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A COMPARISON OF SOIL SAMPLE HOMOGENIZATION TECHNIQUES

#### by

B. A Schumacher, K. C. Shines, J. V. Burton, and M. L. Papp Lockheed Engineering and Sciences Company, Inc. Las Vegas, Nevada 89119

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Project Officer

L. J. Blume Exposure Assessment Research Division Environmental Monitoring Systems Laboratory Las Vegas, Nevada 89193-3478

ENVIRONMENTAL MONITORING SYSTEMS LABORATORY OFFICE OF RESEARCH AND DEVELOPMENT U.S. ENVIRONMENTAL PROTECTION AGENCY LAS VEGAS, NEVADA 89193-3478

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#### **ABSTRACT**

**A** study **was conducted using the most common homogenization techniques, namely, grinding and sieving, riffle splitting (open- and closed-bin), and cone and quartering to homogenize four soils of differing textures to determine the effectiveness, efficiency, and extent of fine particle loss from each technique by examining particle-size distribution, loss on ignition organic matter, and pH. Five passes should be used to effectively homogenize a sample since the least replicate variability almost always occurred after the fifth pass. Riffle splitting was more efficient and had less loss of fines than cone and quartering and is therefore the recommended method for soil homogenization, The closed-bin riffle splitter had a greater apparent ability to contain the loss of fines than the open-bin riffle splitter. Grinding and sieving (random sampling) was the most efficient process yet almost consistently showed greater replicate variability than the other homogenization techniques after the fifth pass thereby reducing its value for soil homogenization.** 

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#### **INTRODUCTION**

**The need for sample homogeneity prior to laboratory analyses has been long recognized by geologists, geologists, chemists, and members of other scientific disciplines. Homogeneity is the degree that the material under investigation is mixed resulting in the random distribution of all particles in the sample. Completely homogenous materials are so rare that they may be considered nonexistent (Ingamells and Pitard, 1986) yet scientists must strive to obtain a homogenous sample in order to obtain data exhibiting minimal error attributable to sample heterogeneity.** 

**Care must be taken during the subsampling phase of soil preparation, during sample transport, unpacking, and transfer to other containers in the laboratory to avoid particle sorting via different particle densities, shapes, sizes, and resistance of certain minerals to mixing, such as magnetite (Muller, 1967; Ingamells and Pitard, 1986; Reeves and Brooks, 1978). Numerous methods have been used to obtain homogeneous soil samples. The various methods range from simple grinding and sieving of the sample to a desired particle-size to various mixing and splitting devices and machines. Each method will be discussed individually with both advantages and disadvantages being presented.** 

**One of the most common methods used to obtain a homogeneous sample is to grind and sieve the soil to a desired particle-size (generally <2-mm) followed by random sampling. This method assumes that the initial material is ground without preference to any given factor, such as color, and that during grinding and sieving, the sample becomes sufficiently homogenized. To further eliminate possible heterogeneity within the sample and to reduce the sample size to the desired quantity for a given analysis, subsamples may be obtained by "spooning" (Carver, 1961) or some other method which involves the random insertion of a spoon or other sampling device into the previously ground and sieved sample. This method is preferably performed rapidly and without extensive visual examination of the sample that could lead to a processor preference in certain cases, e.g., light catching the shiny surfaces of mica flakes leading to preferential inclusion or exclusion of that part of the sample. The "spooning" type of subsampling markedly reduces the time of sample processing in comparison to multiple, successive splitting operations and has been shown to produce equivalent correlation coefficients between observed settling velocities of sands (median r=0.994 using other splitting methods and median r=0.9955 using the spooning technique) when used to obtain 20 gram subsamples from initial 1 to 2 kg sand samples (Carver, 1981).** 

**Two other methods have been devised which are similar to the "spooning" method except they involve an intermediate step between grinding and sieving and subsampling procedures. Gilliam and Richter (1965) used an intermediate step of stirring the sample with a spatula, presumably until visual homogeneity was obtained, before subsampling. Some analysts content themselves by merely shaking the sample in a bottle prior to subsampling and ignore the risk of constituent segregation (Reeves and Brooks, 1978).** 

**An additional method of sample subdivision with the goal of obtaining a representative sample was presented by Allman and Lawrence (1972). Their method is similar to the "spooning**\* **method of Carver (1961) except that a scoop of ground and sieved materials was divided among four containers. The process was repeated continually changing the filling order of the containers until the sample had been quartered or an appropriate sample size had been obtained. As with the spooning process, visual bias as to how much sample and into which container the samples were placed is a concern.** 

**Before proceeding to the more elaborate sample splitting schemes, a discussion of methods used simply to mix (homogenize) the ground and sieved sample is warranted. The simplest of the homogenization processes** is tumbling the sample on a sheet of paper, cloth, or plastic. This process involves the manual rolling of ground **samples such that the sample must tumble upon itself and not just slide along the surface of the sheet (Schuler, 1971; Van Johnson and Maxwell, 1981; Ingamells and Pitard, 1986). This method is effective on sample sizes less than 2 kg, yet** 



