

# **DISTRIBUTION OF CHLORIDE, PH, RESISTIVITY, AND SULFATE LEVELS IN BACKFILL FOR MSE WALLS AND IMPLICATIONS FOR CORROSION TESTING**

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**Progress  
Report  
BDV 25 TWO  
977-03**

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Gainesville, FL**

# PROJECT GOALS

Assure that:

- For chloride, pH, resistivity, and sulfate levels measured in corrosion testing, variability in these levels due to sampling and analytical techniques is much lower than variability within a select backfill stockpile or stratum.
- Corrosion properties of backfill material do not change appreciably over time, especially after emplacement and over the design lifetime of the MSE wall.
- The number of soil samples analyzed prior to acceptance of backfill is appropriate.

# PROJECT TASKS

**Task 1 Literature Review**

**Task 2 Databased Trends in MSE Wall Backfill Properties**

**Task 3 Single-Laboratory Contributions to Method Reproducibility & Proposed Method Improvements**

**Task 4 Heterogeneity of MSE Wall Backfill**

**Task 5 Rainfall-Driven Temporal Changes in Backfill Properties**

**Task 6 Multi-Laboratory Contributions to Method Reproducibility**

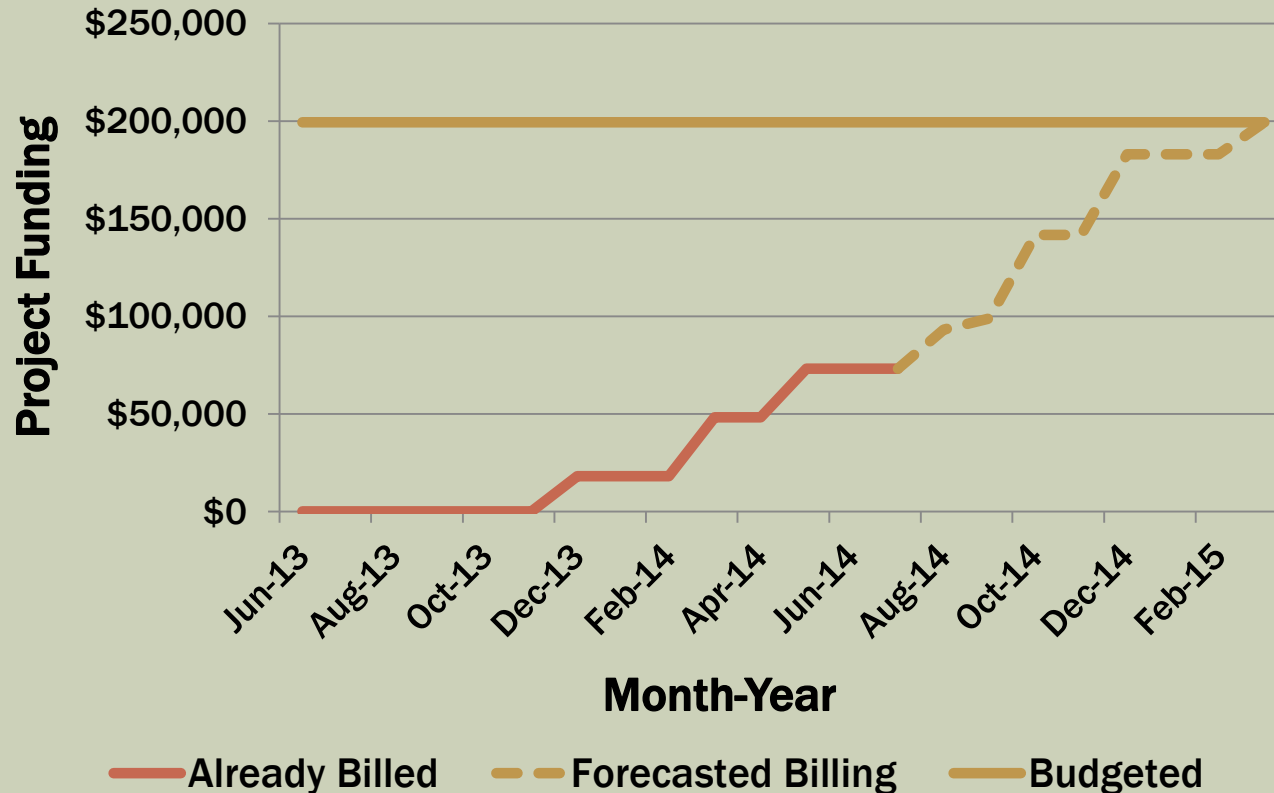
**Task 7 Final Report**

# PROJECT PROGRESS BY TASK

Task	10%	20%	30%	40%	50%	60%	70%	80%	90%	100%
1-A	█	█	█	█	█	█	█	█	█	█
1-B										
2	█	█	█	█	█	█	█	█	█	█
3-A, 3-B	█	█	█	█	█	█	█	█	█	█
3-C, 3-D	█	█	█	█	█	█	█	█	█	█
3-E, 3-F	█	█	█	█	█	█	█	█	█	
3-G, 3-H	█	█	█	█	█					
3-I, 3-J										
4	█	█	█	█	█					
5	█									
6-A	█	█	█	█	█	█	█	█	█	█
6-B										
7										

**Overall Task Progress is 55%**

# PROJECT PROGRESS BY BILLING



**Overall Billed Progress is 37%**

# RESEARCH HIGHLIGHTS FOR FM5-550 pH IN SOIL AND WATER



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# TASK 2 TRENDS

Variable	District	Valid N	Mean	Median	Mode	Freq of Mode	Min	Max	10 <sup>th</sup> Percentile	25 <sup>th</sup> Percentile	75 <sup>th</sup> Percentile	90 <sup>th</sup> Percentile	St Dev
pH	7	412	6.8	6.8	7.3	14	4.0	9.9	5.2	5.9	7.8	8.2	1.1
	1	124	7.4	7.5	7.2	8	4.9	8.8	6.1	7.0	8.2	8.4	0.9

