1 2	Measuring shrinkage of undisturbed soil peds
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5	https://doi.org/10.1002/saj2.20318
6 7 9 10 11 12 13	<ul> <li>Core Ideas:</li> <li>We obtained a soil shrinkage curve from single volume and moisture measurements of 200 peds.</li> <li>The method avoids drawbacks of other techniques that disturb samples.</li> <li>Similar SSCs could be produced with as few as 20 undisturbed peds.</li> </ul> Abbreviations: SSC, soil shrinkage curve; COLE, coefficient of linear extensibility.
14	ABSTRACT
15	Methods to measure shrinkage curves typically either disturb natural aggregate structure
16	or include difficult or slow volume measurement techniques. Additionally, most shrinkage
17	curves are obtained by serial measurement of a few samples. We obtained shrinkage curves by
18	collecting rapid, one-off measurements of volume and moisture content for each of 200
19	undisturbed peds extracted from a field soil, taking measurements as peds slowly dried in the
20	laboratory. The large sample size increased robustness of the shrinkage curve parameter
21	estimates to noise generated by this rapid measurement technique, but a much smaller sample
22	would have resulted in similar parameter estimates.
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24	1. INTRODUCTION
25	Expansion and contraction of soil volume with changes in soil moisture affect a range of

driven by repulsive forces arising when water becomes adsorbed between clay particles, a

properties such as soil strength, hydraulic behavior, and ecological relationships. Expansion is

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28 process that is relatively well understood for many clay minerals and affiliated cations (Sposito

et al., 1999; Hensen & Smit, 2002). In contrast, shrinkage and swelling properties of natural soils
are less predictable because of many factors such as interacting thermodynamic processes (Laird,
2006), mixed mineralogy, solutes, and the presence of organic matter. The latter can inhibit
swelling when adsorbed to clay surfaces or flocculating particles (Yariv, 2002) but can also
increase swelling and resist shrinkage when organic matter creates macropores (Peng & Horn,
2007).

This complexity has led to an emphasis on empirical characterizations of the shrink-swell 35 behavior of natural soils, primarily through the soil shrinkage curve (SSC). The SSC quantifies 36 37 the relationship between soil moisture ratios and volume ratios throughout the phases of soil shrinkage (e.g., Sposito, 1973; McGarry & Malafant, 1987; Braudeau et al., 2004; Peng & Horn, 38 2013). However, in addition to moisture content, expansion and contraction also depend on 39 temperature (Kittrick, 1969) and overburden pressure (Talsma, 1977; Bronswijk, 1990), and are 40 hysteretic (Groenevelt & Bolt, 1972; Tambach et al., 2006; Peng & Horn, 2007). Therefore, 41 detailed interpretation of physical processes from SSCs is not always possible, and simplified 42 model forms for SSCs have been developed for practical use (e.g., Stewart et al., 2016a). 43 Various methods of measuring soil shrinkage and SSCs have been developed, based on 44 45 samples that are ground then repacked (Boivin et al., 2004) or made into a paste (Schafer & Singer, 1976b; Simon et al., 1987; Chertkov, 2003), core samples (Yule & Ritchie, 1980; 46 Grossman & Reinsch, 2002), or intact clods (Brasher, 1966; Tariq & Durnford, 1993). Of these, 47 48 ground and core samples are the easiest to manipulate and measure, whereas intact clods maintain the most native properties of soils that SSCs seek to capture. Methods to control soil 49 50 moisture and measure dimensions and water contents of clods have varied widely, each with 51 their own advantages and disadvantages.

52 Typically, SSCs are constructed by repeatedly measuring volume as soil clods dry. Measuring volume using fluid displacement is a common approach of doing this, but preventing 53 that fluid from altering soil moisture is difficult. Brasher (1966) inhibited infiltration during 54 volume measurement by coating samples in resin, and Tariq and Durnford (1993) encased 55 samples in balloons and applied a vacuum to force balloons to conform to samples. These 56 techniques both have drawbacks, including imperfect prevention of infiltration, imperfect 57 adhesion of coatings to clod exterior, and interference with the shrink-swell properties the tests 58 are intended to measure (Schafer & Singer, 1976; Sander & Gerke, 2007). More recently, sample 59 60 volumes have been measured using multiple-perspective photography (Stewart et al., 2012a) and 3D scanning (Rossi et al, 2008; Sander & Gerke, 2007; Wong, 2019). These techniques are 61 generally more precise than volume displacement but require more complex equipment and 62 analyses. At the same time, nearly all published techniques to measure shrink-swell properties 63 rely on relatively few samples. Thus, many SSCs carry little information about the variability of 64 the soils they seek to characterize. 65

In this paper, we present a method of measuring soil shrinkage that circumvents many typical measurement problems and facilitates construction of SSCs from large numbers of undisturbed samples, with the goal of producing a SSC that is representative of natural, undisturbed soils. In short, we measured volume and moisture content of a large number of samples, each only once, and used this data to construct a SSC. We compare the resulting SSC to existing SSCs obtained using other techniques and quantify the statistical stability of the obtained SSC for smaller subsets of data.