#### **Fig. 1. A twin shell V-blender,**

**caution must be taken to ensure that the sheet material does not contain any element which is to be quantitatively analyzed and does not develop static charges that may lead to segregation of the finer particle sizes.** 

**Homogenization may also be achieved through the use of mechanical mixing devices including a spiral mixer, a cement mixer, and a twin shell V-blender. The spiral mixer involves the rotation of the bottled sample in both horizontal and vertical planes (Van Johnson and Maxwell, 1981). The cement mixer or similar devices involves the rotation of the sample in a chamber with a series of internal baffles that cause the materials to be thoroughly tumbled and mixed. These two methods are useful for samples ranging from less than one pound to several hundred pounds. The twin shell V-blender involves the rotation of two hollow cylinders about an horizontal axis such that the apex describes a circle in the vertical plane (Schuler, 1971). Twin shell blenders are available commercially in sizes ranging from 4 to 16 quarts (approximately 10 to 40 kg of a mineral soil) internal capacity (Fig. 1). However, Ingamells and Pitard (1966) expressed serious doubts as to the value of mechanical splitters. They stated that, 'In general, machines that use mechanical violence and look and sound as though they are efficient are most likely to cause segregation of heavy and light, large and small, and flat and round particles**\***. Further concerns have been expressed concerning sample breakdown to finer particle sizes due to the violent tumbling in the machines.** 

**The riffle splitter (also called a chute splitter, Jones splitter, or sample splitter) is perhaps the most common mechanical method for sample homogenization and/or sample size reduction (Ingram, 1971, Mullins and Hutchison, 1982). The riffle splitter also provides one of the best general methods of sample mixing to obtain bulk sample homogeneity (Ingamells and Pitard, 1986). A riffle** 



**Fig. 2. An open-bin riffle splitter. Fig. 3. A closed-bin riffle splitter.** 

**splitter is a device having an equal number of narrow sloping chutes with alternate chutes discharging the sample in opposite directions into two collection bins (Figs. 2 and 3). Sample homogenization is achieved by repeated pouring of soil through the splitter and combining the halves between passes. The use of the riffle splitter as a subsampling device is done in a similar manner with the exception that after the sample is passed through the splitter, one collection pan is replaced with a clean pan. The material in the 'replaced" pan, which contains about one-half of the original sample, is then passed through the riffle splitter again thereby reducing the volume in the clean pan to one-quarter of its original sample volume. This process of sample reduction is repeated until the desired weight or sample size is obtained.** 

**Many variations on the size and construction materials have been built on the principle of the riffle splitter. For small samples (10 to 60 grams), an efficient microsplitter has been designed by Newton and Dutcher (1970) using balsa wood and glass microscope slides. These authors conducted experiments using 40 gram samples of the fine sand fraction and found only an 1.2% average error when quartering the sample by a P-step halving. The results of Newton and Dutcher (1970) are supported by the earlier work of Griffiths (1953) who found that sample homogenization using a riffle**  splitter produced an overall coefficient of variance (C<sub>v</sub>) between median grain sizes of less than 3% in 9 out of 10 samples run on various rock and sand samples. In the one case where the C<sub>y</sub> was greater than 3%, the **error was attributed to operator differences (16 in the single case vs. 5 or less in the other nine cases). A portable sieving and splitting device for field use has been designed by Ibbeken (1974) which uses the riffle splitter to subsample and process coarse grained sediments. During his research on unconsolidated sediments, no significant differences were found between splits in terms of petrographic mineralogical composition, with the exception of the 125 to 160 mm serpentine fraction (represented by only four pebbles), nor in grain size distribution. Another variation of the riffle splitter found in tile literature was the use of a Single piece of tin-plate bent several times to form a riffle splitter (McKinney and Silver, 1956). The** 



**Fig. 4. The cone and quarter technique.** 

**advantage of this construction method is that free grain flow in the chutes was obtained, without the hindrance of unevenly soldered joints that may be present in other riffle splitters.** 

**The use of riffle splitters and their variations are valuable in splitting samples which range in size from less than 10 grams (Humphries, 1961) to several kilograms (Van Johnson and Maxwell, 1981). Ibbeken (1974) reported that 0.5 to 1 ton (455 to 909 kg) of sample can be processed daily using a standard riffle splitter to reduce the initial quantity to 5 kg subsamples. Although most authors find the use of the riffle splitter to be an effective, efficient method for sample homogenization and sample splitting, several problems exist.** 

**The major source of error involved in using a riffle splitter is the loss of fine particle sizes by "dusting" when processing air-dried or oven-dried samples which contain fine particle sizes (Van Johnson and Maxwell, 1981). The process of riffle splitting requires that a uniform stream of material be poured into the mouth or top of the splitter. Dust loss may occur through the chute ends, in the collection bins, and through the mouth openings (Fig. 2) in an open-bin system or just through the mouth if a closed-bin system is used (Fig. 3).** 

