Making Soil Particle Size Analysis by Laser Diffraction Compatible with Standard Soil Texture Determination Methods

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The standard sieving, pipette, and hydrometer methods for soil particle size analysis (PSA) have three main drawbacks: (i) the procedures are tedious, (ii) the procedures are time consuming, (iii) and the results are protocol dependent. Laser diffraction PSA delivers rapid results using standardized procedures, but so far it has been difficult to reconcile results with those from standard sedimentation methods. The objective of this study was to develop a protocol that would permit direct usage of laser diffraction PSA and render results compatible with current methods. The protocol was developed using 54 standard soil samples from different textural classes. Regression of the laser diffraction PSA against the hydrometer/pipette method yielded R^2 values of 0.92/0.9, 0.92/0.94, and 0.99/0.99 and RMSE values of 0.04/0.05, 0.07/0.06 and 0.05/0.03 for clay, silt, and sand, respectively. These statistics are comparable to those obtained by regressing results of the hydrometer against the sieve and pipette methods. A key factor in securing accurate and precise results was limiting the particle size range of the samples by wet sieving the sand fraction. This created representative samples and stable soil dispersed suspensions, allowing accurate estimations of particle size distribution for clay and silt fractions without empirical transformations. Results obtained with the proposed protocol matched those of standard sedimentation analyses for a wide range of soils, encouraging further adoption of laser diffraction for soil PSA.

Abbreviations: ALP, Agriculture Laboratory Proficiency; NAPT, North American Proficiency Testing; PSA, particle size analysis.

The particle size distribution of the soil mineral fraction modulates physical, chemical, and biological properties, including soils' hydraulic properties, pore size distribution, shrink and swell capacity, erodibility, and sedimentation, with implications for agroecological, mechanical, hydrological, geological, and engineering applications (Bieganowski et al., 2018; Blake and Steinhardt, 2008; Gee and Or, 2002; Hillel and Hatfield, 2005; Merkus, 2009). Hence, multiple bodies have developed particle size classification systems to catalog the effects of particle size distribution on such properties and on the properties and functions of particulate media, including the International Organization for Standardization, the ASTM International standards, as well as the soil texture classification by the US Department of Agriculture Natural Resources Conservation Service (USDA–NRCS) and the International Soil Science Society ISSS, now International Union of Soil Science (IUSS) (Blake and Steinhardt, 2008; FAO, 2014; Gee and Or, 2002; Moeys et al., 2018; USDA, 2017).

The most common methods for soil particle size analysis (PSA) are the sieving, pipette, and hydrometer methods (Gee and Or, 2002). The pipette and hydrometer methods are based on gravitational sedimentation principles and, in

Core Ideas

- **Laser diffraction particle size analysis can produce results compatible with standard pipette and hydrometer methods.**
- **A key step is to wet-sieve the sand fraction after suspending the soil sample in the dispersant solution.**
- **The proposed protocol is faster, uses smaller samples, and provides more detail than standard sedimentation methods.**

G.S. Faé and F. Montes contributed equally to this work.

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combination with sieving, constitute the foundation of soil texture classification standards around the world (Blake and Steinhardt, 2008; Loveland and Whalley, 2000). These methods have three main drawbacks: (i) the procedures are tedious, (ii) the procedures are time consuming, (iii) and the results are protocol dependent. Significant errors arise from variations in chemical dispersant concentration, sample size, type of cylinder used, temperature fluctuations, sieving time, variation of time intervals used to record hydrometer or pipette measurements, and fundamental differences in the methods' assumptions of particle shape (Allen, 1997; Eshel et al., 2004; Gee and Or, 2002; Merkus, 2009; Syvitski et al., 2007). These errors are particularly pronounced at the extremes of the particle size spectrum; sedimentation methods do not reliably measure particles >50 mm because these particles settle rapidly, and these methods also tend to overestimate clay content due to particle–fluid interactions that interfere with the sedimentation process (Allen, 1997; Chung and Hogg, 1985; Matthews, 2007; Merkus, 2009).

Over the past 30 yr, improvements in technology, particularly in computational capacity and sensor accuracy, have opened the possibility of laser diffraction for particle size analysis (Agrawal and Pottsmith, 2000; Bieganowski et al., 2018; Di Stefano et al., 2010). This has translated into an increased number of articles related to laser diffraction PSA in the soil science literature, summarized in the recent review by Bieganowski et al. (2018). Laser diffraction PSA has many advantages, including the use of standardized procedures to deliver rapid results, with more precise differentiation into a high number of size intervals. More detailed soil particle size information will enable advances in the understanding of fundamental hydraulic, hydrologic, structural, and biological soil processes, including development of more accurate pedotransfer functions for soil hydraulic properties (Schaap et al., 1998), and new insights into biological processes regulating organo-mineral associations (Oliveira et al., 2018). In addition, soil particle size distribution determines the soil surface area available to interact with organic matter and soil microorganisms (Borkovec et al., 1993; Hassink and Whitmore, 1997) controlling physical and biological processes that occur at or below the pore scale.

However, soils can be composed of a broad mixture of minerals and particle sizes, which results in the following challenges for laser diffraction PSA. First, it is difficult to obtain a representative proportion of the coarser particles from the original sample due to particle size segregation in dry samples and rapid sedimentation of large particles in liquid-dispersed samples. Second, once a sample is obtained, it is difficult to disperse all the particles in the sample into a stable suspension to interact with the laser source. Third, it has not been possible to match results from laser diffraction with standard soil texture determination methods without prior calibration based on the texture classification, organic matter and carbonate content, or the development of dataset-specific conversion equations (Di Stefano et al., 2010; Lamorski et al., 2014; Makó et al., 2017; Taubner et al., 2009).