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### 74 **2. METHODS**

## 76 **2.1 Sample collection**

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Soil samples were collected from the floodplain of the Mississippi River (30.282° N, 78 91.089° W) in Iberville Parish, Louisiana. The soil is mapped by USDA-NRCS as Sharkey clay, 79 a very-fine, smectitic, thermic Chromic Epiaquert, which, together with its hyperthermic version 80 Schriever clay, extends over more than 16,700 km<sup>2</sup> of the alluvial valley of the Mississippi 81 River. The soil is well structured, composed of weak, medium (<30 mm) peds that were 82 83 subangular and blocky or wedge-shaped with slickenside boundaries typical of Vertisols. The collection site was forested, with tree species dominated by sugarberry (*Celtis laevigata*), green 84 ash (Fraxinus pennsylvanica), and American elm (Ulmus americana). 85 The basic units of measurement for this study were 200 naturally formed peds extracted 86 with minimal deformation in the laboratory from two neighboring, 19 L, intact, cylindrical soil 87 monoliths. The soil monoliths were obtained in the field from 15 to 60 cm in depth (omitting the 88 organic-rich surface layer). This was done by removing the soil surrounding the samples to the 89 desired depth, then gently picking away at the edges of the monolith until a 19 L (5-gallon) 90 91 bucket could slide over it. With the bucket over the monolith, a shovel was used to pry the sample from the ground, then the monolith was immediately covered with plastic wrap to reduce 92 evaporation. Organic matter was 1-3%, estimated by loss on ignition at 550°C; texture was 2% 93 94 sand, 60% silt, and 38% clay ( $\leq 2.00 \,\mu$ m), estimated by laser diffraction, though the silt was very fine and the clay fraction was ~53% when corrected for bias compared to the sieve-pipette 95 96 method (Morales et al., 2021).

### 98 2.2 Sample analysis

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Individual peds, averaging 17.6  $\text{cm}^3$  and ranging from 6.47 to 30.7  $\text{cm}^3$ , were removed 100 101 from the large monoliths for analysis by picking with a knife, which caused the soil peds to naturally break away. Fifteen to twenty sample peds were extracted every 3-5 days for almost 102 two months, and were analyzed for volume, mass, and gravimetric moisture content. Each ped 103 was individually placed into a 250 mL overflow beaker for 2 minutes and the mass of water 104 displaced by the ped out of the overflow beaker was recorded. The contents of the beaker (water, 105 106 ped, and any slaked soil) were then poured through a coffee filter that had previously been weighed. The filters and soil samples were then dried at 105°C for 48 hr and the oven-dry mass 107 of each sample was obtained. 108 109 The soil monoliths were nearly at field capacity when they were collected and left loosely covered with plastic wrap between sample ped extractions to allow the soil to slowly dry and 110

shrink. We wanted to characterize the SSC for the typical range of soil moisture for these

112 lowland sites, which experience frequent rainfall and are often connected to shallow

113 groundwater, so we omitted the dry end of the SSC.

114 The moisture ratio,  $\vartheta$  (volume of water / volume of solids) of each ped, was obtained as 115

116 
$$\vartheta = \frac{(W_f - W_o)/\sigma_w}{W_o/\sigma_c}, \tag{1}$$

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118 where  $W_f$  and  $W_o$  are mass of fresh and oven-dry peds, respectively, and  $\sigma_w$  and  $\sigma_c$  are density of 119 water and clay, respectively. The void ratio, *e* (volume of voids / volume of solids), was obtained 120 as

$$e = \frac{V - W_o / \sigma_c}{W_o / \sigma_c},$$
(2)

where *V* is total volume of the ped. We assumed densities of  $\sigma_w = 1.00 \text{ g} \cdot \text{cm}^{-3}$  and  $\sigma_c = 2.75$ g·cm<sup>-3</sup> (Flint & Flint, 2002).

