A) Methodology for analysis of mass concentration for unconsolidated, well-mixed slurries (analogous to those being acoustically tested in Figure 2 of Luna DOE proposal).

1. At Luna approximately a 10 mL aliquot of a given well-mixed slurry should be pipetted from the approximate center of the acoustic sampling chamber (as measured horizontally as well as vertically).

2. The volume sampled by each pipette must be recorded by Luna as accurately as possible. VIMS will provide the appropriate pipettes.

3. Each pipetted sample should be released into a pre-weighed and pre-labeled disposable vial and capped for later analysis at VIMS. VIMS will provide the pre-labeled/pre-weighed vials with caps.

4. Each slurry concentration of interest should be sampled as described above in triplicate to aid in our verifying the quality control of the procedure.

5. The pipettes are meant to be reused for multiple slurry samples, so it is essential that they be rinsed well between each use.

6. After transport of the sample vials to VIMS, the vials will be reweighed at VIMS before opening.

7. Each vial sample will then be rinsed into a pre-washed and pre-weighed aluminum dish with de-ionized water, making sure that all the particles are transferred.

8. The vials and dishes will then be placed in a 65 degree Celsius drying oven overnight.

9. The vials and dishes will be cooled in a desiccator and then re-weighed.
10. The vials and dishes will be put back in the oven again for at least 1 hour and re-weighed after cooling in the desiccator. (The vials are also weighed to help confirm that all solids were successfully removed and, as a fail-safe, account for any solids that may have permanently adhered to the inside of the vial.)

11. Step 10 will be repeated until two consecutive weights are within 0.5 mg.

12. Analysis of the above weights and volumes will provide the solids concentration by weight, including statistical uncertainty, for each well-mixed slurry case considered by Luna.

B) Methodology for analysis of mass concentration for partially consolidated/settled slurries (analogous to those being acoustically tested in Figure 1 of DOE proposal):

1. The partially consolidated/settled slurries acoustically sampled at Luna will be recreated at VIMS by using the same water/particle mixtures and settling times as those employed at Luna.

2. Two-dimensional X-radiographs of the settled slurries (analogous to the example shown in Figure 11 of the DOE proposal) will be acquired using the VIMS x-ray generator and digital flat panel detector.

3. The X-radiographs will provide 1 mm resolution (in a 2-D plane) of x-ray attenuation gray scale. This gray scale will then be calibrated to slurry density using the following slurry weight analysis.

4. After x-raying a given settled slurry, the water will be carefully decanted from above the slurry, and the slurry will be sliced horizontally into 1 cm-thick layers.
5. After slicing, each layer will be individually and separately homogenized by mixing (without adding or losing fluid from the layer in question).

6. Approximately 10 cubic cm of each homogenized layer will be subsampled from each homogenized horizontal slice and will be placed in a pre-weighed aluminum dish.

7. The dish (containing the subsample) will be weighed and placed in a 65 degree Celcius drying oven overnight.

8. The dishes will be cooled in a desiccator and then re-weighed.

9. The dishes will be put back in the oven again for at least 1 hour and re-weighed after cooling in the desiccator.

10. Step 9 will be repeated until two consecutive weights are within 0.5 mg.

11. Analysis of the above weights and volumes will provide the average solids concentration by weight of each 1 cm interval of the settled slurry.

12. The average solids concentrations determined for each 1 cm layer will be regressed against the average x-ray attenuation gray-scale for each corresponding 1 cm layer to provide a transfer function between gray-scale and solids concentration.

13. After gray-scale calibration, the details of the x-radiographs will provide Luna with O(1 mm) resolution of solids concentration in 2-D.
Testing and Analysis of Surrogate Tank Waste Slurries
VIMS Report 2

Date: April 6, 2010

Submitted to: Fritz Friedersdorf and Jake Siegel
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Prepared by: Carl Friedrichs and Grace Cartwright
Virginia Institute of Marine Science
PO Box 1346, Greate Road
Gloucester, VA 23062
Email: cfried@vims.edu, gracec@vims.edu
Office Phone: 804-684-7303, 804-684-7206
### 0.03 Micron

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### 0.05 Micron

#### Diagram

![Diagram of settling time vs. concentration for 0.05 Micron](chart)

#### Table

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In each case, a full liter mixture was poured into a 1 liter beaker with dimensions:
- Inner diameter = 10.3 cm (Luna chamber = 3.965 " = 10.07 cm)
- Beaker height = 15.2 cm (Luna chamber = 5.75" = 14.61 cm)
- Sample height = 7.3 cm (Luna chamber centerline height = 2.875" = 7.3 cm)

In each case, the VIMS sample was stirred until homogeneous. The zero time sample was taken while still stirring. Using a syringe, we pipetted 10 mL at sample times of 0 sec, 30 sec, 60 sec, 90 sec, 120 sec and 180 sec. The zero time sample was collected while still stirring. 0 time is also when the stirrer was turned off. A video recorder was used in an attempt to help get the settled thickness as a function of time.

It was found that most of the videos were non-conclusive for settling thickness (see Luna03.m4v to see the best settling video captured.) We will rerun the experiments using a digital x-ray to better determine the depth-dependent concentrations and settling thicknesses a function of time. Unfortunately, it will be mid-April before the repaired VIMS x-ray generator will be available for our use.

