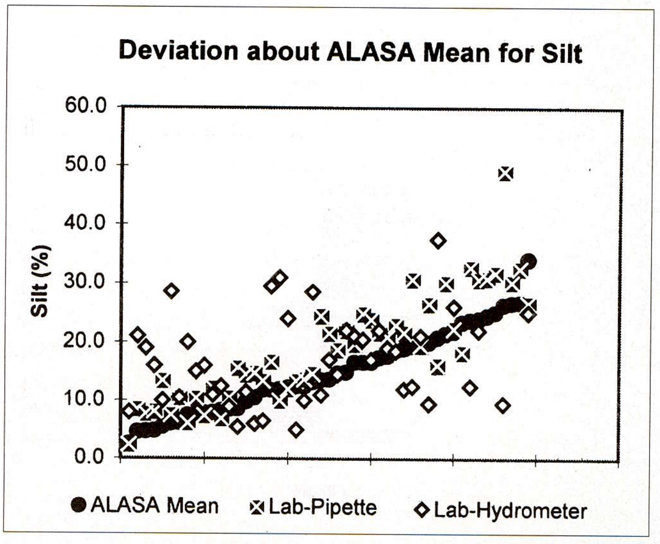


Figure 2. Comparison of laboratory results with ALASA mean for percentage silt.

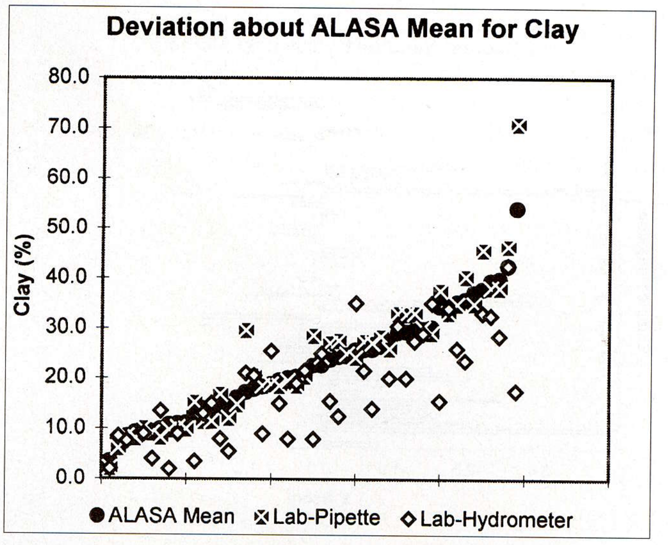


Figure 3. Comparison of laboratory results with ALASA mean for percentage clay.

Table 1. Summary of laboratory micro-pipette and hydrometer results with ALASA data

	Particle Size Fraction					
	Sand		Silt		Clay	
	Pipette	Hydrometer	Pipette	Hydrometer	Pipette	Hydrometer
Percentage of results lower than ALASA mean	10.2	53.5	81.6	62.8	46.9	25.6
Mean z-score[1]	0.7	1.3	1.0	0.8	0.5	1.4
A-rating (%)	74	58	63	58	90	42
B-rating (%)	24	30	27	37	8	40
C-rating (%)	1	7	2	5	2	9
D-rating (%)	0	5	8	0	0	9

[1]Mean z-score is the mean of individual z-scores converted into positive scores

Performance ratings based on z-scores:
A-rating (excellent): z <-1 or z <1
B-rating (satisfactory): z -1 to -1.9 or z= 1 to 1.9
C-rating (poor): z -2 to -2.9 or z 2 to 2.9
D-rating (very poor): z >-2.9 or z > 2.9