A critical step in making laser diffraction and the sieving, hydrometer, and pipette methods comparable is identifying equivalent Stokes' and light scatter diameters for clay and silt fractions. Sedimentation, sieving, and laser diffraction PSA measure different particle properties. When sedimentation methods are used, the nominal particle size diameter measured is the Stokes' diameter (i.e., "the diameter of a sphere having the same settling rate as the particle under conditions of Stokes' law") (Merkus, 2009). The Stokes' diameter is different from the nominal sieve diameter (i.e., "the diameter of particles that just pass through the apertures of a sieving medium") measured by sieving and the nominal light scatter diameter (i.e., "the diameter of a spherical particle with the same optical properties that produces the distinctive light scattering pattern") measured by laser diffraction (Merkus, 2009). The equivalent Stokes', sieve, and light scatter diameters for a homogeneous material vary depending on the density, shape, and optical parameters of the particles composing the material. Therefore, the nominal particle size distribution varies depending on the method used, as stated in the International Standard for Particle Size Analysis-Laser Diffraction Methods ISO:13320 (International Organization for Standardization, 2009).

The soil texture classification from the USDA–NRCS uses 2 µm as the nominal equivalent Stokes' diameter to differentiate between the clay and silt fractions when using standard sedimentation methods. Estimates for the equivalent light scatter diameter have varied between 6 µm (Miller and Schaetzl, 2012), 8 µm (Konert and Vandenberghe, 1997), and 9 µm (Fisher et al., 2017). Makó et al. (2017) detected a small variation in this threshold, ranging from 6.6 to 5.8 µm in soils with and without organic matter. Arriaga et al. (2006) used the USDA–NRCS 2 µm threshold and modified the refractive index and absorption index to match pipette method analysis results. However, Eshel and Levy (2007) warned against this approach, arguing that the refractive index that produced the best results (1.42) was below the ranges of accepted values for soil minerals (1.54 for quartz and 1.49 for calcite) and would produce distorted particle size distributions.

After an analysis of 41 soil samples from California, Eshel et al. (2004) stated that relationships between particle size distribution obtained with laser diffraction and standard sedimentation methods varied across textural classes and that no consistent relationship could be formulated. Other studies have shown relationships between particle size data obtained with laser diffraction and sedimentation methods for different regions, but correlations differed between datasets, and high deviations from the 1:1 regression line were observed in the smallest size class (Buurman et al., 2001; Konert and Vandenberghe, 1997). Taubner et al. (2009) used sieving to exclude coarse particles from the suspension, improving the collection of homogeneous aliquots, but concluded that laser diffraction analysis could not be used for texture classification of soils without regression-transformed size fractions and validation using a sedimentation method. Miller and Schaetzl (2012) showed that 11.5% of their 1485 soil samples changed textural class when the laser diffraction analyses were repeated, attributing the changes to subsampling errors. They concluded that the putative precision of laser-generated particle size data decreased with coarser particles due to misrepresentation of the complete population of particles in the sample and the effect of large particles on the sand percentage volume. Hence, reducing the particle size range of the sample could increase precision by reducing measurement variability.

Even though there are international standards associated with laser diffraction and standard sedimentation methods, difficulties in making the results of both methods compatible has hindered the adoption of laser diffraction PSA method by the soil science community (Bieganowski et al., 2018; Di Stefano et al., 2010; Lamorski et al., 2014). Therefore, the objective of this study was to develop a laser diffraction protocol without the need for empirical transformations for soil PSA that produced results comparable to those obtained by standard USDA–NRCS sedimentation methods (Blake and Steinhardt, 2008; USDA, 2017). The protocol developed is based on two key methodological improvements: (i) separation of the sand fraction through wet sieving after dispersion of the soil sample and before laser diffraction analysis, as suggested by Taubner et al. (2009), and (ii) the use of a laser diffraction instrument that overcame accuracy limitations of the instrument used by Taubner et al. (2009).

MATERIALS AND METHODS

Selection of Soil Samples

Standard soil samples from the North American Proficiency Testing (NAPT) Program, which operates under the Soil Science Society of America assisting interlaboratory evaluation of analytical data, and the Agriculture Laboratory Proficiency (ALP) Program managed by Collaborative Testing Services Inc., were used to compare soil texture results obtained with the sieve/pipette and the hydrometer methods—as described by Gee and Bauder (1986)—with results obtained using the laser diffraction PSA protocol developed in this study. Both programs used the Soil, Plant, and Water Reference Methods for the Western Region (Gavlak et al., 2013), the Recommended Soil Testing Procedures for the Northeastern United States (Northeast Coordinating Committee for Soil Testing, 2011), the Recommended Chemical Soil Tests Procedures for the North Central Region (North Central Regional Research, 2015), and the Soil Test Methods from the Southeastern United States (Sikora and Moore, 2014). North American Proficiency Testing quarterly soil analysis reports from 1998 to 2017, comprising 400 soil samples analyzed by 115 analytical testing laboratories (NAPT, 2018), and ALP quarterly soil analysis reports from 2013 to 2017, comprising 70 soil samples analyzed by 104 analytical testing laboratories (ALP, 2018), were digitized and assembled into a database using R statistical software (R Core Team, 2018). The reports provided a robust statistical summary of soil test results that included the median and the median absolute deviation for each of the 470 soil samples. The median values for clay, silt, and sand were used to assign each sample to a textural class of the USDA–NRCS textural classification using the ‘Soiltexture’ Package in R (Moeys et al., 2018). A total of 54 of these soils standards, comprising six to eight soil samples from each USDA–NRCS textural class in the database, were requested to the Penn State Ag Analytical laboratory soil standards repository for laser diffraction PSA. The properties of these samples are presented in Fig. 1 and Table 1.