The 1.0 Micron settling experiments were the first size class we tried, and they need to be repeated due to our initially imperfect sampling techniques. We will rerun the 1.0 micron experiment this week (i.e, during the week of April 5-9).
Testing and Analysis of Surrogate Tank Waste Slurries
VIMS Report 3

Date: April 17, 2010

Submitted to: Fritz Friedersdorf and Jake Siegel
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Charlottesville, VA 22903
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Office Phone: 434-220-0143

Prepared by: Carl Friedrichs and Grace Cartwright
Virginia Institute of Marine Science
PO Box 1346, Greate Road
Gloucester, VA 23062
Email: cfried@vims.edu, gracec@vims.edu
Office Phone: 804-684-7303, 804-684-7206
### 1.0 Micron 1st Run

#### 1.0 Micron

#### 1st Run

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<th>Time (sec)</th>
<th>5% wt/wt g/L conc</th>
<th>10% wt/wt g/L conc</th>
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1.0 Micron Rerun

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In each case, the VIMS sample was stirred until homogeneous. The zero time sample was taken while still stirring. Using a syringe, we pipetted 10 mL at sample times of 0 sec, 30 sec, 60 sec, 90 sec, 120 sec and 180 sec. The zero time sample was collected while still stirring. 0 time is also when the stirrer was turned off. A video recorder was used in an attempt to help get the settled thickness as a function of time.

It was found that most of the videos were non-conclusive for settling thickness. We will rerun the experiments using a digital x-ray to better determine the settling thicknesses as a function of time. We hope to begin the x-ray measurements during the week of April 26th.

The 1.0 Micron settling experiments were the first size class we tried, and we decided to repeat them due to unexpected variability that suggested an initially imperfect sampling technique. We reran the 1.0 micron experiment, and the results of both runs are contained here. The “rerun” showed somewhat similar variability, and we now conclude that such variability may be inherent.

The only clear outlier is the 90 sec sample during the first run for the 15% case. A logical interpolation to replace this measurement might be to average the 60 and 120 sec results for the first run. Then it might be logical to use an average of the “1st run” and “rerun” for the best available direct sampling estimates to compare with the acoustic measurements of the 1.0 Micron case.
This is great news Fritz. We'll make sure our final invoices are in to Luna as soon as possible. Cheers, Carl

Sent from my iPhone

On May 24, 2010, at 11:31 AM, Fritz Friedersdorf <friedersdorf@lunainnovations.com> wrote:

Carl,

The results are very nice and the profiles are pretty revealing. I was able to include this matter in the Phase II proposal/final report. Thanks for all your efforts on this program. According to my records, I have received three reports, and used the x-ray data to prepare the final report. I think you have met the necessary deliverables for the subcontract. If you have produced x-ray data for the higher concentrations, I would like to receive that data, but otherwise I consider this work completed.

Best regards,
Fritz

Fritz Friedersdorf
office 434.220.0148
cell 540.808.8947

---

From: Carl Friedrichs [mailto:cfried@vims.edu]
Sent: Thursday, May 20, 2010 12:43 PM
To: Fritz Friedersdorf
Cc: Grace Cartwright
Subject: Fwd: LUNA X-ray profiles

Hi Fritz,

Sorry we weren't able to connect by phone yet this week. In the meantime, here is Grace's latest analysis based on both averaging and taking the median value of color intensity across the width of the portion of the x-ray images containing the slurry. The pdf file contains plots, and the two asc files are ASCII text files containing the actual data shown in the plots.

Assuming the 0 min 00 sec case is well-mixed, one could use its shape to correct the other profiles for spatial imperfections in the x-ray detector. Then color intensity values from X cm above the bottom of the settling chamber could be regressed against the pipette samples we filtered earlier to calibrate the color intensity to particle concentration.

Cheers, Carl

Carl T. Friedrichs, Professor of Marine Science
Virginia Institute of Marine Science
School of Marine Science, College of William and Mary
Mail: VIMS, P.O. Box 1346, Deliveries: VIMS, Route 1208, Gloucester Road
Gloucester Point, VA, 23062-1346, USA
tel. +1-804-684-7303, fax. +1-804-684-7250, email cfried@vims.edu
http://www.vims.edu/~cfried
Begin forwarded message:

From: Grace M Cartwright <gracec@vims.edu>
Date: May 19, 2010 1:06:50 PM EDT
To: Carl Friedrichs <cfried@vims.edu>
Subject: LUNA X-ray profiles

Carl,

Attached are the mean and median profiles of the x-rays for 1.0 Micron 5% wt/wt.

The columns a profile for each of the following elapsed times:

```
t= [  
  '0 min 00 sec',  
  '0 min 34 sec',  
  '0 min 53 sec',  
  '1 min 29 sec',  
  '1 min 47 sec',  
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  '2 min 20 sec',  
  '2 min 34 sec',  
  '2 min 53 sec',  
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  '4 min 01 sec',  
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  '4 min 38 sec',  
  '4 min 57 sec',  
  '5 min 16 sec',  
  '5 min 34 sec',  
  '5 min 51 sec']
```

The y axis, the rows, correspond to image pixels which I converted to cm using the following to generate the y axis for my plots

```
Y=-29.9875:0.0125:0
```

I then plotted with a -Y:

```
plot(Median_X,-Y) and plot(AVG_X,-Y) to get the y-axis to change in cm from the bottom because the x-ray image is actually measured with 0,0 being the top left corner.
```
Grace