**Several studies have been conducted comparing the effectiveness of the riffle splitter to other homogenization techniques. Wentworth et al. (1934) compared the riffle splitter with a rotary splitter (to be discussed) and found that the rotary splitter more accurately split an initial sample of known grain size distribution into subsamples with similar grain size distributions than the riffle splitter. Kellagher and Flanagan (1956) in a comparison experiment among a multiple-cone splitter (to be discussed), cone and quartering (to be discussed), and a riffle-based microsplitter found the microsplitter was the worst for both accuracy and precision of grain frequency percentages for subsampling three different weights (5, 10, and 20 grams) of an artificially created very coarse and coarse sand fraction mixture. In a similar type of study, Mullins and Hutchison (1982) compared**  the C<sub>v</sub> among sand fraction contents of several soils and ranked, in order of best to worst, the



**Fig. 5. A rotary splitter.** 

**rotary subsampling, riffle splitting, cone and quartering, and spoon sampling, in terms of their ability to homogenize a sample. These authors did note, however, that the best and worst methods were only significantly different at the 10 percent level.** 

**Perhaps the best known sample splitting method is the classical cone and quarter technique, This technique involves pouring the sample into a cone, flattening the cone, dividing the flattened cone into four equal divisions (quartering), and then removing 2 opposite quarters (Fig. 4). The remaining two quarters are repiled into a cone and the process is repeated until the desired sample size is obtained. Variations on the process are possible which can enhance the speed of sample size reduction by using just one quarter (chosen at random) to continue the splitting process or which allow this method to be modified to homogenize a large sample. The use of the cone and quarter method to homogenize a sample involves the removal of the first quarter and repiling it into a cone followed by the subsequent repiling of the opposite quarter and then the remaining two quarters to reform a single cone (Raab et al., 1990). This process is repeated several times until sample homogeneity is achieved.** 

**Several sources of error for this method have been identified. Van Johnson and Maxwell (1981) reported that during the cone and quarter process on large samples (several kilograms), there is the danger of unequal segregation of heavier materials during the flattening and coning of the sample. Similar to the riffle splitting techniques, dusting is also a possible source of error during cone formation. Sample loss from the inability to recollect all the soil from the underlying material, the ability of the sample to "cling" to the underlying material via static charges, and sample embedment are all further sources of error. Muller (1967) placed a limitation on the cone** 



**Fig. 6. A multiple-cone splitter,** 

**and quarter technique to samples greater than 50 grams and stated that this method is most successful in the field for sample processing of larger sample sizes. Raab et al., (1990) used the cone and quarter method to homogenize large volumes of soil (450 kg) and then subsampled the repiled cone with a 2-foot long plastic tube (Z-inch internal diameter). They then sieved the sample into 3 particle size classes (2-mm to 0.105 mm, 0.105 mm to 0.053-mm, less than 0.053-mm) and subsampled using a riffle splitter. A resieving to check particle class weights of the three divisions between the six subsamples resulted in the finding that no significant difference (error level = .05) among the subsamples indicating a homogenous mixture had been obtained.** 

**Several other sample splitting/homogenizing devices found in the literature include a rotary splitter (Muller, 1967, Schuler, 1971; Allman and Lawrence, 1972), a brass disc microsplitter (Brewer and Barrow, 1972), and a multiple cone splitter (Kellagher and Flanagan, 1956). The rotary splitter involves the pouring of the sample into a feeding hopper located above a rotating disc containing sample bottles or pans (Fig. 5). The disc is mechanically rotated and the sample is divided among the collecting bottles or pans. The brass disc microsplitter was designed by Brewer and Barrow (1972) for subsampling small particulate samples which range up to 1 gram in the particle size range between lo and 200 microns. This splitter divides the sample by passing it through a fluted** 

**brass disc which contains several holes to separate the sample into different collection bottles. The device also has a mechanical vibrator system to ensure complete collection of the fine particles, The multiple-cone splitter of Kellagher** and Flanagan (1956) consists of three funnels and brass cones vertically mounted which **are capable of splitting small** samples (10 to 400 grams) **into 5 mg subsamples which are collected in one of four divisions in the base pan (Fig. 6). These authors statistically compared the precision and accuracy of grain frequency percentages obtained using their device with a riffle-based microsplitter and the cone and quarter technique and found that their splitter was better in both categories than the riffle splitter while being better than the cone and quartering in terms of accuracy only.** 

**Due to the overwhelming concurrence (although not unanimous) in the literature as to the value of the cone and quarter and riffle splitting techniques in sample homogenization and the common use of random sampling after grinding and sieving of the soil, investigations were undertaken to determine the effectiveness (degree of homogenization), efficiency (time consumption), and the extent of the loss of fine particles by these methods during the homogenization of large soil samples.** 

