Influence of Ore Physical Properties on the Transportable Moisture Limit for Barged Materials

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This study will correlate the physical properties of processed iron ore prepared for barge transportation to its Transportable Moisture Limit (TML). The TML is a rudimentary test developed to identify moisture contents of processed ore at which they may liquefy during transportation. If liquefaction occurs during transportation serious safety issues develop. Currently Doe Run is having difficulties in determining the ore properties that control their measured TML values and consequently are unable to develop an ore processing plan that will allow the ore to be loaded and shipped. This research will attempt to correlate properties such as grain size distribution, relative density, particle size and shear strength with the TML. These properties are known to control liquefaction of sands under earthquake loading and consequently should be important in the TML test.
OBJECTIVE OF THE STUDY

The objective of this study was to identify the physical parameters of Doe Run Lead ore concentrate that influence the Flowable Moisture Content (FMC) and subsequently the Transportable Moisture Limit (TML).

Background

Measurements of the FMC made at Doe Run Company’s Viburnum Laboratory have been found to be higher than subsequent measurements made at port facilities in Louisiana. Consequently, the ore cannot be loaded on barges and shipped until it has been dried. This study was commissioned to determine the factors controlling the FMC and to identify the cause of changes in the FMC.

Preparation of Ore

The lead concentrate being studied comes from the Doe Run Company ore processing facilities near Viburnum, MO. The ore is extracted from the earth and sent through primary, secondary, and tertiary crushing. After crushing it is screened. The material retained on the screens is sent back through the crushers. The ore passing the screen is sent to the milling process. During this process the ore is run through a cyclone feed slump. The overflow is then sent to the lead conditioner. After the conditioner, the ore travels through lead roughers and scavengers. The ore passing this point is sent to cleaners. The material that remains in the rougher is sent to the zinc conditioner where they pass through a zinc rougher and scavenger. The material retained during this process is sent to the tailings dam. The material that passes the roughers are sent to cleaners and on to the zinc thickener and on to filters and then to transportation. The material that is not sent to the zinc-conditioning unit is sent to a lead-copper absorber. At this point it is passed to a copper rougher. The copper that comes off is sent to a copper cleaners and thickeners. Then it is filtered and transported. The lead that is retained at the lead-copper absorber is sent to a lead thickener and on to a lead filter and from there it is transported.

The ore, when ready for transportation, is 100% finer than the No. 100 sieve.

Transportable Moisture Limit

It has been found that moist, loosely deposited fine-grained ore stockpiles can liquefy during transportation by barges. This liquefaction occurs when the vibrations caused by the motion of the barges causes the wet ore to densify (compact). The body weight of the ore is transferred to the water existing in the voids between the ore particles and shear resistance is loss. The ore becomes a very dense liquid and this jeopardizes the stability of the vessel.

The maximum moisture content defining the liquefaction phenomenon is called the Transportable Moisture Limit (TML). This parameter is computed from the results of the
TML  ISO 12742:2000 Copper, lead and zinc sulfide concentrates -- Determination of transportable moisture limits -- Flow table method, a standard set by the National Maritime Association. The test procedure is presented below.

An extensive literature search was conducted to determine the basis of this test. However, there was little available documentation. The only information found was from the National Maritime Association itself. The only other similar test found was the ASTM standard for Flow of Hydraulic Cement Mortar. This standard was obtained and compared with the TML standard and found to have striking similarities. These similarities however were not able to answer the question of the factors involved in flow; it only was a subjective measurement for acceptable consistency.

**Issues:**

During transportation the lead sample seem to increase their moisture content by at least one half of a percent. This makes the TML unacceptable when the lead reached its destination. We need to ensure that the material does not liquefy during transportation. In the past there have been instances of barges tipping over as a result of a liquefied material. We are attempting to find controllable factors that effect TML in order to allow the material to be shipped safely.

**Scope of Work**

Objective: Determine the influence of ore physical properties on the TML.

Purpose: Identify why different ore samples have different TML values.

Procedure: The following list of tests was initially proposed for this study.

