# Stormwater Particulates Sampling and Processing Concerns

Robert Pitt, Ph.D., P.E., BCEE, D.WRE
Emeritus Cudworth Professor of Urban Water Systems
Department of Civil, Construction, and Environmental Engineering
University of Alabama, Tuscaloosa, AL, USA

Observed/Perceived Problems with Stormwater Particulate Sampling and Analyses

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## Stirred and Settled Sample, Showing Settleable Solids (Madison high-efficiency street cleaning tests)





Bedload in corrugated stormdrain and mound of settleable material at discharge into wet detention pond after many years of operation at ski resort at Snowmass, CO (drain from several acre resort parking area having sand applications for traction control).



Results of Verification Monitoring of a Popular Hydrodynamic Device by WI DNR and USGS (Madison, WI)			
Sampled solids load in	1623 kg		
Sampled solids load out	1218 kg		
Trapped by difference	405 kg (25% removal)		
Actual trapped total sediment	536 kg (33% actual removal)		
Fraction total solids not captured by automatic samplers	8% (131 kg missed by sampler, out of 1623 kg in sampler)		

Standard automatic water samplers with single intakes at bottom of pipes. Influent samplers are affected by large particles while effluent samplers should not be, assuming most any stormwater control is capable of removing the larger particles that stress the samplers.

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# TSS (Total Suspended Solids) vs. SSC (Suspended Sediment Concentration) and PSD (Particle Size Distribution) Relationships

#### Two separate issues:

- sampling to obtain representative water samples with all particulates of interest, and
- laboratory processing to represent all particulates.

Most problems result in loss of large particles. The combination of methods used affects modeling approach, especially particle size distributions and confusion between TSS and SSC.

drainage compromised about 4% of Madison area total solids discharges (WI DNR and USGS) monitoring).

Bed load in storm





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## Sampling Effects on Particulate Solids Characteristics

- Sampling issues associated with stratified flows and bedload.
  - Sampler intakes on bottom of pipe may collect more bedload than represented in well-mixed sample, and
  - sampler tube velocity may not be able to transport large particles to sample bottles

These are two opposite problems that seldom cancel each other out nicely.

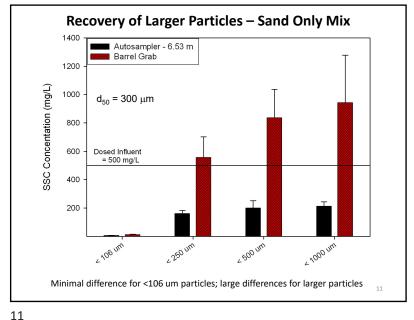
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### **Questions for Collection and Analysis of Solids**

Sample Collection → Preparation → Analysis

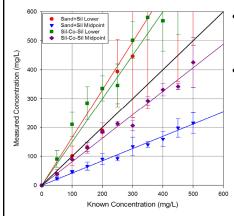
- Collection method?
- · Location in the flow stream?
- · Effectiveness of autosamplers? Where in the flow stream?
- Sample processing methods?
- Sample analytical methods?
- Particle size distribution effects?
- Impact of variability on final solids analysis?



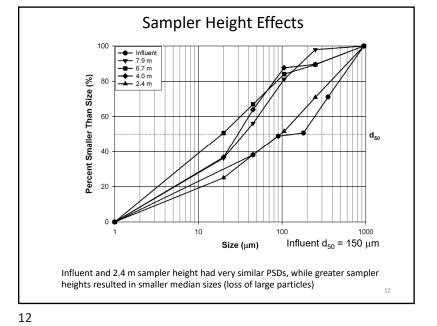


### **Sample Location Effects on PSD**

(Standard Methods 2540D)



- Pipet location in sample bottle critical in TSS results.
- Low sampling locations had greater concentrations and mid-point sampling locations had smaller concentrations than known standard concentration.