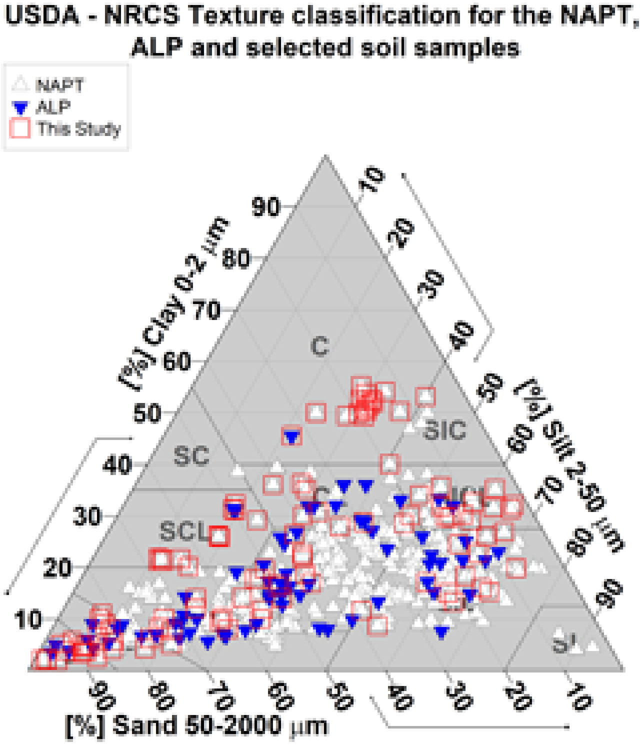

Fig. 1. Textural composition of soil samples according the USDA–NRCS texture triangle. White triangles represent the soil texture classification corresponding to the median clay, silt, and sand content reported for each sample in the quarterly North American Proficiency Test report. Blue triangles represent the soil texture classification corresponding to the median clay, silt, and sand content reported for each sample in the quarterly Agriculture Laboratory Proficiency report. The red squares surrounding triangles identify the samples selected for this study.

Sample Preparation and Sieving

A representative, well-mixed, and dry 5-g soil material (<2 mm) sample was collected from each of the 54 selected standard materials according to the best fundamental sampling error calculation proposed by Rawle (2015). The 5-g material sample was placed in a 470-mL container with 100 mL of a 5% sodium hexametaphosphate solution, vigorously mixed, and left to soak overnight to disperse the soil particles. The following day, distilled water was added to make up 300 mL of solution, after which samples were mixed for 5 min in a Triple-Spindle Drink Mixer HMD400 (Hamilton Beach Brands Inc.) to obtain a stable soil particle suspension sample (Polakowski et al., 2015). After mixing, the soil solution was sieved through a 53-µm mesh to separate sand-sized particles and to determine the sand fraction (f_{sa}, kg kg^{-1}). The sieve-removed particles were oven-dried at 105°C for 24 h, and f_{sa} was calculated as the ratio of the mass of the 53-mm sieved dried particles (M_{sa}, kg) and the total dried soil sample mass (M_T, kg).

Laser Diffraction Particle Size Analysis

Laser diffraction PSA used the Malvern Mastersizer 3000 laser diffractometer equipped with a He-Ne red light at 632.8 nm wavelength and an LED blue light at 470 nm wavelength, a 600-

Table 1. Information about the standard soil samples used in the laser particle size analysis: sample identification, soil classification, and median values for clay, sand, total organic carbon (TOC) and carbonate ($CaCO_3$) as stated in the North American Proficiency Test and Agriculture Laboratory Proficiency quarterly reports used in this study.‡