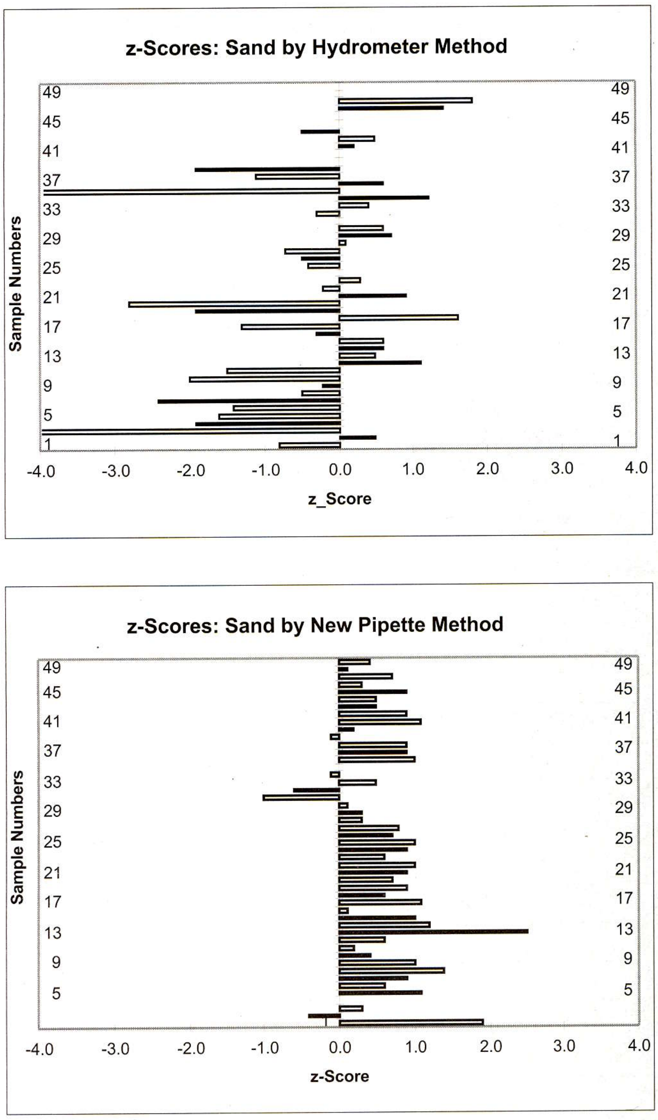
method were typified by a lack of bias but with many values excessively too high, 12% received C- or D-ratings. In contrast sand analysis by the micro-pipette procedure yielded 98 % of the results A- or B-rated but with a strong bias to results higher than the ALASA mean. With silt analysis most results received A- or B-ratings though the micro-pipette method gave readings that were consistently higher than the ALASA mean. In clay measurement the micro-pipette method gave accurate and unbiased results with 90% of the results given an A-rating. Our hydrometer results were poor with 18% of the results C- or D-rated and with a tendency to overestimate the true value badly.

Experience shows that the measurement of particle size distribution can be quite variable between laboratories. In the ALASA data set, which involved up to 12 laboratories, the CVs were 13 %, 40 % and 23 % for sand, silt and clay respectively (Table 2). In our laboratory replicate

Table 2. Variability between laboratories, between runs and within runs

	Coefficient of Variation (%)					
	Sand		Silt		Clay	
	Range	Mean[4]	Range	Mean	Range	Mean
ALASA inter-lab comparison[1]	1.6 to 116.7	12.9	7.2 to 107.7	39.5	5.3 to 60.0	22.6
Lab within run comparison[2]	0.1 to 7.0	1.7	0.7 to 11.2	6.2	1.2 to 6.9	3.3
Lab between run comparison[3]	0.1 to 2.1	0.8	5.7 to 17.2	12.5	3.5 to 16.7	8.6

[1]49 sets of measurements over 3 years involving about 12 laboratories
[2]9 soils analysed in singly in two different runs
[3]5 soils analysed in duplicate in the same run
[4]The average of CVs of individual soils, or sets of soils analysed by different labs with the ALASA data