## Loss of Large Particulates in Sampling Lines (100 cm/sec sample line velocity)

Percentage loss of particulates	Critical settling rate (cm/sec)	Size range, μm (1.5 to 2.5 sp. gr.)
100	100	8,000 – 25,000
50	50	3,000 – 10,000
25	25	1,500 – 3,000
10	10	350 – 900
1	1	100 – 200

Problem isn't sample line velocity (few particles >100 um, resulting in expected errors of <10%), but location of intake is; therefore need bedload sampler

**Sample Splitting Methods** 

- Always need to obtain subsamples for different laboratory analyses
- As an example, most particulate analyses require 100mL of sample, while the total sample volume is likely 1+L
- Typical subsampling methods to split the sample volume include:
  - Shake and pour into graduated cylinder
  - Pipette while on a stir plate
  - Funnel (cone) splitter

Simple methods to obtain representative sample: create cascading and well-mixed flow at sampling location (well-mixed flow with bedload and no stratification). Examples shown for gutter and pipe flow installations.







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# Sample Splitting for Volume and Sediment Accuracy

- USGS studies found that "shaking and pouring" (or worse, pipetting) 100 mL subsamples from sample bottles for TSS analyses frequently leads to unacceptable errors.
- The USGS found that if the sand fraction (>63 micrometers) comprised less than 25% of the total sample mass, then preferred cone or churn splitting methods were in reasonable agreement with pouring or pipetting methods.
- Since we are concerned with the complete range of particle sizes, and that some source area samples, or some seasonal outfall runoff samples, may exceed this amount of sandsized particles, stormwater sample splitting needs to be done with churn, or preferably, cone splitters.
- As part of a sediment transport in swales project and a large residential/commercial monitoring project, we evaluated different sampling splitting methods for a wide range of stormwater sediment conditions.

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# Comparison of Three TSS (total suspended solids) and SSC (suspended sediment concentration) Analytical Methods

vortex

EPA TSS 160.2 Shake sample bottle vigorously then pour aliquot into graduated cylinder Standard Methods ASTM SSC D3977TSS 2540D 97B
Use stir plate and pipet at middepth in bottle and midway
between wall and

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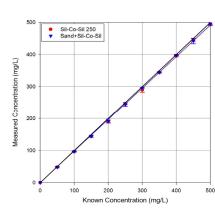
TSS (shake and pour vs. stir and pipette) shake and pour vs. stir and pipette Significant difference: 22% bias, shake and t-Test: Paired Two Sample for Means Stir and Shake pour low Pipette 1200 Mean 133 160 y = 1.22x Variance 19818 31015 1000 Observations 59 800 Pearson Correlation 0.99 Hypothesized Mean Difference 0 58 t Stat -4.99 P(T<=t) one-tail Results of parallel tests using 59 stormwater samples from Kansas City stormwater research stir and pipette TSS vs. SSC no significant difference (for the number stir and pipette TSS vs. SSC t-Test: Paired Two Sample for Means of samples evaluated) Stir and SSC cone Pipette splitter y = 0.93x 160 158 800 Mean 700 Variance 31015 26095 Observations 59 500 Pearson Correlation 0.95 400 Hypothesized Mean Difference Ω 58 0.27 1200 P(T<=t) one-tail 0.40

USGS/Dekaport cone splitter used to separate sample into smaller volumes for different analyses.



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### Suspended Sediment Concentration Compared to Known Laboratory Additions



 SSC methodology (cone splitter) represents the entire sample – regardless of sample particle size distribution.