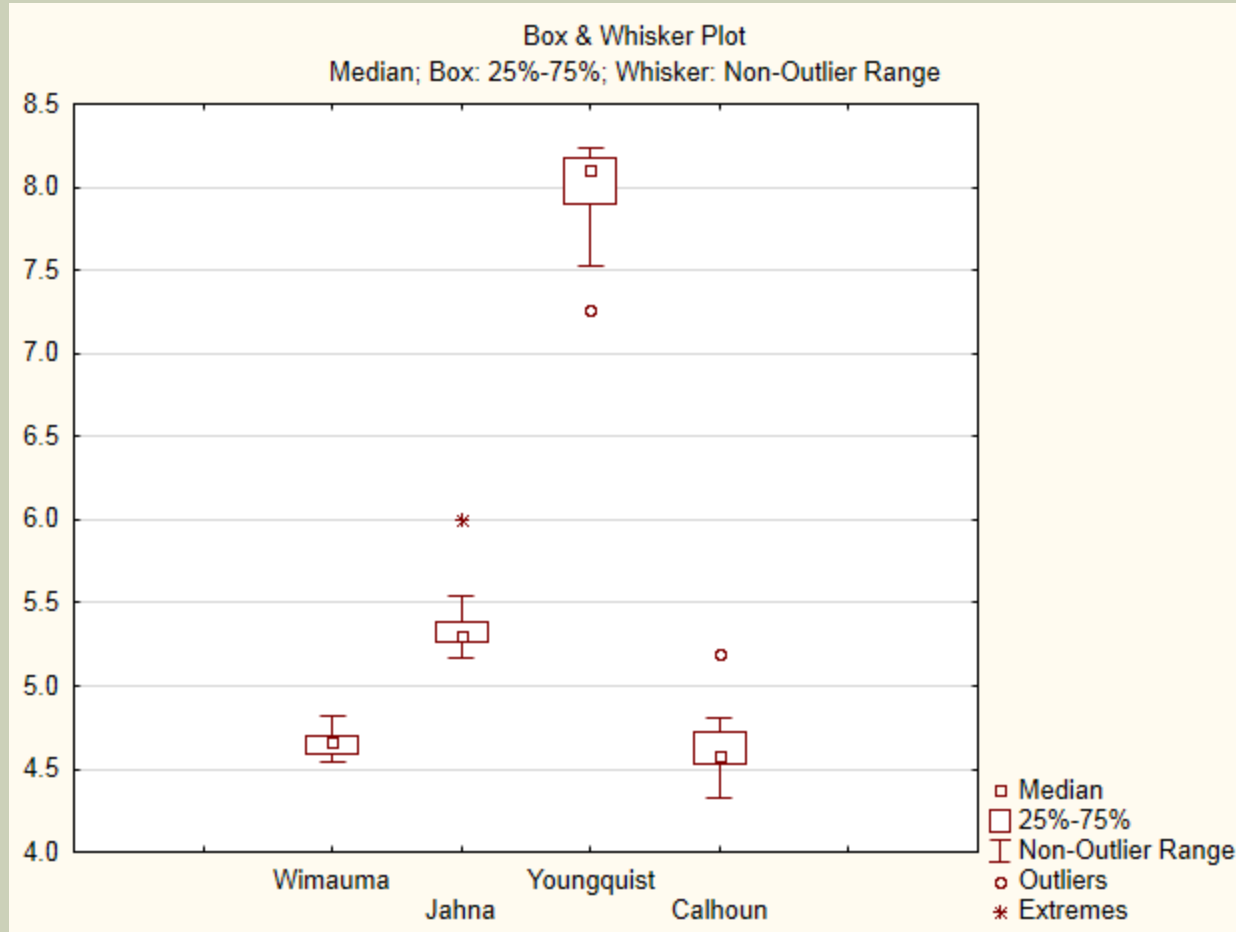
For pH, 3% of the samples were below pH 5 or above pH 9; the failure rate was 1% for District 1 and 4% for District 7.

# TASK 3 SINGLE-LABORATORY PERFORMANCE

Sample	N	Average	St Dev	%RSD	%RE
pH 5 Buffer	5	5.00	0.01	0.23	0.32
pH 9 Buffer	5	9.02	0.00	0.00	-0.22
Santa Fe River Sand	9	7.97	0.06	0.73	2.13
Starvation Hill Sand	13	7.82	0.07	0.90	3.10