#### **MATERIALS AND METHODS**

**Bulk samples (approximately 15 kg) of surface horizons of the Overton clay (fine, montmorillonitic, calcareous, thermic Mollic Haplaquepts), Gila silt loam (coarse-loamy, mixed, calcareous, thermic Typic Torrifluvents), Calico loam (coarse-loamy, mixed calcareous, thermic Typic Torrifluvents), and Jean gravelly loamy fine sand (sandy-skeletal, mixed, thermic Typic Torrifluvents) were collected in Clark County, Nevada (Speck, 1965). Surface horizons with different textures were selected to represent soil systems with varying sensitivities to the loss of fines that may occur during sample homogenization.** 

**Moist bulk samples were split into three subsamples (approximately 5 kg each) and air-dried. Samples were ground and sieved through a 2-mm (10 mesh) sieve with three random samples being collected from each of the subsamples Prior to any homogenization procedures. The three original subsamples from each soil series were "homogenized" by either passing the soil seven times through the cone and quarter technique, an open bin riffle splitter, or a closed bin riffle splitter. Three samples of approximately 60 g each were collected after the first, third, and fifth passes. Seven samples were collected after the seventh pass through each of the homogenization processes. Samples were collected from the riffle splitters by placement of the receiving bottle under randomly selected chutes prior to the sample being poured through the splitter. Samples from the cone and quarter technique were collected by pouring the sample over the collection bottles during cone rebuilding and subsequently removing the bottles when they were full prior to completion of the cone using all four quarters.** 

**A timed experiment was conducted to determine the efficiency of each splitting technique and involved the passing of the previously "homogenized" soil (from the seventh pass) through the three homogenization procedures seven times without collection of soil samples after any intermediate passes. Sewn samples were collected after the timed experiment in which the soil had now been passed fourteen times through the homogenization process.** 

**Three parameters were selected to determine the effectiveness of the homogenization method and loss of fines from the system, namely, particle-size analyses (for loss of inorganic fines), loss on ignition (LOI) organic matter (for organic fine losses) and pH (for bulk chemical changes). Particle-size distribution (<2-mm) was determined using the pipette method described by Gee and Bauder (1986). Soil samples were oven-dried at 110**\* **C** overnight and loss on ignition organic matter content was determined gravimetrically after heating at 450° C for a minimum of 6 hours. The pH values were determined in a 1:2 soil:0.01 *M* CaCl<sub>2</sub> solution ratio (McClean, 1982). **Levels of confidence were determined by analysis of variance (ANOVA).** 

#### **RESULTS AND DISCUSSION**

**It should be noted that pass no. 0 represents the random samples collected prior to any sample homogenization process other than preparatory grinding and sieving. Complete data tables containing individual analyses of sand, silt, clay, sand fractions, pH (no standard deviations or %RSDs presented), air-dry moisture contents, and LOI organic matter as well as means , standard deviations, and %RSDs for replicate and cumulative data are presented in the Appendix**.

#### **Observed Sources of Soil Loss**

**Loss of fines via dusting was most apparent during the use of the open bin riffle splitter. Dust loss occurred from the mouth of the riffle splitter due to the air-dried soil hitting the baffles and sliding down the chutes as well as in the collecting bin from the soil falling upon itself, Apparent fine particle losses from use of the closed bin riffle splitter were less noticeable than for the open bin riffle splitter. Dust losses occurred through cracks within the riffle splitter and through the mouth into which the soil was being poured. However, although the overall dust loss appeared** to **be less, additional soil loss was observed within the closed bin riffle splitter where soil collected on internal ledges and around the outside of the collecting bins. Little loss of fines, via dusting, was observed using the cone and quarter technique. Dusting occurred only during the piling of the quarters upon each other to form a new cone. Soil losses were observed due to an inability to completely transfer all soil materials during new cone formation.** 

#### **Influence of Sample Homogenization on PH**

**No significant differences were found in pH values among replicates regardless of soil texture, splitting method, or the number of the pass from which the subsample was obtained (Table 1). All replicate pH values met the intralaboratory precision goals set for the Mid-Appalachian soil survey (0.10 pH unit). The ranges in pH for the Jean gravelly loamy fine sand and Calico sandy loam were 7.7 to 7.8. The Gila silt loam had pH values of 7.8 in all samples while the Overton clay had pH values ranging from 7.9 to 8.0.** 

#### **Influence of Sample Homogenization on Particle-Size Distribution**

**All replicate total sand, silt, and clay contents met the intralaboratory precision goals set for the Mid-Appalachian soil survey of 3.0 wt% standard deviations for sand and silt; 2.0 wt% standard deviations for clay. Standard deviations for total sand, silt, and clay contents ranged from 0.05 to 1.30, 0.05 to 2.59, and 0.00 to 200, respectively (Table 2).** 