			Sieve/pipette method				Hydrometer method					
Sample	**Location**	**Soil classification**	**Sand**	**Silt**	**Clay**	***n*†**	**Sand**	**Silt**	**Clay**	***n*†**	**TOC**	**$CaCO_3$**
				– % –				– % –			– % –	
2011-116	Antigo, WI	coarse-loamy over sandy or sandy-skeletal, mixed, superactive, frigid Haplic Glossudalfs	37.0	54.2	8.7	4	38.2	50.6	11.6	30	1.1	0.1
2011-117	Umatilla, OR	coarse-loamy, mixed, superactive, mesic Xeric Haplocalcids	82.9	12.9	5.6	4	76.5	17.5	6.1	30	0.5	0.4
2011-118	Fresno, CA	coarse-loamy, mixed, superactive, mesic Ustic Haplocalcid	78.3	17.5	4.2	4	73.5	22.0	5.0	30	0.9	0.4
2011-119	Sturgeon Bay, WI	coarse-loamy, mixed, superactive, frigid Typic Haplorthods	54.7	33.1	11.8	4	56	33.7	10.0	30	2.8	1.3
2011-120	San Juan, UT	clayey, mixed, active, thermic, shallow Abruptic Durixeralfs	50.1	30.7	16.2	4	43.9	38.4	17.0	30	0.9	5
2012-101	San Luis Obispo, CA	fine-loamy, mixed, superactive, thermic Calcic Haploxerolls	12.5	37.3	50.2	5	17.0	30.0	51.4	43	3.1	12.1
2012-102	Monmouth, NJ	fine-loamy, mixed, active, mesic Typic Hapludults	65.9	21.9	12	5	64.8	21.0	14.0	43	0.7	0.5
2012-103	Riley, KS	coarse-silty, mixed, superactive, nonacid, mesic Typic Udifluvents	72.0	20.1	7.7	5	62.5	29.6	8.0	43	0.6	1.7
2012-106	Fresno, CA	coarse-loamy, mixed, superactive, thermic Fluvaquentic Haploxerolls	87.2	10.3	2.5	5	83.9	12.0	4.0	38	0.5	0.3
2012-108	Door, WI	fine, mixed, active, mesic Typic Hapludalfs	49.3	34.2	16.7	5	50.4	30.2	19.7	38	2.5	1.1
2012-109	Farmington, UT	fine-loamy, mixed, superactive, mesic Cumulic Haploxerolls	44.1	35.9	18.7	5	43.0	34.9	21.7	38	2.8	3.4
2012-110	San Patricio, TX	fine-loamy, mixed, superactive, hyperthermic Typic Argiustolls	67.5	10	21.2	5	66.8	11.7	21.2	38	0.5	0.4
2012-112	Grand, UT	fine-loamy, mixed, superactive, calcareous, mesic Typic Torrifluvents	42.7	34.9	22.6	5	42.1	31.2	26.4	40	1.5	9.1
2012-113	Grand Forks, ND	fine-loamy, mixed, superactive, frigid Calcic Hapludolls	33.8	39.1	27.9	5	38.8	32.0	30.0	40	2.7	0.6
2012-114	Kent, DE	fine-loamy, siliceous, semiactive, mesic Typic Hapludults	88.3	8.03	3.7	5	87.2	8.0	4.75	40	0.6	0.3
2012-115	Baxer, TX	clayey-skeletal, smectitic, thermic Lithic Haplustolls	96.7	1.6	2.03	5	95.7	2.8	2.2	40	0.6	0.3
2012-116	Caldwell, KY	fine-silty, mixed, active, mesic Oxyaquic Fragiudalfs	15.0	69.8	15.2	2	17.5	64.5	18.3	36	1.1	0.7
2012-117	Linn, OR	fine-silty, mixed, superactive, mesic Cumulic Ultic Haploxerolls	73.8	18.0	8.3	2	72.1	18.0	10.1	36	0.4	0.7
2012-118	Arlington, WI	fine-silty, mixed, superactive, mesic Typic Argiudolls	8.2	72.0	19.8	2	13.1	63.8	22.7	36	2	0.8
2012-119	Waushara, WI	mixed, mesic Typic Udipsamments	89.2	7.3	3.4	2	88.7	6.4	4.62	36	0.4	0.5
2013-102	San Luis Obispo, CA	fine, smectitic, thermic Vertic Haploxerolls	17.9	33.0	52.6	5	16.0	31.5	52.0	40	2.8	11.3
2013-105	Wilacy, TX	fine-loamy, mixed, superactive, hyperthermic Udic Argiustolls	55.1	18.3	26.0	5	54.9	19.6	25.8	40	0.6	4.2
2013-109	San Luis Obispo, CA	fine-loamy, mixed, superactive, thermic Calcic Haploxerolls	18.9	31.8	49.3	4	16.4	29.3	53.7	34	2.6	18.5
2013-111	Bailey, TX	fine-loamy, mixed, superactive, thermic Aridic Paleustalfs	89.8	5.6	5.6	4	85.0	8.4	6.6	39	0.9	0.5
2013-114	San Luis Obispo, CA	fine, smectitic, thermic Aridic Haploxererts	21.9	32.0	49.4	4	26.6	22.5	50.0	39	1.8	2
2013-119	Allegheny, PA	fine-loamy, mixed, semiactive, mesic Typic Hapludults	57.0	34.2	14.1	4	50.3	35.0	14.0	37	1.2	0.8
2014-103	Cache Junction, UT	fine, mixed, superactive, mesic Typic Natrixerolls	4.0	56.8	35.2	3	12.9	51.3	35.6	36	1.4	5.3
2014-111	Larimer, CO	fine-loamy, mixed, superactive, mesic Aridic Argiustolls	36.0	30.9	30.7	5	36.0	28.4	36.2	37	1.3	6.6
2014-119	Riley, KS	fine, smectitic, mesic Pachic Argiustolls	14.7	57.1	31.8	3	20.8	48.5	30	39	1.8	0.5

Continued

Table 1. Continued.