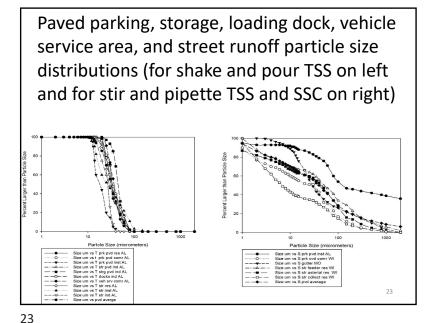
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Stormwater Particle Size
Distributions (PSD) from Different
Source Areas Compared by TSS or
SSC Sample Splitting Methods

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Roof runoff particle size distributions (for TSS shake and pour on left and for TSS stir and pipette and SSC on right)

Particle Size (micrometers)

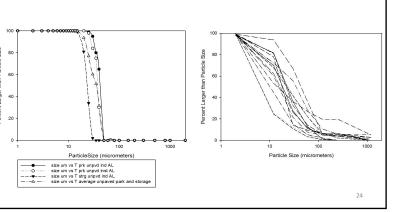
Particle Size (micrometers)

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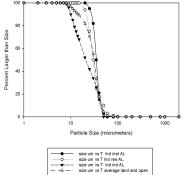
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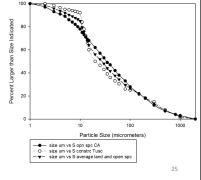
Non-paved parking and storage area runoff particle size distributions (for shake and pour TSS on left; SSC on right)



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Landscaped, open space, and construction site runoff particle size distributions (for shake and pour TSS on left and for stir and pipette TSS and SSC on right)





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Wet Sieving for Particle Size Distributions and Pollutant Associations by Particle Size

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#### **Particulate Sampling and Analyses Conclusions**

- The largest particles found in sheetflows from source areas are preferentially deposited along the flow paths and drainage system.
- Shake and pour TSS methods do not measure these large particles, while stir and pipette TSS and SSC methods do include these particles.
- Most outfall particle size distributions lack these large particles, and different TSS or SSC methods do not result in significant PSD differences at the outfalls. Better sampling methods reduce the variability of the results.
- "Short" drainage systems that do not retain the large particulates do result in different particle size distributions if different methods are used.
- Appropriate PSDs must be matched with the correct TSS or SSC values with modeling stormwater particulates.

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Large sample volume (about 5 L) separated into subsamples using cone splitter. The sample is first poured through a 1,200 μm fiberglass window screen to remove leaves and grass clippings, and coarse sediment that would clog the splitter. This captured material is also analyzed. Each subsample is about 1 L in volume. These can then be split again using the cone splitter for ten 100 mL subsamples. Each of these 100 mL subsamples can be filtered to obtain filtrate only having particles smaller than the sieve or filter.





Tab 2-B - WinSLAMM Theory and Practice 11/21/2023



All-plastic vacuum filtering setups are used with a series of polycarbonate membrane filters (10, 5, 2, 1, 0.45µm). The filtrates are then chemically analyzed.

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**Wet Sieving Procedure Outline** 

- About 12 sieves and membrane filters are used for the wet sieving for particle size analyses (and for particulate strength analyses for pollutants).
- Determine the volume needed for the chemical analyses for each size fraction. Particulates (for PSD analyses) require about 100 mL, but will likely be greater due to low concentrations.
- Use cone splitter to separate the original sample into needed subsample volumes for the number of sieves and filters to be used (plus additional subsamples for replicate analyses for QA/QC).
- Pour one subsample through one of the sieves or filters (not in a stack, as there will be very little sediment captured on each sieve).
- Place the filtrate in a pre-weighed evaporating dish and evaporate to dryness, and re-weigh.



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### PSD of the test sediments 100 90 80 Cumulative mass (%) 70 60 50 40 30 20 10 10 100 1000 Particle diameter (micro meter)-log scale 33

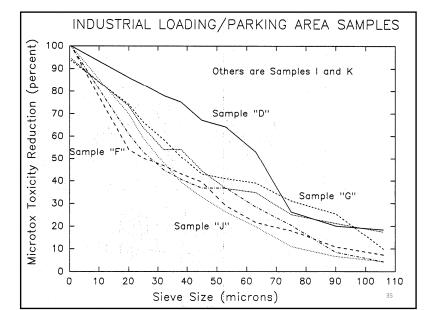
### **Particle Size Pollutant Associations**

- Concentrations of particles associated with different particle sizes are used to better design stormwater controls and to identify pollutant sources.
- Samples are first split using the cone splitter, and the individual subsamples are further individually separated using a variety filters and sieves.
- The filtered samples are then individually analyzed, and the concentrations are determined by difference. Sediment samples can also be examined by saving the filters, or by removing some of the captured debris from the sieves.