**The lowest standard deviations among replicates were almost always found after the samples had been passed five times through the homogenization process regardless of homogenization technique used or soil texture (Table 2). When exceptions did occur, the standard deviations were not significantly different from the fifth pass and occurred in either the random sample or after the first pass through the homogenization process. In the later case, the sample was simply halved via riffle Splitting or was effectively a random sample from the first flattened quarter for the cone and quarter technique. At the 90% or greater confidence interval, the random (pass 0), first, third, seventh, and timed experiment passes had 36, 33, 53, 86, and 97%, respectively, of the samples having significantly greater replicate variances than the fifth pass. If a 75% or greater confidence limit was used, pass 0, 1, 3, 7, and T had 53, 56, 78, 98, and 100% of the samples, respectively, had significantly greater variances than the fifth pass. Earlier passes (pass 0, 1, and 3) resulted in fewer samples with significantly different variances among the replicates than in passes after the fifth, yet one-third to more than one-half of the samples had significantly greater variances than found after five homogenization passes. These data indicate that any attempts to further homogenize the soil after the fifth pass through the various homogenization methods markedly increased the variability among replicates and thus the heterogeneity of the sample regardless of soil texture or homogenization technique.** 





 $a =$  data presented are the means of 3 replicates for splits 1 through 5 and means of 7 replicates for splits 7 and T.

 $\mathbf{Q}$ 



#### **Table 2. Standard deviations between replicate analyses for particle-size distribution and loss on ignition organic matte\*.**

a - data presented are the standard deviations of 3 replicates for splits 1 through 5 and means

 $b - * = 75\%; ** = 90\%; ** * = 95\%$  confidence levels as determined by ANOVA. Significant<br>differences are compared to the fifth pass.

### Table 3. Efficiency of various homogenization techniques.<sup>a</sup>



 $a - * =$  first sample homogenized by the given method.<br>b - Mean-1 = mean of timed experiment excluding first<br>sample homogenized by the given method.

Soil	Open Bin <b>Riffle Splitter</b>	Closed Bin <b>Riffle Splitter</b>	Cone and Quarter Technique
Jean	$-0.03$ <sup>*</sup>	nc	$+0.06$
Calico	$-0.02$	$-0.10$	$-0.56$
Gila	$+0.09$	$-0.22$	$-0.57$
Overton	$-0.36$	-0.19	$-0.42$

Table 4. Loss of fines between the seventh pass a<br>timed experiment.

 $a - + =$  clay increase;  $- =$  clay loss; nc = no change.

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#### **Influence of Sample Homogenization on Loss on Ignition Organic Matter**

**The lowest standard deviations among replicates for LOI organic matter content were found after the fifth pass, with only two exceptions, similar to the results for standard deviations in particle-size distribution (Table 2). The two exceptions were noted in the Jean gravelly loamy fine sand (after the homogenized by the cone and quarter technique), yet neither standard deviation was significantly less than the standard deviation of the fifth pass. When a confidence interval of 90% or greater was established, 56, 33, 50, 100, and 91% of the replicates from passes 9, 1, 3, 7, and T, respectively, had significantly greater variances than those found in the fifth pass. At the 75% or greater confidence level, two-thirds or more of the replicates (83, 66, 83, 100, and 91% for passes 0. 1, 37, and T, respectively) had significantly greater variances. These results support our earlier findings that the sample appears to have achieved the greatest homogeneity after the fifth pass through the homogenization process.** 

#### **Homogenization Technique Efficiency**

**The average time required to homogenize a sample using seven passes was 5.00 (range 4 to 5 minutes), 5.50 (range 4 to 7 minutes), and 30.75 minutes (range 24 to 44 minutes) for the closed-bin riffle splitter, open-bin riffle splitting, and cone and quartering, respectively (Table 3). The slightly longer time required to perform open-bin riffle splitting compared to closed-bin riffle splitting was attributed to an initial lack of experience in the use of a riffle splitter by the technician. Excluding the first time use of either riffle splitter, the average time required for open-bin riffle splitting decreased to 5.00 minutes per sample compared to 4.3 minutes per sample required for the closed-bin riffle splitter. Excluding the first use of the cone and quarter technique by the technician, the average time required to homogenize the sample decreased from 30.75 to 26.33 minutes per sample. These results indicate that the use of the riffle splitter for homogenization required markedly less time per sample than samples homogenized by the cone and quarter technique and was thus more efficient.** 

#### **Loss of Fines**

**Loss of fines was determined by comparison of clay contents between the seventh split and timed experiment. Clay contents generally decreased after the additional seven passes through the homogenization procedures but the overall clay content loss was very small (less than 0.6%) and could be attributed to expected variance in the homogenization and analytical methods (Table 4). It was interesting, however, to note that the greatest clay content decreases were found when the soils underwent cone and quartering as the homogenization process. This result was due to a twofold effect in which the inability to recover all the soil from the underlying paper led to a greater clay loss from gap filling between the larger sand particles and perhaps due to static charge development on the paper leading to a retention of the charged clay particles.** 