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### 127 **2.3 Volume measurement calibration**

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129 Measuring volume using an overflow beaker requires a correction factor because overflow continues for tens of seconds as the final drips overcome surface tension, generating 130 131 error (Hughes, 2005). To reduce this error, we performed an experiment in which we added known volumes of water to the overflow beaker and measured the mass of the outflow every 10 s 132 for 2 min. We used the results to standardize time in the overflow beaker and to obtain a 133 correction factor to estimate volumes. Based on results, we standardized submersion time for 134 each ped at 2 min and added 6.6% to the estimated volume of each ped. 135 136 2.4 Modeling and statistical analysis 137 138 Given that an important motivation of this work is to fit practically applicable SSCs 139 created from soils that were collected under field conditions instead of theoretically detailed 140 SSCs generated by imposed lab conditions, we fit the simple model form derived by Stewart et 141 142 al. (2016a) to our data, as

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$$\phi_{\text{ped}}(U) = \left(\phi_{\max} - \phi_{\min}\right) \left(\frac{\varepsilon + 1}{\varepsilon + U^q}\right) + \phi_{\min} , \qquad (3)$$

where  $\phi_{\text{ped}} = e/(1+e)$  is porosity of the ped at normalized moisture ratio  $U = \vartheta/\vartheta_{\text{max}}$  and  $\varepsilon$  and q146 147 are fitting parameters related to structural and residual shrinkage, respectively. The bounds  $\phi_{max}$ ,  $\phi_{\min}$ , and  $\vartheta_{\max}$ , are practical limits under the assumption that soils are within the range of typical 148 field moisture and relatively non-hysteretic equilibrium in the field following repeated shrink-149 150 swell cycles (as described by Tripathy et al. 2002). We chose values of  $\phi_{\min} = 0.35$  ( $e_{\min} = 0.53$ ) from a separate test of the bulk density of oven-dried soil,  $\phi_{max} = 0.73$  ( $e_{max} = 2.70$ ) empirically 151 from our data, and assumed  $\vartheta_{\text{max}} = e_{\text{max}}$  (complete occupation of voids by water; Peng & Horn, 152 153 2005; Stewart et al., 2016a). We fitted  $\varepsilon$  and q numerically, using a Gauss-Newton numerical iteration in SAS 9.4 (SAS Institute; Cary NC, USA) to minimize sum squared error for the model 154 155 fit to data.

To examine the sensitivity of  $\varepsilon$  and q to sample sizes smaller than the full 200 peds, we also fitted  $\varepsilon$  and q for 30 subsamples each of sizes n = 10, 20, ..., all peds, with each subset consisting of n observations chosen randomly, without respect for sample moisture, and without replacement from the full dataset.

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#### 161 **3. RESULTS**

162 The combination of the choice to use a large number of peds, combined with the rapid 163 water displacement method to estimate ped volume and moisture, resulted in some outliers 164 (Figure 1). We excluded thirteen nonphysical datapoints (i.e., with measured  $\vartheta > e$ ) but retained 165 all others. Filtering data to further exclude more outliers resulted in small changes to parameter 166 estimates for the SSC. In short, the method proved robust to outliers.



169 <u>Figure 1.</u> Soil shrinkage curve of Stewart et al. (2016) fitted to observations of individual ped 170 moisture ratios and void ratios. Open circles indicate nonphysical results that were removed 171 before curve fitting. Inset is  $\kappa$ , the second derivative of the soil shrinkage curve, plotted as  $-\kappa$ 172 following Groenevelt and Grant (2001).

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The best-fit SSC we obtained ( $\varepsilon = 3.2$ , q = 1.5) was dominated by peds with  $\vartheta$  between 0.9 and 1.4. Several weeks of slow drying in the laboratory produced no samples where  $\vartheta < 0.5$ . In the range we measured, the soil was undergoing proportional shrinkage, where  $\vartheta$  and e were nearly linearly related with slope near 1. Behavior of the wettest samples suggests the possibility of structural shrinkage (slope < 1). However, there was no inflection in the second derivative of the SSC ( $\kappa$ ) that has been used to identify the transition point between proportional and structural shrinkage (Figure 1 inset), possibly because data were too sparse in this region for robust interpretation. The fitted curve indicated a transition between proportional and residual shrinkage by an inflection point in  $\kappa$  near  $\vartheta = 0.5$ , but lacking any data for drier samples, this inflection point is only conjectural.

The SSCs generated using randomly selected subsamples of peds resulted in similar curve shapes as the full dataset of 200 peds (Figure 2). However, using fewer peds resulted in greater variability and some aberrational curve fits (e.g., curves generated using 20 peds; Figure 2a). The least variation in the fitted curves across the subset fits was near  $\vartheta = 1.0$  where the data were densest. The best-fit SSC model parameter sets were influenced by aberrational fits for smaller sample sizes (Figure 3). Increasing the number of sampled peds n = 70 for *q* and n = 110 for  $\varepsilon$  was required for  $\pm$  one standard deviation limits to become <10% of the parameter values.