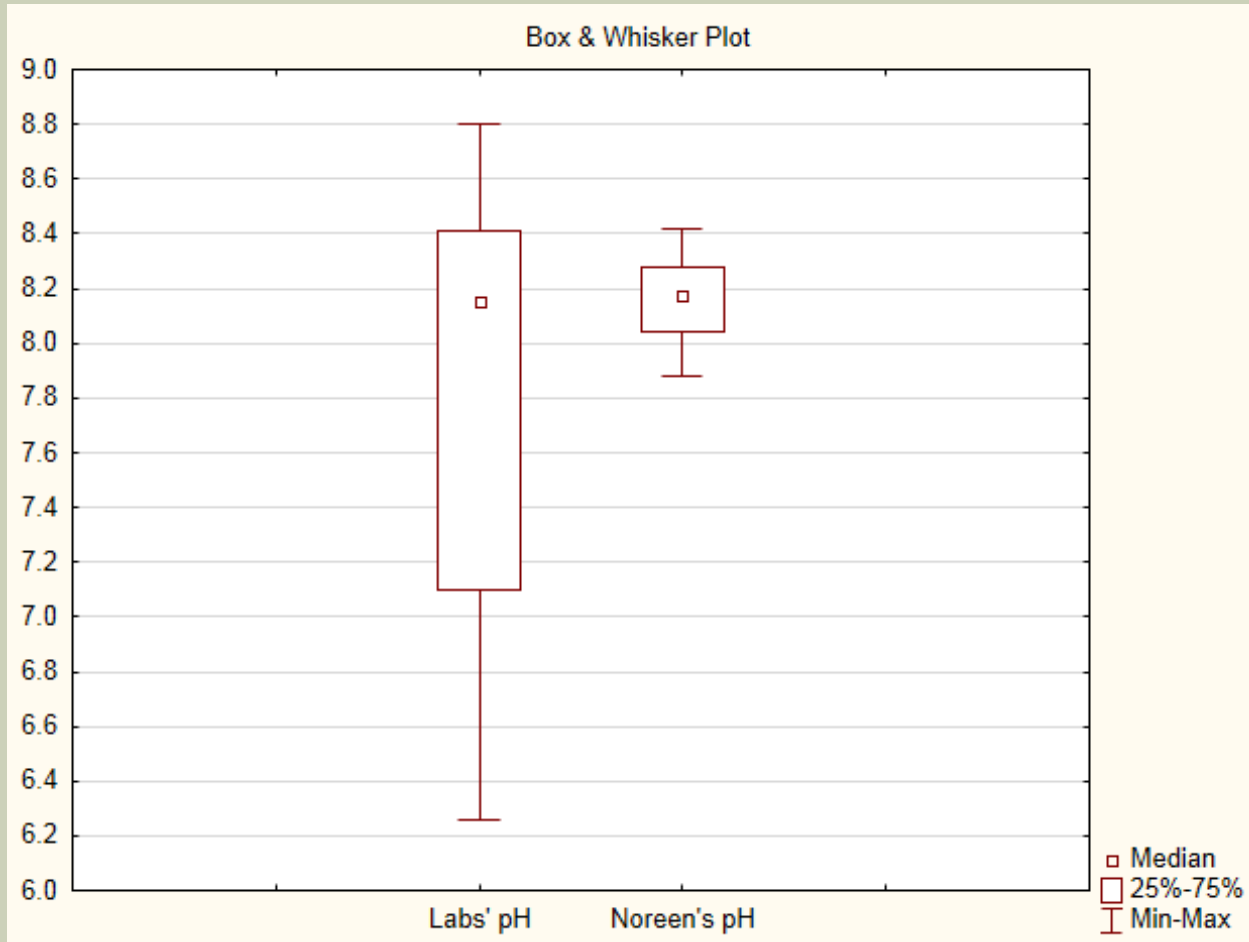
# TASK 4 HETEROGENEITY OF BACKFILL



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pH 9 Buffer	5	9.02	0.00	0.00	-0.22
Santa Fe River Sand	9	7.97	0.06	0.73	2.13
Starvation Hill Sand	13	7.82	0.07	0.90	3.10
Calhoun Sand	12	4.64	0.21	4.61	18.5
Jahna Sand	12	5.37	0.22	4.11	15.5
Wimauma Sand	12	4.66	0.09	1.87	6.0
Youngquist Sand	12	7.98	0.30	3.81	12.3