#### **CONCLUSIONS**

**me use of riffle splitting to homogenize a bulk soil sample was more efficient and had less of fines than cone and quartering and is therefore the recommended homogenization technique. me use of a closedbin riffle splitter was preferred to an open-bin riffle splitter due to its greater apparent ability to contain and reduce the loss of fines from dusting. Only five passes, instead of seven, should be used to obtain the most homogeneous sample, in terms of particle-size distribution and loss on ignition organic matter, due to the overwhelming evidence that the least variability among replicates occurred after the fifth pass using all three splitting techniques and all four soil textures. Random sampling after grinding and sieving was the most efficient homogenization method, since no additional sample preparation was involved, yet these samples almost consistently had greater replicate variabilities than the other homogenization techniques after the fifth pass reducing the value of this technique for soil sample homogenization.** 

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APPENDIX

DATA TABLES

**NOTE: It should be noted that the split or pass no. 0 represents'the random samples collected prior to any sample homogenization process other than the preparatory grinding and sieving. Other sample designations have been assigned such that for the first seven passes, the first digit is the pass number while the second digit is the replicate number (i.e. 1-1 = pass 1, replicate 1). The timed experiment sample numbers are. indicated by a Treplicate number (i.e. T-1 = timed experiment, replicate 1). An overall mean (MEAN ALL) was calculated for all sand, silt, and clay contents while standard deviations, and %RSDs were calculated for only the total sand, silt,**  and **clay percentages for each pass and the overall mean.** 

Sample No.	Jean loamy fine sand			Calico Ioam		Gila silt loam		Overton clay				
	OB	CB	CQ	OB	СB	CQ	OB	CВ	CQ	OB	CВ	$\infty$
0.1	7.7	7.8	7.8	7.7	7,7	77	7,8	7.7	7.8	7,8	8.0	79
0.2	7.7	7.8	7.8	7,7	7.8	7.7	7,8	7.8	7,8	7.9	8.0	7,9
$0-3$	7.7	7.8	7.8	7.7	7.8	7.7 <sup>1</sup>	7,8	7.8	7.8	7,9	8.0	7.9
$\uparrow$	7.7	7.7	7.7	7.7	7,7	7.7	7.8	7.8	7,8	7.9	8.0	7.9
$\frac{1}{2}$	7.7	7.7	7.7	7,7	7.7	7,7	7,8	7.8	7.8	7.9	8.0	7.9
$1-3$	7.7	7.7	7.7	7.7	7,7	7.7	7.8	7.8	7.8	7,9	8.0	7.9
$3-1$	7.7	7.7	7.7	7.8	7,8	7.7	7.8	7.8	7.8	79	8.0	7.9
$3-2$	7.7	7.7	7.7	7.7	7.8	7,7	7.8	7.8	7.8	7,9	8.0	7,9
$3-3$	7.7	7.7	7.7	7,7	7.8	7.7	7.8	7.8	7.8	7.9	8.0	7.9
$5 - 1$	7.7	7,7	7.7	7.7	7,7	7.7	7.8	7.8	7.8	7.9	8.0	7,9
5.2	7 <sub>1</sub>	7.7	7.7	7.8	7.7	7.7	7.8	7.8	7.8	7.9	8.0	7.9
53	7.7	7.7	7.7	7.7	7.7	7.7	7.8	7.8	7.8	7,9	8.0	7.9
$7 - 1$	7.7	7.7	7.7	7.7	7.7	7.7	7.8	7,8	7.8	7.9	0.8	7.9
7 <sub>2</sub>	7.7	7,7	7.7	7.7	7,7	7 <sub>7</sub>	7,8	7.8	7.8	7.9	0.0	7.9
7.3	7.7	7.7	7.7	7.7	7 <sub>7</sub>	7 <sub>7</sub>	7.8	7.8	7.8	7.9	0.8	7.9
7.4	7.7	7.7	7.7	77	7,7	7.7	7.8	7.8	7.8	79	0.8	7,9
7.5	7.7	7.7	77	7.7	7,7	7.7	7.8	7.8	7,8	7.9	8.0	7.9
$7-6$	7.7	7.7	7.7	7.7	77	7.7	7.8	7.8	7.8	7.9	8.0	7.9
$7 - 7$	7.7	7 <sub>7</sub>	7.7	7.7	7.8	7.7	7.8	7.8	7,8	7.9	8.0	7,9
$\mathsf{T}$	7.7	7.7	7.7	7,7	7.8	7.7	7.8	7.8	7.8	7,9	0.0	7.9
7.2	7.7	7.7	7.7	7.7	7,8	7,7	7.8	7.8	7.8	7,9	8.0	7.9
$7.3\,$	7.7	7.7	7.7	7.7	7.8	7.7	7.8	7.8	7.8	7.9	8.0	7.9
$T-4$	7.7	7,7	7.7	7.7	7.8	7.7	7,8	7.8	7.8	7.9	8.0	7.9
7.5	7.7	7.7	7.7	7 <sub>7</sub>	7.8	7.7	7.8	7.8	7,8	7.9	8.0	7,9
T 6	7.7	7.7	7.7	7,7	7.8	7.7	7.8	7.8	7.8	7,9	8.0	79
$7-7$	7.7	7.7	7.7	7.7	7.8	77	7.8	7.8	7.8	7.9	8,0	7.9