Sample	Location	Soil classification	Sieve/pipette method				Hydrometer method				TOC	$CaCO_3$
			Sand	Silt	Clay	*n*†	Sand	Silt	Clay	*n*†		
2015-101	Linn, MO	fine, smectitic, mesic Aquertic Argiudolls	2.3	64.2	32.1	1	14.3	55.7	29.7	41	2.1	0.6
2015-103	San Luis Obispo, CA	fine-loamy, mixed, superactive, thermic Calcic Pachic Haploxerolls	6.1	57.4	31.6	1	18.0	31.0	50.4	41	2.6	9.7
2015-108	Linn, MO	fine, smectitic, mesic Aquertic Argiudolls	6.7	66.5	26.9	3	15.0	56.0	29.9	39	2.1	0.5
2015-109	Felton, DE	fine-loamy, siliceous, semiactive, mesic Typic Hapludults	89.0	7.45	3.6	3	88.0	7.3	4.8	39	0.6	0.3
2015-113	San Patricio, TX	fine-loamy, mixed, superactive, hyperthermic Typic Argiustolls	66.7	11.5	21.6	5	67.2	11.2	21.8	36	0.5	0.4
2015-115	Linn, MO	fine-silty, mixed, semiactive, thermic Typic Paleudults	9.0	63.5	32.6	5	11.1	57.5	29.0	36	2.1	0.5
2015-118	Linn, MO	loamy, kaolinitic, thermic Arenic Kandiudults	3.3	64.4	31.3	4	11.9	57.3	30.2	36	2.1	0.5
2016-111	Sao Luis Obispo, CA	fine-loamy, mixed, superactive, thermic Calcic Haploxerolls	13.0	32.8	54.1	7	16.0	30.3	52.5	39	2.7	17.2
2016-114	Cache, UT	fine-silty, mixed, superactive, mesic Calcic Pachic Argixerolls	22.8	45.4	28.4	7	29.4	44.0	28.7	39	3	1.3
2017-101	Centre, PA	fine, mixed, semiactive, mesic Typic Hapludalfs	15.0	62.0	25.0	7	16.3	58.0	24.0	35	1.7	0.8
2017-102	Grand Forks, ND	sandy, mixed, frigid Oxyaquic Hapludolls	83.7	7.48	8.01	7	82.5	9.2	8.01	35	1.8	0.7
2017-103	Penobscot, ME	sandy, isotic, frigid Typic Haplorthods	65.0	26.0	9.80	7	63.9	27.0	9.0	35	2.5	0.6
2017-104	Gallatin, MT	loamy-skeletal over sandy or sandy-skeletal, mixed, superactive, frigid Typic Argiustolls	35.0	47.5	18.0	7	40.2	41.6	17.5	35	3.7	2.8
2017-105	Grand, UT	mixed, mesic Typic Torripsamments	91.8	2.00	3.76	7	92	3.9	5.0	35	0.4	0.6
2017-107	Grand Forks, ND	sandy, mixed, frigid Oxyaquic Hapludolls	82.0	7.00	11.0	3	82.6	9.0	9.0	35	1.9	0.6
2017-108	Larimer, CO	fine-loamy, mixed, superactive, mesic Aridic Argiustolls	40.9	27.0	36.0	3	35.8	28.7	35	35	1.3	5.5
2017-109	Cache, UT	fine-silty, mixed, superactive, mesic Typic Calcixerolls	19.3	48.0	40.0	3	17.3	49.6	34.0	35	2.5	5.8
2017-111	Cache, UT	loamy-skeletal, micaceous Ustic Dystrocryepts	53.0	29.2	17.8	5	52.2	31.2	17.8	38	2.5	2.7
2017-112	Minnehaha, SD	fine-silty, mixed, superactive, mesic Udic Haplustolls	10.0	64.0	26.0	5	15.5	61.1	24.5	38	2.9	1
2017-113	Caroll, NH	coarse-loamy, mixed, active, frigid Aquic Dystrudepts	62.2	28.0	10.2	5	63	27.5	9.55	38	4.7	0.5
2017-114	Grand Forks, ND	sandy, mixed, frigid Oxyaquic Hapludolls	83.0	7.2	10.0	5	82.1	9.0	8.0	38	1.7	0.6
2017-115	Cumberland, IL	fine, smectitic, mesic Mollic Albaqualfs	15.8	68.0	16.0	5	22.0	63.3	15.0	38	1.3	0.4
SRS-1508	Pinal County, AZ	fine-loamy, mixed, superactive, calcareous, hyperthermic Typic Torrifluvents	–	–	–	0	49.6	17.8	31.1	26	1.7	1.8
SRS-1604	Sonoma County, CA	fine, mixed, semiactive, mesic Typic Haploxerults	–	–	–	0	50.0	18.5	31.3	30	2.2	0.2
SRS-1709	Sonoma, CA	loamy, mixed, superactive, thermic Lithic Haploxerepts	–	–	–	0	33.0	22.0	45.0	28	5.4	0.6

† The number of analytical laboratories analyzing the sample resulting in the reported median.

‡ Source: North American Proficiency Testing Program (NAPT, 2018), Agriculture Laboratory Proficiency (ALP) Program (ALP, 2018), University of California Davis California soil resource Lab SoilWeb (O'Geen et al., 2017).

mL Hydro LV dispersion unit, and 101 size bins covering particle sizes from 0.01 to 3500 μm (Malvern Panalytical Inc.). This is in contrast with the Analysette 22 (Fritsch GmbH) instrument used by Taubner et al. (2009), which was equipped with a 638-nm laser source and measurement range of 0.290 to 295 μm distributed into 62 bins.

A subsample of the suspension of the fractions passing the 53-μm mesh sieve was taken with a wide-mouth transfer pipette and added to the wet dispersion unit of the Mastersizer 3000. Enough suspension was added to increase the obscuration level to ~10%. A maximum stirring speed of 3500 rpm and 100% sonication power were applied for the duration of each measurement event (~2 min). Some samples were replicated three times to test the reproducibility and consistency of the subsample grabbing method. From the dispersion unit, the sample was circulated in the measurement cell where the interaction of particles and light occurred. Sample replication showed very low variability (CV ranged from 0.6 to 5.8% when sonication was applied); therefore, only one reading per sample was taken for the selected NAPT and ALP sample analyses. Sample analyses used the Mie scatter-

Table 2. Threefold cross-validation statistics for clay content determined by randomly dividing the dataset in thirds. Two thirds of the dataset were used in the clay–silt cutoff optimization routine, and the remaining third was used for validation.