193 Figure 2. Realizations of fitted soil shrinkage curves from 30 subsets of (a) 20, (b) 40, and (c)

194 120 peds per subset; each subset consisted of peds selected randomly without replacement. Each

195 panel contains 30 lines.



Figure 3. Effect of sample size on parameter estimates of (a) ε and (b) q, obtained from 30
subsets of peds of varying sizes selected randomly without replacement. Circles indicate mean
and solid lines indicate ± one standard deviation of estimates obtained in each 30-subset
population.

# **4. DISCUSSION**

Our method of creating SSC by collecting rapid, one-off measurements of volume and 204 moisture content for each of 200 undisturbed peds produced results comparable to previous 205 studies (Tariq & Durnford, 1993; Sanders & Gerke, 2007; Stewart et al., 2016a). However, 206 because our method is based on multiple peds, it may produce different results compared to 207 traditional measurements, which typically use a single ped to generate SSCs or to calculate 208 209 simplified shrinkage indices such as the coefficient of linear extensibility (COLE). As an example of the latter, the  $e_{\text{max}}$  and  $e_{\text{min}}$  values used while fitting our SSC curve (Figure 1) 210 resulted in a relatively large estimate for COLE: 0.30 for our samples, compared to 0.14-0.18 for 211 212 whole-soil COLE measured by the National Cooperative Soil Survey (NCSS) in a nearby soil pit (Pedon ID: 88LA047002). This discrepancy can likely be attributed to uncertainty in our 213 214 constrained  $e_{max}$  and  $e_{min}$  values. For one, using multiple samples increased the likelihood of 215 outlier values, either by bad measurements or inherent soil variability. Estimating  $e_{\text{max}}$  and  $e_{\text{min}}$ from clusters of data at the upper and lower extrema of the SSC, rather than the individual 216 217 extreme values, yields a COLE  $\approx 0.19$ , which is much closer to the NCSS-measured values. For another, our method does not allow for control of the water potential of the samples, making it 218 impossible to determine the exact sample volume (and void ratio) associated with 1/3 bar 219 220 potential, which is used in traditional COLE measurements (Soil Survey Laboratory Methods Manual, 1996). Finally, the peds used in this study were nearly six times smaller than the saran-221 coated clods analyzed by the NCSS (i.e., ~100 cm<sup>3</sup>). Oven-dry bulk densities are often over-222 223 estimated when using relatively small soil samples, as these sample exclude crack space that forms within and between larger clods (Tisdale, 1951). Future users should keep these 224 225 differences in mind when reporting COLE values using this method. At the same time, additional

studies should investigate relationships between ped-based estimates of the SSC versus thosefitted using other methods.

Natural variability in the studied field soil plays an important role in the estimated SSC 228 229 using this multiple-ped technique. The resulting characteristics of the SSC can be considered assets or liabilities depending on eventual use of the SSC. For instance, if using the SSC as the 230 231 basis for field water flux modeling (e.g., Stewart et al., 2016b), parameter estimates based on multiple samples at different  $\vartheta$  would be desirable. Our subsampling provided mostly consistent 232 SSC curves and parameters with as few as 20 peds and no constraints on ped moisture content; 233 234 designing the data collection to ensure a wide range of water contents could further reduce methodological uncertainty. On the other hand, the SSC we obtained is more statistical than 235 physical because each ped was measured only once. Therefore, the data may be less useful for 236 robust parameter estimates for curves with multiple inflection points that allow extraction of 237 physically meaningful parameters. Heterogeneities in, for example, organic matter, could smooth 238 239 out inflection points and make them difficult to identify.

The SSC we obtained is not fully representative of shrink-swell behavior in the field at 240 macro scale because all data we obtained was for the unloaded state, and because we used 241 242 natural aggregates (peds) as samples rather than a full solum or natural clod that includes cracks. For this reason, in situ methods that characterize crack dimensions and connectivity may be 243 necessary for robust hydrological modeling in expansive clay soils (e.g., Favre et al., 1997; 244 245 Stewart et al., 2012b; Stewart et al., 2013; Ackerson et al., 2017). Nonetheless, the proposed method is low cost and easy-to-use, and therefore can be widely used by those interested in 246 247 characterizing the shrinkage behaviors of natural soils.

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## 249 **CONFLICTS OF INTREST:** The authors declare no conflict of interest.

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