# TASK 6 MULTI-LABORATORY PERFORMANCE



# TASK 6 MULTI-LABORATORY PERFORMANCE

## On The Road With Santa Fe River Sand

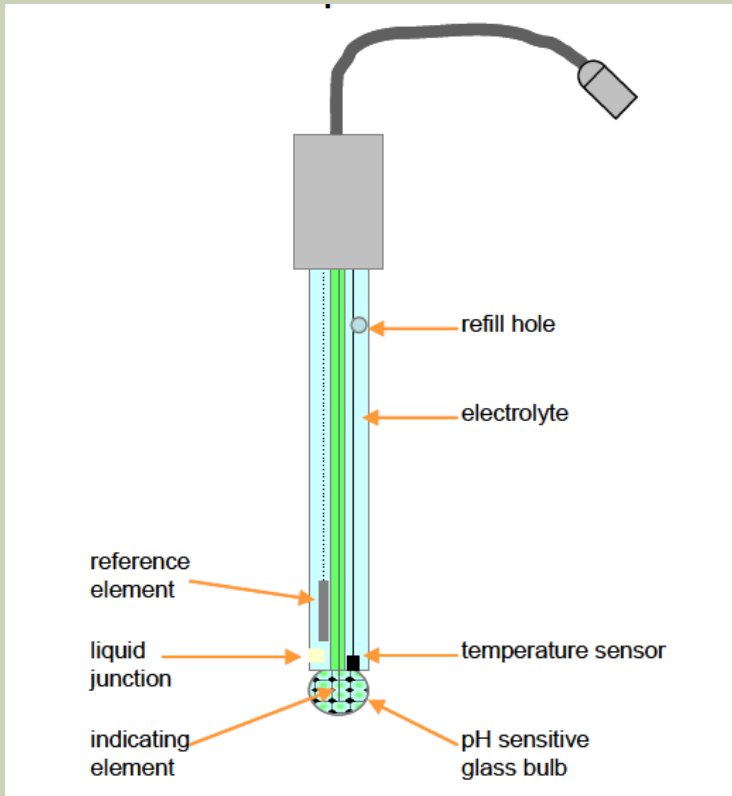
pH Measured by	N	Average	St Dev	%RSD	%RE
USF Laboratory	9	7.97	0.06	0.73	2.13
USF On-Site Audit	14	8.15	0.15	1.83	6.62
Inter-Laboratory	15	7.85	0.80	10.1	32.4

*Note: Results for 6 FDOT and 9 commercial laboratories; these results do not include variability from field sampling, transport, and storage of soil.*

# TASK 6 MULTI-LABORATORY PERFORMANCE

Transported soil at ambient vs cool temperatures  
Stored under ambient conditions or in the refrigerator  
Used 100 g or 100 ml of soil  
Tested “as is” or air dried  
Used soil from resistivity measurement  
Waited zero to 30 min or more to test sample  
Stirred once or up to three times  
Put soil/water mixture on shaker table for three 10-min intervals  
Stirred or did not stir sample during measurement  
Took reading when stable light came on or waited until reading was stable for 1 min  
Prepared soil water/mixture in a beaker, bottle, or disposable cup  
Calibrated with one, two, or three buffers  
Used fresh buffers or re-used buffers for calibration  
Stored pH electrode in distilled water, tap water, buffer, KCl, or dry  
Used glass electrode or solid state electrode  
Refilled glass electrode or used disposable electrode  
Kept electrode for up to 10 yrs

# TASK 1 LITERATURE REVIEW



Basic parts of a 3-in-1 combination electrode.

## MSE Wall Backfill

### Problem:

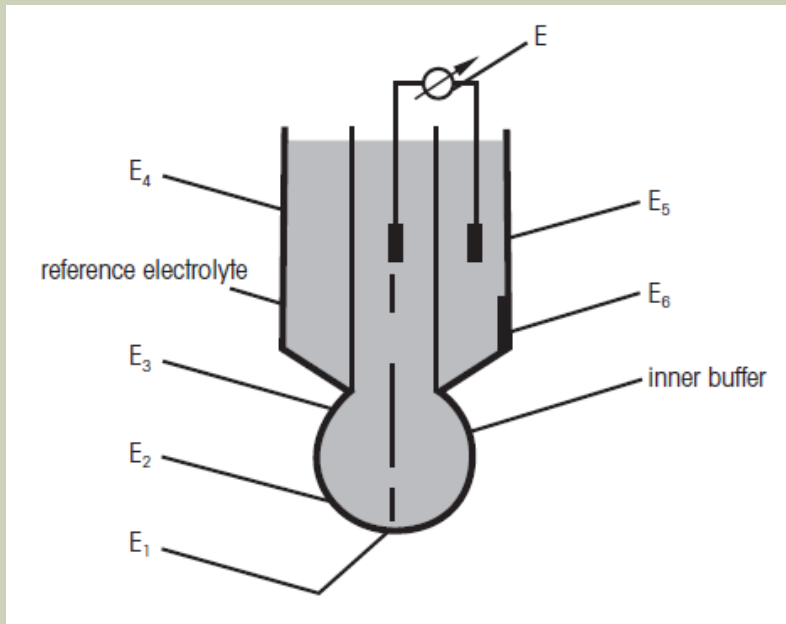
pH measurement is slow, drifting, noisy, non-reproducible, or inaccurate.