Table A-1. The 0.01 M CaCl, pH values for individual samples in the homogenization study\*

 $a \cdot 0B$  = open-bin riffle splitter; CB = closed-bin riffle splitter; CQ = cone and quarter technique.

**Table A-2. Particle-size data for the Jean loamv fine sand homoaenized bv own-bin riffle splitting.** 

SOIL TYPE - SAND<br>SPLITTING METHOD - OPEN BIN RIFFLE SPLITTER



**Table A-3. Particle-size data for the Jean loamv fine sand homogenized by closed-bin riffle splitting.** 

SOIL TYPE - SAND<br>SPLITTING METHOD - CLOSED BIN RIFFLE SPLITTER



## **Table A-4. Particle-size data for the Jean loamy fine sand homogenized by cone and quartering,**





SOIL TYPE - LOAM SPLITTING METHOD - OPEN BIN RIFFLE SPLITTER







### **Table A-7. Particle-size data for the Calico loam homogenized by cone and quartering,**



SOIL TYPE - LOAM<br>SPLITTING METHOD - CONE AND QUARTER

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Table A-8. Particle-size data for the Gila silt loam soil homogenized by open-bin riffle splitting,

SOIL TYPE - SILT LOAM SPLITTING METHOD - OPEN BIN RIFFLE SPLITTER



### **Table A-9. Particle-size data for the Gila silt loam homogenized by closed-bin riffle splitting.**

SOIL TYPE - SILT LOAM SPLITTING METHOD - CLOSED BIN RIFFLE SPLITTER



**Table A-10. Particle-size data for the Gila silt loam soil homogenized by cone and quartering.** 



SOIL TYPE - SILT LOAM<br>SPLITTING METHOD - CONE AND QUARTER

**Table A-11. Particle-size data for the Overton clay homogenized by open-bin riffle splitting,** 



SOIL TYPE  $-$  CLAY SPLITTING METHOD - OPEN BIN RIFFLE SPLITTER **Table A-12. Particle-size data for the Overton clay homogenized by closed-bin riffle splitting.** 

SOIL TYPE - CLAY SPLITTING METHOD - CLOSED BIN RIFFLE SPLITTER



**Table A-13. Particle-size data for the Overton clay soil homogenized by cone and quartering** 

SOIL TYPE - CLAY SPLITTING METHOD - CONE AND QUARTER



Table A-14. Air-dried moisture and LOI organic matter contents for the Jean loamy fine sand homogenized<br>by open-bin riffle splitting.

SOIL TYPE - SAND<br>SPLITTING METHOD - OPEN BIN RIFFLE SPLITTER



**Table A-15. Air-dried moisture and LOI organic matter contents for the Jean loamy fine sand homogenized by closed-bin riffle splitting.** 

SOIL TYPE - SAND SPLITTING METHOD - CLOSED BIN RIFFLE SPLITTER SAMPLE AIR-DRY OM CONTENT **NO**  $($ ----------  $Wt$   $(-$ -------- $0 - 1$  $0.00$  $0.77$  $0 - 2$  $0.13$  $0.78$  $0 - 3$  $0.00$  $0.65$  $1 - 1$  $0.00$  $0.76$  $1 - 2$  $0.00$  $0.70$  $1 - 3$  $0.00$  $0.70$  $3 - 1$  $0.00$  $0.76$  $3 - 2$  $0.00$  $0.89$  $3 - 3$  $0.16$  $0.79$  $0.00$  $0.83$  $5 - 1$  $5 - 2$  $0.19$  $0.77$  $5 - 3$  $0.00$ 0.76  $7 - 1$  $0.00$ 0.85  $7 - 2$  $0.26$ 1.03  $7 - 3$  $0.00$  $0.73$  $7 - 4$  $0.19$  $0.94$  $7 - 5$  $0.00$  $0.61$  $7 - 6$  $0.28$ 0.85  $7 - 7$  $0.00$ 0.79  $0.00$ 0.55  $T-B-S-1$  $0.00$  $0.96$  $T-B-S-2$  $0.00$  $0.90$  $T-B-S-3$  $T-B-S-4$  $0.00$ 0.89  $T-B-S-5$  $0.00$  $0.69$  $T-B-S-6$  $0.00$  $0.60$  $T-B-S-7$  $0.00$ 0.68 MEAN 0  $0.04$  $0.74$ MEAN 1  $0.00$  $0.72$ MEAN<sub>3</sub>  $0.05$  $0.81$ MEAN 5  $0.06$  $0.79$ MEAN 7  $0.10$  $0.83$ MEAN TIM  $0.75$  $0.00$ MEAN ALL  $0.05$  $0.78$  $S.D.$   $O$ 0.075  $0.073$ **&RSD 0** 173.205 9.950  $S.D. 1$  $0.000$  $0.031$ %RSD 1  $0.000$  $0.000$  $S.D. 3$  $0.091$ 0.068  $kRSD$  3 173.205 8.434  $S.D. 5$ 0.038  $0.111$ **&RSD 5** 173.205 4.787  $S.D. 7$  $0.133$  $0.137$  $kRSD$  7 127.680 16.580  $0.000$  $S.D. T$  $0.000$  $0.162$ **%RSD T**  $0.000$ S.D. ALL 0.089  $0.114$ **&RSD ALL** 190.690 14.616 **Table A-16. Air-dried moisture and LOI organic matter contents for the Jean loamy fine sand homogenized by cone and quartering.** 