Tests:
1. Specific gravity
2. Initial Moisture content
3. Grain size Distribution
   a. Sieve
   b. Hydrometer
4. Relative Density
   a. Maximum
   b. Minimum
   c. As deposited
   d. As compacted in the TML test
5. Degree of saturation
   a. As deposited
   b. As compacted in the TML test
6. Shear strength
   a. As deposited
   b. As compacted in the TML test.
7. **TML ISO 12742:2000 Copper, lead and zinc sulfide concentrates -- Determination of transportable moisture limits -- Flow table method**

However, it soon became apparent that tests number 1 (Specific Gravity), 2 (Grain Size Distribution) and 3 (Shear Strength) were not appropriate. The specific gravity of the ore was relatively constant and determined by assay of the concentrate as was the grain size distribution. It also is obvious that the shear strength of the ore does not influence the parameters under study.

**TML ISO 12742:2000 Test Procedure**

Laboratory Test procedure for Transportable Moisture Limit (TML) on Lead and Zinc Concentrates

**Scope:** Determine as received moisture content.
Determine the flow moisture of the material under impact of a flow table apparatus.
Determine TML (90% of flow moisture)

**Equipment:**
- Standard flow table mounted to an appropriate base.
- Mould.
- Tamper.
- A top loader balance with a weight capacity of 10,000 grams to more with a 0.1 gram readability.
- Glass graduated cylinders. 100 mL and 10 mL capacities.
- Mixing container. (2 gallon bucket)
- Drying oven that can maintain a temperature of 105°C.

**Procedure:**
1. Split out three approximately 2000 gram representative samples of the concentrate.
2. To sample one run an as received moisture content test.
3. To the second sample run a flow moisture as follows: (The third is kept if you exceed the flow point and need to start over.
4. Fill the mould with three equal portions of concentrate manually tamping 35, 25, 20 times respectfully.
5. Position mould in center of flow table, Tap side of mould with tamper and remove.
6. Crank flow table 50 times evenly over a 2 minute period.
7. Observe if the material flows.
8. If not add water. The amount will depend on the appearance of the material. The dryer it looks the more you would add not to exceed 15 mL at one time.
9. Mix thoroughly by hand using a gloved hand or salad fork.
10. Perform steps 2-6 again adding water each time until flow has been established.
UMR Test Procedure Developed to Determine Flowable Moisture of Lead Sample

A study of the TML procedure from a geomechanics perspective identified some procedural issues that may contribute to test result scatter and consistency. Consequently, the test procedure was modified to attempt to reduce that scatter.

**Objective:** To evaluate degree of saturation and flowable moisture as a function of the amount of energy required to obtain flow point.

**Procedure:**

1. Split two approximately 2000 gram representative samples.
2. Use the first sample for an as received moisture content by placing in a 105°C oven overnight.
3. Use the second sample to test as follows:
4. Add enough water to ensure the sample is completely wet. In most cases 75-100 mL of water.
5. Mix water in at 25 mL intervals with a salad fork.
6. Weigh the sample and record the value.
7. Fill the mould approximately 1/3 of the way full with the sample.
8. Weight the remaining sample and record.
9. Use measuring device to determine the height at each of the notches on the flow table. Use these to get an average height.
10. Fill the mould to approximately the 2/3 point and repeat steps 8 & 9.
11. Fill the mould to the top and scrape off any excess sample and repeat step 8. Repeat step 9 if the material is not flush with the top of the cone.
12. Crank the table at a rate of 50 blows per two minutes.
13. When the sample is completely wet around the perimeter and has reached the flow marks it has achieved flow, record the number of blows.
14. Take a representative sample of the tested specimen and place in a moisture tin of known weight.
15. Weigh the sample and place in the 105°C oven overnight.
16. Place remaining sample on a glass plate and spread out. Let dry approximately one hour and repeat steps 6-16 again.
17. Continue the process until the sample reaches approximately 100 blows to achieve flow.
18. The flow moisture contents are plotted at their corresponding number of blows, and a logarithmic line is draw based on these data.
19. The flow moisture at 50 blows is interpolated based on the logarithmic relationship
UMR Procedure for Moisture Content Change with Time

When moisture was added to lead concentrate during testing, an apparent change in moisture content over time was noted. That is, additional moisture was mixed into a specimen, and the mixture was allowed to cure, typically overnight. The lead concentrate behaved noticeably different following curing. That is, a lesser number of blows were required to reach flow after curing compared to immediately after mixing additional water into the sample. In addition, the moisture content of the concentrate determined by oven drying was higher than expected based on the amount of moisture added.