Statistic	Training set 1	Validation set 1	Training set 2	Validation set 2	Training set 3	Validation set 3
Intercept	−0.01	−0.01	−0.001	−0.004	−0.006	−0.002
Slope	1.01	0.93	0.98	1.14	0.97	0.94
RMSE, g g^{-1}	0.05	0.02	0.04	0.06	0.04	0.04
R^2	0.89	0.99	0.95	0.89	0.92	0.92
n	36	18	36	18	36	18
Threshold, µm	6		7		6	

ing general purpose model for quartz material with nonspherical shape mode, a particle refractive index of 1.543, a particle absorption index of 0.01, and a water refractive index of 1.33. After each measurement, the Hydro LV cell was washed with distilled water twice before a new sample was added, and the analyses were always performed with degassed water. Background scattering by the solution was measured periodically after every 25 samples to verify the cleanliness of the measurement cell. The laser diffraction analyses were performed at the Penn State Materials Characterization Laboratory, which performs nominal particle size calibration with Duke Standards TM Polymer Microspheres NIST traceable diameter 8.9 ± 0.5 µm on a monthly basis or on demand.

Clay and silt contents, as a mass fraction of the sample, were obtained by subtracting the amount of sand from each sample (M_T - M_{sa}), multiplying the remaining mass of the sample by the volume fraction of clay or silt obtained from laser diffraction analysis, and then dividing by the total mass of sample (Eq. [1] and [2]):

$$f_{\mathrm{cl}} = \frac{\left(M_{\mathrm{T}} - M_{\mathrm{sa}}\right) \times f_{\mathrm{cl-LD}}}{M_{\mathrm{T}}} \qquad [1]$$

$$f_{\mathrm{si}} = \frac{\left(M_{\mathrm{T}} - M_{\mathrm{sa}}\right) \times f_{\mathrm{si-LD}}}{M_{\mathrm{T}}} \qquad [2]$$

where f_{cl} is the mass fraction of clay in the sample, $f_{\mathrm{cl\text{-}LD}}$ is the percent cumulative volume of particles less than a clay–silt cutoff of 6 µm (discussed below), f_{si} is the mass fraction of silt in the sample, and $f_{\mathrm{si\text{-}LD}}$ is the percent cumulative volume of particles greater than the clay–silt cutoff. This approach assumes spherical particles and equal particle density to partition the clay–silt mass into clay and silt fractions, the same assumptions used in sedimentation methods.

Table 3. Comparison of the coefficient of determination and root mean square error for the fraction of clay and silt using the standard clay–silt cutoff of 2 µm and the previously recommended 6 and 9 µm.

		Clay		Silt	
Cutoff, µm	Methods	R^2	RMSE	R^2	RMSE
2	hydrometer vs. pipette	0.96	0.03	0.93	0.06
2	laser diffraction vs. hydrometer	0.75	0.18	0.86	0.22
2	laser diffraction vs. pipette	0.82	0.17	0.91	0.19
6	laser diffraction vs. hydrometer	0.92	0.04	0.92	0.07
6	laser diffraction vs. pipette	0.90	0.05	0.94	0.06
9	laser diffraction vs. hydrometer	0.90	0.07	0.93	0.05
9	laser diffraction vs. pipette	0.88	0.08	0.91	0.07

Comparing Methods

The clay–silt cutoff was selected through an optimization routine developed in Microsoft Excel to minimize the difference between the standard sample clay and silt fractions obtained using the proposed laser diffraction PSA protocol and the median clay and silt fractions reported by the NAPT and the ALP programs (Table 1). The optimization routine searched for the clay–silt particle size threshold over a similar range to the 2- to 9-µm threshold range reported in the literature and discussed above (Arriaga et al., 2006; Fisher et al., 2017; Konert and Vandenberghe, 1997; Makó et al., 2017; Miller and Schaetzl, 2012; USDA, 2017) using the bin sizes of 1.88, 2.13, 2.42, 2.75, 3.12, 3.55, 4.03, 4.58, 5.21, 5.92, 6.72, 7.64, and 8.68 µm reported by the Mastersizer 3000.

To check the stability of the clay–silt cutoff, a threefold cross-validation (James et al., 2013) was conducted by randomly splitting the dataset in three groups, calculating the clay content with two-thirds of the data, and validating the results against the remaining third of the data. The cross-validation was performed three times, using the three different two thirds of the 54-observation dataset as training models and the other three respective one-thirds of the dataset as validation (Table 2).

Sand, silt, and clay fraction results obtained using the laser diffraction protocol were compared with NAPT and ALP sieve/pipette and hydrometer-reported medians using linear regression analysis. The 1:1 regression line provides a visual assessment of the magnitude of the errors, and the R^2, the slope, and the intercept provide quantitative measures of the bias between PSA methods.

RESULTS

The clay–silt particle size cutoff that yielded the best agreement between the laser diffraction PSA and the NAPT and ALP reported results was the 5.92 µm Mastersizer 3000 size bin (Tables 2 and 3). As expected, the standard USDA–NRCS 2 µm clay–silt cutoff produced inferior results, which agrees well with previous reports (Makó et al., 2017; Miller and Schaetzl, 2012). Using 6 µm as the clay–silt threshold produced robust results in the threefold cross-validation and good agreement with the NAPT- and ALP-reported data, with values of the regression intercept and slope close to the 1:1 line, high R^2, and low RMSE (Table 2). Furthermore, the magnitude of the RMSE between laser diffraction PSA results and the reported NAPT and ALP data ranged between 0.04 and 0.07, which is very close to the magnitude of the regression RMSE between pipette and hydrometer methods (0.03–0.06) (Table 3; Fig. 2). Analysis of the regression residuals indicated no significant correlation with total organic C or carbonate content, which agrees with prior reports by Fisher et al. (2017).