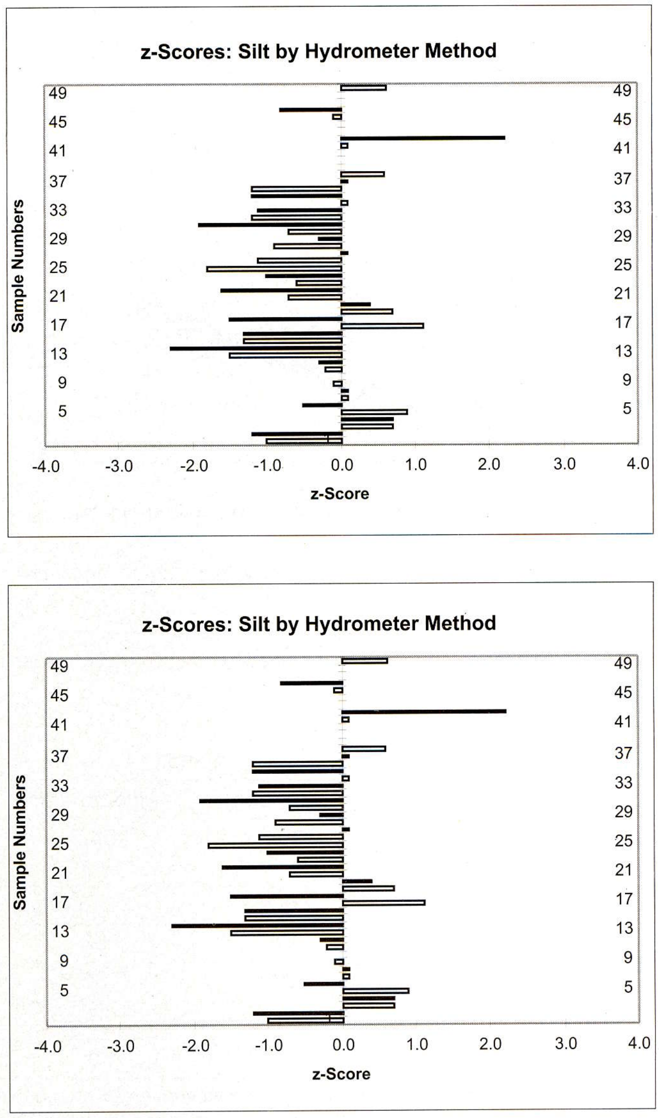


Figure 4. Z-scores for sand by hydrometer and micro-pipette methods.

Figure 5. Z-scores for silt by hydrometer and micro-pipette methods.

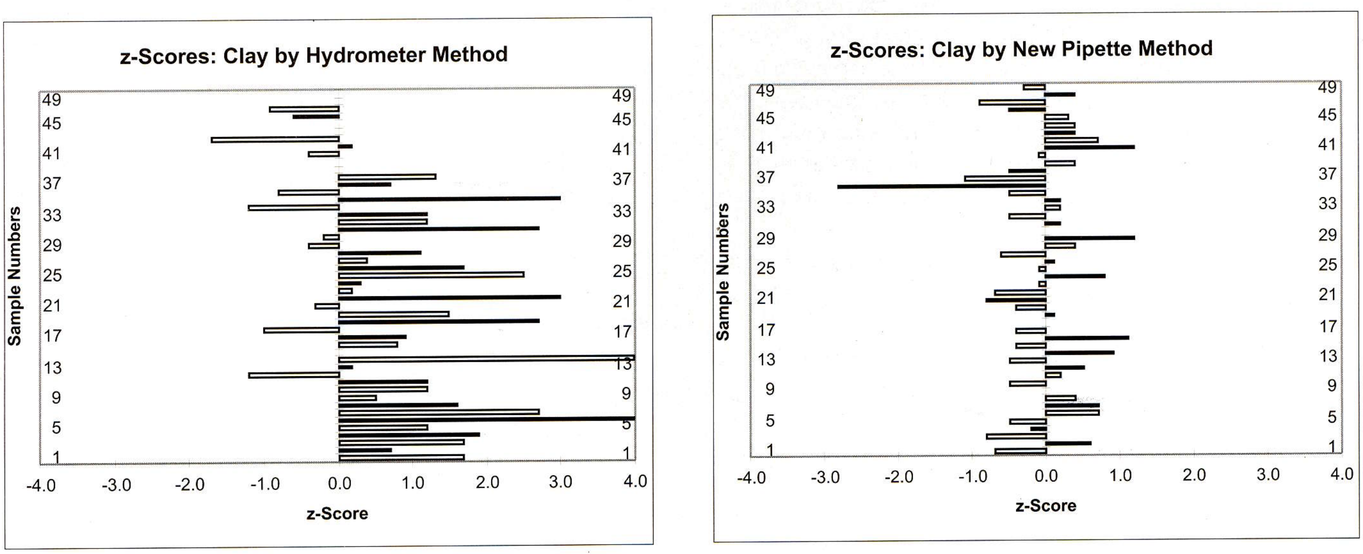


Figure 6. Z-scores for clay by hydrometer and micro-pipette method.

measurements for different soils in 5 separate runs gave CVs of 1 %, 13 % and 9 % for the same fractions. The CVs for within-run comparisons were 2%, 6% and 3% for sand, silt and clay respectively.

DISCUSSION AND CONCLUSIONS

The micro-pipette method gave more consistent results for sand and clay than our hydrometer procedure though, silt analysis showed similar variability. Our poor performance by the hydrometer method may have been due to incomplete dispersion of some samples since a rather mild shaking was employed. However, accurate reading of the hydrometer is often difficult particularly in soils that produce a froth after agitation by the stirring paddle. The micro-pipette method has several features that tend to produce greater reproducibility and accuracy. Determination of sand, silt and clay concerns weighing operations whereas the hydrometer method a mixture of density and weight is involved. Sampling by micro-pipette is precise and reproducible as long as the positioning of the sampling tip and calibration of the pipette is checked. The general lack of improvement in silt analysis may have been related to variation due to the very short sedimentation time for the silt plus clay estimation. About 20-30 samples can be conveniently handled by either method per day each day although the micro-pipette procedure uses one-fifth the amount of laboratory chemicals.

It is concluded that the micro-pipette method is a more reliable method than our current hydrometer method and gives acceptable results when compared to data from an inter-laboratory study involving conventional pipette and hydrometer methods.

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