To evaluate this, water was added and mixed with one sample of lead concentrate. The sample was split into four specimens. One specimen was immediately placed in the drying oven to determine the initial moisture and four specimens were sealed in plastic zipper lock bags, with two in single bags and two in triple bags. The starting weight of the samples and bags was recorded, two specimens (one single bag and one triple bag) were placed in a moist room with 100% humidity, and the other two specimens were placed in a moderate humidity lab room. The weights of the bags were periodically monitored to determine if moisture was migrating through the plastic bag to the lead concentrate. At 1, 2 and 3 weeks, a portion of the sample was removed and the moisture content determined by oven drying.

RESULTS

The as-received moisture contents were determined by UMR and compared to the as-provided moistures determined by Doe Run, and the results are presented in the following Table 1.

<table>
<thead>
<tr>
<th>Bucket</th>
<th>Doe Reported MC</th>
<th>Doe Run</th>
<th>UMR MC</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Trial 1</td>
<td>2</td>
<td>3</td>
</tr>
<tr>
<td>B</td>
<td>8.39</td>
<td>8.732</td>
<td>8.519</td>
</tr>
<tr>
<td>C</td>
<td>7.41</td>
<td>7.331</td>
<td>7.319</td>
</tr>
<tr>
<td>D</td>
<td>6.62</td>
<td>7.135</td>
<td>7.118</td>
</tr>
<tr>
<td>E</td>
<td>5.98</td>
<td>6.644</td>
<td>6.246</td>
</tr>
<tr>
<td>Glover</td>
<td>6.79</td>
<td>6.878</td>
<td>6.809</td>
</tr>
</tbody>
</table>

The apparent change in moisture content with time was evaluated by weighing sealed, previously moistened specimens. While the sealed samples exhibited negligible change in total weight over the course of the experiment, the moisture content exhibited an appreciable increase from the moisture content determined immediately following mixing. The results are presented in the following Table 2.
Table 2: Moisture Content Changes with Time

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Sealed in</th>
<th>Special Conditions</th>
<th>Moisture Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Immediately Mixing</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Following 1 Week</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>2 Week Cure</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3 Week Cure</td>
</tr>
<tr>
<td>B-1</td>
<td>1 bag</td>
<td>100% Humidity</td>
<td>7.4</td>
</tr>
<tr>
<td>B-2</td>
<td>1 bag</td>
<td></td>
<td>7.4</td>
</tr>
<tr>
<td>B-3</td>
<td>3 bags</td>
<td>100% Humidity</td>
<td>7.4</td>
</tr>
<tr>
<td>B-4</td>
<td>3 bags</td>
<td></td>
<td>7.4</td>
</tr>
</tbody>
</table>

The unit weights of the tamped lead concentrate were measured prior to operation of the flow table. The range of unit weights for all samples, and sample C and E in particular, are presented in the following Table 3.

Table 3: Unit Weights of Specimens

<table>
<thead>
<tr>
<th>Dry Unit Weight (pcf)</th>
<th>Wet Unit Weight (pcf)</th>
<th>Wet Unit Weight (pcf)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Range</td>
<td>Average</td>
<td>Standard Deviation</td>
</tr>
<tr>
<td>All samples</td>
<td>267.8-293.7</td>
<td>283.1</td>
</tr>
<tr>
<td>Sample C, only</td>
<td>279.3-293.7</td>
<td>286.9</td>
</tr>
<tr>
<td>Sample E, only</td>
<td>267.8-285.0</td>
<td>275.8</td>
</tr>
</tbody>
</table>

For each sample, flow moisture contents were plotted as a function of number of blows necessary to induce flow. A typical curve is presented as follows:

Figure 1: Flow Moisture Content and Number of Blows to Flow – Bucket C

The Doe Run and UMR TML test results are presented in Table 4:
### Table 4: TML Test Results