This study used a refractive index of 1.54 (corresponding to quartz) and an absorption index of 0.01. However, the Mastersizer 3000 allows recalculation of results using different scattering parameters, and we also recalculated our results using the refractive index values of 1.52 and absorption index of 0.1 proposed by Bieganowski et al. (2018). The major difference was a shift in the clay–silt cutoff from the 5.92 μm to the adjacent 5.21 μm bin. This was a very small change with minor impact on the texture classification corresponding to the standard sedimentation results reported by NAPT and ALP.

DISCUSSION

Eshel and Levy's statement that "there is no room to match particle size distribution data obtained by the LD [laser diffraction] to those obtained by the pipette" (Eshel and Levy, 2007) refers to the difficulties in obtaining laser diffraction PSA results for soils with different particle sizes, densities, shapes, and optical material properties that are consistent with sedimentation methods. The laser diffraction protocol presented in this work overcomes some of these limitations and is able to obtain results comparable with standard soil texture determination methods from a broad geographic area of the United States that are representative of most soil textural classes and that vary substantially in soil taxonomic classification, total organic C, and carbonate content. Implementing the idea proposed by Taubner et al. (2009) of limiting the particle size range to obtain consistent representative soil particle suspensions, in combination with technological improvements on laser diffraction instrumentation, allowed consistent estimations of particle size distribution for clay and silt fractions. The protocol presented is robust regarding variation in laser diffraction analytical parameters such as the refractive index and the absorption index. Varying the refractive index within reasonable bounds produced small changes in the particle analysis. Similarly, varying the clay–silt size cutoff from 2 to 9 μm produces PSA results that remained close to those reported by NAPT and ALP (Table 3).

With consistent soil laser diffraction PSA results, the issue of equating laser diffraction and sedimentation results is transformed into an empirical problem with various practical solutions, as has been demonstrated by Fernlund et al. (2007) for sieving and image analysis methods and by Garboczi et al. (2017) for laser diffraction, sieving, and a combination of X-ray computer tomography and spherical harmonic analysis. The solution chosen in this work was based on three steps: (i) separa-

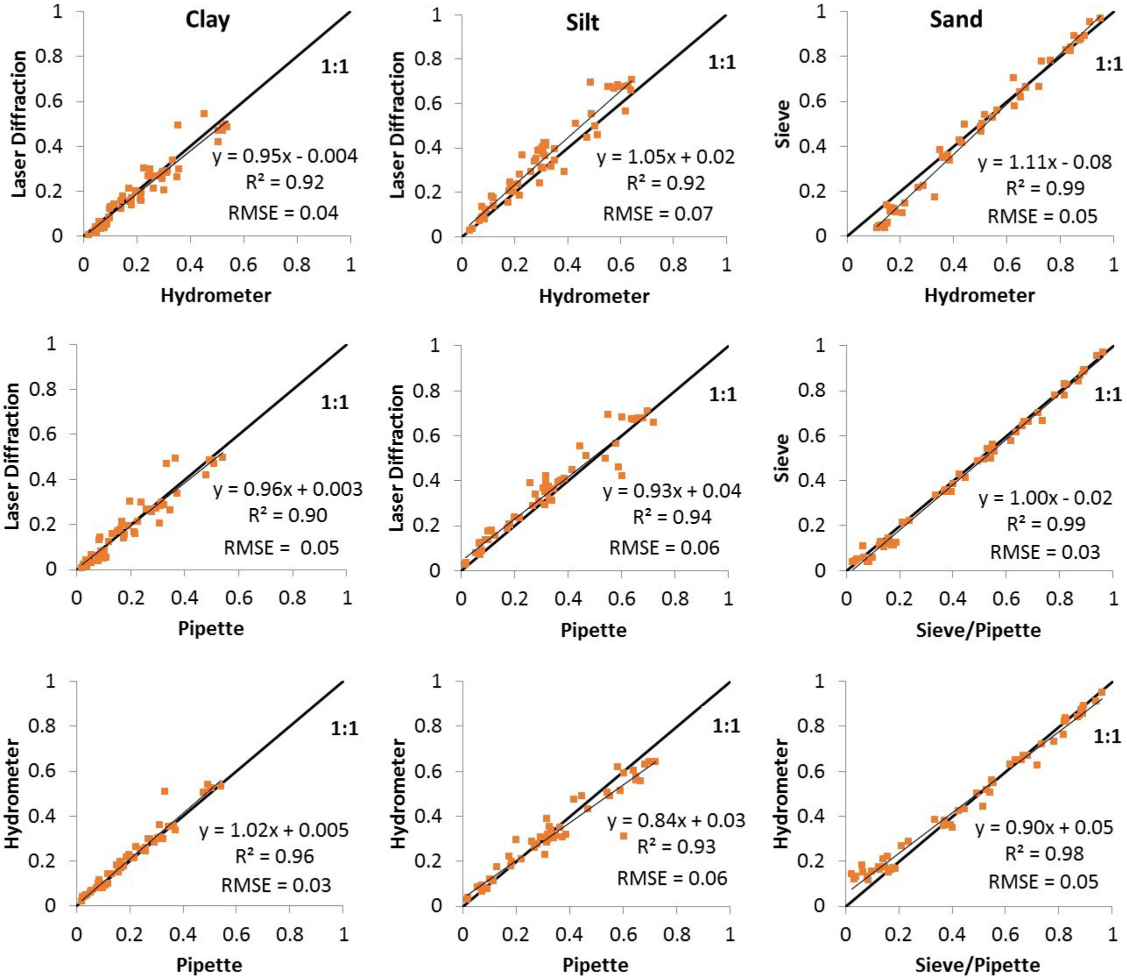

Fig. 2. Regression analysis comparing the proposed laser diffraction protocol and the reported North American Proficiency Test and Agriculture Laboratory Proficiency sieve/pipette and hydrometer for the 54 standard samples selected.

tion of the sand fraction via sieving, as in the sieve/pipette method, (ii) choice of a nominal particle laser diffraction diameter that optimized the matching of clay and silt fractions between the two methods, and (iii) use of the results of the laser diffraction PSA to describe the nominal particle size distribution (i.e., the relative proportion of nominal light scatter particle diameter distribution within the clay and silt fractions).