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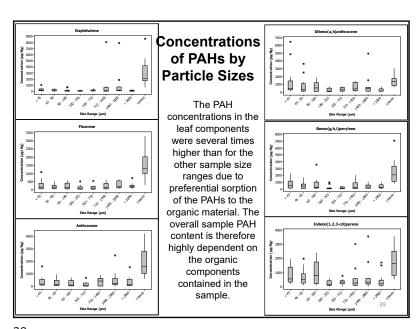


### Residential and commercial area example: Average percent reductions in pollutant discharges after controlling down to indicated particle size:

	250 µm	45 µm	10 µm	2 µm	0.45 µm
Suspended Solids	22	71	95	94	100
Turbidity	23	41	72	77	86
COD	0	23	36	37	40
Total Phosphorus	12	32	48	51	52
Zinc	2	15	23	30	31
Copper	4	14	34	30	36
Cadmium	0	8	0.1	0.1	7
COD Total Phosphorus Zinc Copper	0 12 2 4	23 32 15 14	36 48 23 34	37 51 30 30	40 52 31

For these samples, the control of filterable pollutants (using chemical precipitation, ion exchange or sorption, for example) is also necessary for high levels of control. Control down to about 35  $\mu m$  (removal of all particulates larger than this size) can result in about 80% TSS reductions (a common goal), but that would only result in about <25 to 50% control of total forms of other stormwater pollutants (probably lower than desired) for this example.

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**Pollutant Strengths of Stormwater Particulates** 

- Pollutant strengths are the contaminant concentrations associated with the particulate matter in the stormwater.
- Particulate strengths are determined by calculating the pollutant concentration only associated with the particulates (measured as TSS or SSC, depending on how the sample was collected and analyzed) in the runoff water.
- They are calculated by the following equation, and are usually expressed as mg pollutant/kg solids:

(total conc. – filterable conc.)
particulate solids conc.

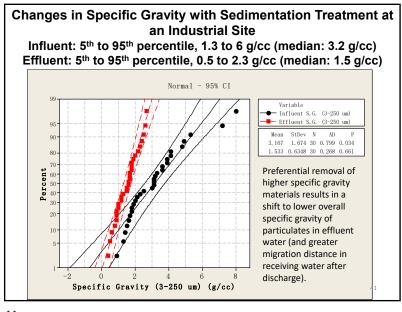
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### Specific Gravity and Volatile Solids of Sediment Collected from Stormwater Treatment Device

Sieve size range (um)	Average Specific Gravity (g/cc)	Average Volatile Solids (%)
Large organic material (leaves, etc.)	0.84	81.2
>2800	0.66	70.9
1400 - 2800	1.15	57.8
710-1400	1.43	42.7
355-710	2.56	26.1
180-355	2.76	19.4
75-180	2.97	20.6
45-75	3.30	25.7
<45 (Pan)	3.46	26.0

Specific gravity decreases as the volatile solids content increases; larger particle sizes have lower specific gravity and greater volatile solids as they contain larger amounts of light-weight organic debris for these industrial area stormwater sediment samples. Their settling rates are still large due to their large sizes.



### **Particulate Strength Conclusions**

- Knowing the distribution of pollutants associated with different sized stormwater particles allows more accurate determinations of their sources, transport, and control.
- Urban stormwater quality models can use this information when routing stormwater particulatebound pollutants from their source areas and then through the drainage system and stormwater controls.
- The discharged particle size distributions and associated pollutants can then be used in receiving water models to calculate their fates and effects.

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