<table>
<thead>
<tr>
<th>Bucket</th>
<th>Trial</th>
<th>Method</th>
<th>UMR</th>
<th>Doc Run</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>TML</td>
<td>TML</td>
</tr>
<tr>
<td>Bucket B</td>
<td>Trial 1</td>
<td>W-D</td>
<td>7.21</td>
<td>8.06</td>
</tr>
<tr>
<td>Bucket C</td>
<td>Trial 1</td>
<td>W-D</td>
<td>7.94</td>
<td>7.86</td>
</tr>
<tr>
<td></td>
<td>Trial 3</td>
<td>W-D</td>
<td>8.28</td>
<td>7.86</td>
</tr>
<tr>
<td>Bucket D</td>
<td>Trial 1</td>
<td>W-D</td>
<td>6.60</td>
<td>7.71</td>
</tr>
<tr>
<td>Bucket E</td>
<td>Trial 1</td>
<td>W-D</td>
<td>7.23</td>
<td>7.58</td>
</tr>
<tr>
<td></td>
<td>Trial 2</td>
<td>D-W</td>
<td>7.19</td>
<td>7.58</td>
</tr>
<tr>
<td></td>
<td>Trial 3</td>
<td>W-D</td>
<td>7.33</td>
<td>7.58</td>
</tr>
<tr>
<td>Glover</td>
<td>Trial 1</td>
<td>W-D</td>
<td>7.80</td>
<td>7.34</td>
</tr>
<tr>
<td></td>
<td>Trial 2</td>
<td>D-W</td>
<td>7.87</td>
<td>7.34</td>
</tr>
<tr>
<td></td>
<td>Trial 3</td>
<td>D-W</td>
<td>8.60</td>
<td>7.34</td>
</tr>
</tbody>
</table>

### ANALYSIS AND CONCLUSIONS

The study of the flow moisture limit test and procedures related to the measurement of the TML was focused on the physical procedures and physical properties of the ore. Significant conclusions are discussed below.

**Moisture Content Measurements**

Based on the UMR determined as-received moisture content trials, single-operator variations of 0.1 to 0.2 percent were observed. Based on comparison between Doe Run and UMR determined moisture contents, inter-laboratory variations of up to 0.7 percent were noted. The moisture content data was plotted in a Youden Scatter diagram, as follows.

![Youden Scatter Plot](attachment:image.png)

**Figure 2: As Received Moisture Contents**
Unit Weights and TML Results

The total and dry unit weights varied appreciably from trial to trial in TML Testing. This variation likely results in differing strengths and corresponding TML results. The UMR TML results varied from 0.25 to 1.3 percentage points from provided Doe Run TML results. Appreciable differences between Wet to Dry and Dry to Wet UMR TML tests were not noted. The Doe Run and UMR TML results are compared in the following plot.

![Youden Scatter Diagram](image)

Figure 3: TML Results

Moisture Content Changes with Time

The sealed samples exhibited an apparent increase in moisture content of 0.7 to 0.8 percent over a 1-week period, while no corresponding change in weight was observed in the sealed specimens. We speculate this increase in moisture content is due a chemical reaction with the added moisture that results in additional weight being lost in the drying oven. The reaction may take place with an additive on the surface of the concentrate particles, thereby influencing the physical behavior of the bulk concentrate.

As we understand, the concentrate is not shipped in sealed containers and maybe free to gain or lose moisture due to sunlight, humidity or other climatic factors. If the moisture content varies in transit, the reaction with added moisture described above may occur and the properties of the concentrate and the TML may be altered by the variation in moisture content.
RECOMMENDATIONS

Changes in TML During Shipment

It is apparent that some form of chemical reaction is occurring in the ore early in the storage process when moisture is made available. This reaction is increasing the measured moisture content without increasing the total weight of the material. This phenomenon is possibly occurring during shipment of the ore from Missouri to Louisiana. The ore is exposed to a moist atmosphere thereby adsorbing water and changing the available water in the ore body. Consequently when the FML test is conducted after transit, the TML is lower. Since chemical analysis was not a part of this study, this chemical reaction needs to be investigated thoroughly to define its controlling parameters.

Flow Moisture Test Procedure

Based on the results of this investigation, we recommend the following changes to the existing TML test:

* Hand Tamping should be replaced by tamping with a controlled effort. This should provide more uniform unit weights between trials

* Flow moisture should be determined based on several tests at varying moisture contents/numbers of blows, and a logarithmic relationship. This will help to increase test precision, and reduce the need to add small amount of water to produce slumping at exactly 50 blows.

Based on the range of the TML results, consideration should be given to the statistical significant of a relatively small number of tests compared to the volume of material shipped.

FURTHER INVESTIGATIONS

A study of the chemical reactions that appear to be occurring due to the addition of moisture need to be initiated.

In addition, we recommend that consideration be given utilizing in-situ cone penetrometer measurement as an alternative or supplement to the present TML test for assessing ore liquefaction potential. This test method would characterize a large volume of material relatively quickly, and results could be correlated to lab measured shear strengths and liquefaction potential.