As indicated by Eshel et al. (2004) and Merkus (2009), one of the advantages of laser diffraction is the rich information of particle size distribution (Fig. 3). Although textural classes provide a useful indication of the particle size distribution, it is well known that soils that have similar fractions of clay, silt, and sand may have contrasting particle size distributions. This is shown in Fig. 3, where one of the samples has a particle size distribution with a large fraction in the finer portion of the clay range. An important additional advantage of laser diffraction over sedimentation methods is the speed of the analysis and reduction of the error introduced by the time of reading and the operator reading the hydrometer level (Allen, 1997; Syvitski et al., 2007). With the described setup, the Mastersizer apparatus throughput was 15 samples per hour, which allowed analysis of 400 soil samples in 2 wk. It would have taken more than 2 mo to do the same analyses using the current lab setup for the hydrometer technique with 11 Bouyoucos cylinders. In addition, by wet sieving the sand from the soil particle suspension, segregation and sedimentation errors are minimized, facilitating the collection of a representative suspension sample as suggested by Taubner et al. (2009).

Eshel and Levy (2007) advocated for building a direct link between laser diffraction PSA results soil properties, as is currently used for sedimentation-based methods, bypassing the need for calibrating results of these methods. The work presented in this study supports that approach. However, there has been minimal progress toward building a direct link between laser diffraction PSA and soil properties since 2007, and it seems that an intermediate matching step is necessary to progress toward that goal.

The laser diffraction protocol presented in this research produced soil PSA results that match standard sedimentation methods within their expected error. Laser diffraction soil PSA is information rich, fast, and robust. Furthermore, the current average texture analysis operating cost of US$ 22 ± 11 (based on informal surveys of 10 US soil laboratories) could be reduced significantly once the initial investment in the laser diffraction equipment is discounted. Additionally, although the potential of laser diffraction and particle imaging methods will continue to expand with advances in sensor technology and machine learning techniques for data mining (Merkus, 2009), there is little room for further technical advancement using standard sedimentation methods. Instead of developing functions to convert laser diffraction analysis results to sedimentation equivalent data (Makó et al., 2017), pedotransfer functions could be developed de novo using laser diffraction PSA results.

CONCLUSIONS

The laser diffraction protocol for soil PSA proposed in this work uses a small soil sample; is more robust, simpler, and faster than the current sedimentation methods; and significantly expands the quality of the data collected from soil texture analysis into a detailed particle size distribution. Limiting the particle size range of the samples by wet sieving the sand fraction overcame the difficulties inherent in obtaining representative samples and stable soil–dispersed suspensions, allowing accurate estimation of clay and silt fractions. The assumptions that form the basis of sedimentation methods were used to develop a protocol that matches results from laser diffraction and standard sedimentation methods for a wide range of soils. Rather than defaulting

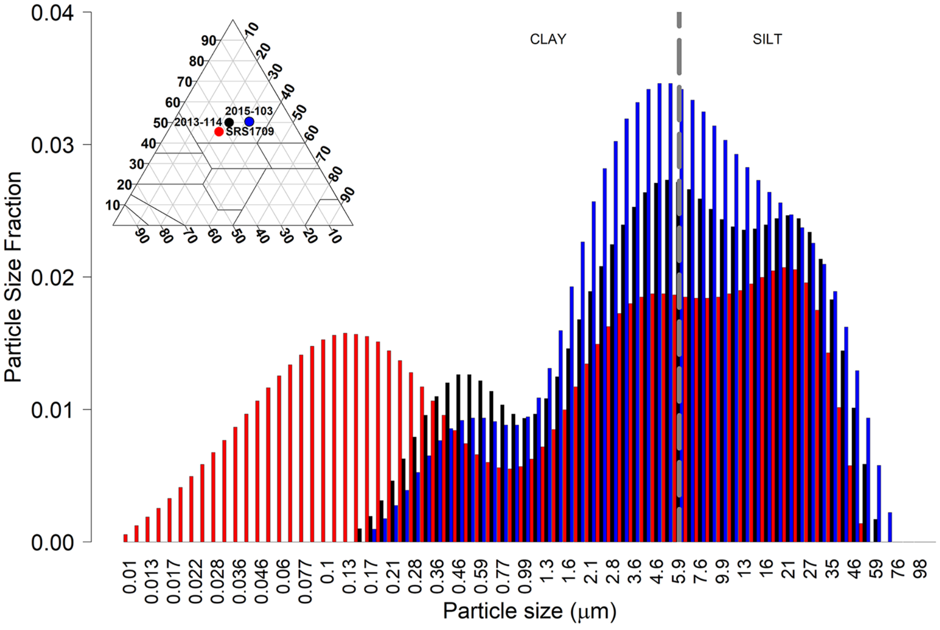

Fig. 3. Particle size distribution determined by laser diffraction of samples 2013-114, 2015-103, and SRS-1709, with the same clay texture but very different particle size distributions. See Table 1 for a detailed sample description.

to standard sedimentation methods, results obtained with the protocol presented here encourage further adoption of laser diffraction methods in PSA.

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