**Table A-17. Air-dried moisture and LOI organic matter contents for the Calico loam homogenized by open-bin riffle splitting.** 

SOIL TYPE - LOAM SPLITTING METHOD - OPEN BIN RIFFLE SPLITTER SAMPLE AIR-DRY OM CONTENT NO  $0 - 1$  $0.72$  $3.10$  $0 - 2$  $0.71$  $3.13$  $0.83$  $0 - 3$  $3.29$  $1 - 1$  $0.87$ 3.28 3.24  $1 - 2$  $0.87$  $1 - 3$  $0.79$ 3.22  $3 - 1$ 0.72  $3.18$  $3.26$  $3 - 2$  $0.68$  $3 - 3$  $0.78$ 3.31  $5 - 1$  $0.69$ 3.23  $5 - 2$ 0.73  $3.22$  $0.78$ <br> $0.72$  $5 - 3$ 3.27  $7 - 1$  $3.23$  $7 - 2$  $0.74$  $3.20$  $7 - 3$  $0.77$ 3.26  $7 - 4$  $0.74$ 3.22  $7 - 5$  $0.80$  $3.22$  $7 - 6$  $0.82$ 3.18  $7 - 7$  $0.66$  $3.13$  $T-R-L-1$  $3.20$  $0.62$  $T-R-L-2$  $0.67$  $3.23$  $T-R-L-3$  $0.61$ 3.05  $T-R-L-4$  $0.62$  $3.10$  $T-R-L-5$  $0.64$  $3.00$  $T-R-L-6$  $0.78$ 3.15  $T-R-L-7$  $0.60$ 2.97  $0.75$  $3.17$ MEAN 0 MEAN 1  $0.84$  $3.25$ MEAN 3  $0.73$  $3.25$ MEAN 5  $0.73$  $3.24$ MEAN 7  $0.75$  $3.21$ MEAN TIM  $0.65$ 3.10 MEAN ALL  $0.73$  $3.19$  $S.D. 0$ 0.069  $0.103$ **&RSD 0** 9.139 3.260  $S.D. 1$ 0.050 0.032 %RSD 1 5.924 0.988  $S.D. 3$ 0.050 0.062 **&RSD 3** 6.887 1.919  $S.D. 5$  $0.041$  $0.024$ %RSD 5 5.648 0.750 1.256  $S.D. 7$ 0.052  $0.040$ %RSD 7 6.895  $S.D. T$ 0.063  $0.101$ **&RSD** T 9.766 3.256 S.D. ALL 0.076  $0.085$ **&RSD ALL** 10.438 2.681



**Table A-18. Air-dried moisture and LOI organic matter contents for the Calico loam homogenized by closed-bin riffle splitting.** 

**Table A-19. Air-dried moisture and** LOI **organic matter contents for the Calico loam homogenized by**  cone and **quartering.** 



**Table A-20. Air-dried moisture and LOI organic matter contents for the Gila silt loam homogenized by open-bin riffle splitting.** 

SOIL TYPE - SILT LOAM<br>SPLITTING METHOD - OPEN BIN RIFFLE SPLITTER



Table A-21. Air-dried moisture and LOI organic matter contents for the Gila silt loam homogenized by<br>closed-bin riffle splitting.



Table A-22. Air-dried moisture and LOI organic matter contents for the Gila silt loam homogenized by<br>cone and quartering.

SOIL TYPE - SILT LOAM<br>SPLITTING METHOD - CONE AND QUARTER



Table A-23. Air-dried moisture and LOI organic matter contents for the Overton clay homogenized by<br>open-bin riffle splitting.



SOIL TYPE - CLAY SPLITTING METHOD - CLOSED BIN RIFFLE SPLITTER



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Table A-25. Air-dried moisture and LOI organic matter contents for the Overton clay homogenized by cone and quartering.