### Causes:

Electrolyte is contaminated, liquid junction is clogged, glass bulb is damaged, reference element is depleted, wire is broken, or buffers are contaminated.

# TASK 1 LITERATURE REVIEW

Sources of electrical potential in a combination electrode; ideally, E2 through E6 are held constant and E1 varies with the hydronium ion concentration in the solution.



## MSE Wall Backfill

### Problem:

pH measurement is slow, drifting, noisy, non-reproducible, and inaccurate.

### Causes:

Low electrical conductivity of sample, differences between low ionic strength solutions and normal ionic strength buffers, change in the liquid junction potential, and absorption of carbon dioxide.

# TASK 3 PROPOSED IMPROVEMENTS

- Standardize equipment and electrode capabilities
- Develop and include QA/QC for pH electrode
  - Reach a stable soil reading within ~1 min
  - Read buffer pH  $\pm 0.05$  pH units of buffer concentration
  - Calibrate to a % slope within 90% to 102%
  - Check that offset is within  $\pm 25$  mV
- Provide more detail in procedures
  - Calibrate with three fresh buffers
  - Take steps check electrode performance
  - Provide steps for cleaning, storing, and filling pH electrode
- Make pH method more robust
  - Air dry and sieve soil?
  - Use an ionic strength adjuster—add 0.1 g KCl to samples?
  - Test more samples?



# TASK 3 PROPOSED IMPROVEMENTS



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# QUESTIONS OR COMMENTS?



**Jahna Sand Mine, Haines City, Florida**

# TASK 3 SINGLE-LABORATORY PERFORMANCE

Ruggedness Study		pH Determination							
Original	Change	1	2	3	4	5	6	7	8
100 ml (ML) soil	100 g (G) soil	ML	ML	ML	ML	G	G	G	G
room temp (RT)	cold (C)	RT	RT	C	C	RT	RT	C	C
30 min wait	no wait	30	0	30	0	30	0	30	0
distilled (DSTL)	deionized (DI)	DSTL	DSTL	DI	DI	DI	DI	DSTL	DSTL
pH probe 1	pH probe 2	1	2	1	2	2	1	2	1
stir gently (G)	no stir (NS)	G	NS	NS	G	G	NS	NS	G
wetted soil	dry soil	WET	DRY	DRY	WET	DRY	WET	WET	DRY
<b>pH Results: 8.09 ± 0.18</b>		<b>7.99</b>	<b>8.01</b>	<b>7.90</b>	<b>8.18</b>	<b>7.94</b>	<b>8.40</b>	<b>8.00</b>	<b>8.31</b>

# TASK 3 SINGLE-LABORATORY PERFORMANCE

Original	Change	Difference	pH is higher if
30 min wait	no wait	<b>-0.267</b>	measured without delay
100 ml soil	100 g soil	<b>-0.143</b>	water to soil ratio is higher
distilled (DSTL) water	deionized (DI) water	<b>-0.027</b>	diluted with deionized water
room temp (RT) sample	cold sample (C)	<b>-0.012</b>	sample is colder
stir gently (G) with probe	no stir (NS) with probe	<b>0.027</b>	sample is gently stirred
soil is wetted overnight	soil is dry	<b>0.102</b>	soil is wetted overnight with 10% water
pH probe 1	pH probe 2	<b>0.118</b>	measured with probe 1