Turbidity Testing Discussion

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FACE TO FACE

Turbidity- What is it? Why Measure it?

- Turbidity is typically defined as the relative clarity of a liquid, or the cloudiness or haziness of a liquid. The haziness in the liquid is caused by large numbers of particles suspended in the liquid. When light is passed through the liquid the particles cause the light to scatter.
- Turbidity testing as it relates to chromite sand can be thought of as a measurement of the light scattering caused by suspended particles in a liquid.
- When evaluating chromite the particles that stay suspended in the water are generally thought to be low melting point silicate impurities which are thought to contribute to certain types of casting defects.
  - see paper "Chromite *Double Skin Defect on Heavy-Section Steel Castings*" by J.D. Howden for more information on these types of defects.
- So lower turbidity values are generally thought to be advantageous in the casting process
- The typical test method for most turbidity tests involves agitating chromite sand in water for a fixed amount of time and then allowing the heavy chromite sand particles to settle for a fixed amount of time and measuring the resulting water for turbidity.

Outline of Discussion

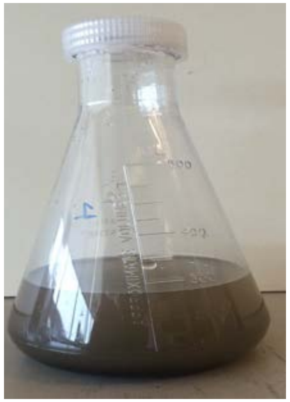
1) Two major controls are required to ensure reliable, consistent results:
    1) Sample Preparation
    2) Instrument for measurement of turbidity

2) Review of historical procedures and instruments
3) Experiments to develop new standardized sample preparation
4) Proposal of new method

Sample Preparation Discussion

Sample Preparation

- All Turbidity tests require a chromite sand sample to be agitated in a container with water (typically de-ionized or distilled)
  - historically the agitation has been done by hand
- After agitation, the material is left to settle for a given amount of time and then the liquid is poured off into a separate container. The resulting liquid is them measured for turbidity
- The agitation time, aggressiveness of the agitation and settling time all have a significant effect on the measured turbidity



Turbidity samples after agitation

Measurement Test Procedure Discussion

Historical Method- Jackson Candle

- Turbidity has historically been tested and reported using the Jackson Candle method or Jackson turbidity test .
- The test is performed with a Jackson turbidity apparatus consisting of a flat bottom glass or borosilicate cylinder (Jackson tube), a ring stand to hold the cylinder and a candle placed below the cylinder.
- The candle is lit and placed approximately~10cm below the flat bottomed glass cylinder
- The candle flame is viewed from above (through the bottom of the cylinder) while the turbid liquid is poured slowly into the top of the cylinder
- The endpoint is determined when the operator can no longer see the candle flame clearly through the bottom of the cylinder- the flame is obscured by the liquid

Historical Method- Jackson Candle

- The cylinder is given a scale or "calibrated" with solutions of known concentrations of silica (SiO_2 in ppm) prepared from diatomaceous earth
- The known solutions are added to the cylinder while observing the candle flame and the endpoint for each standard is marked on the side of the cylinder creating a scale to compare unknown samples against.
    - The further the marking from the candle flame the lower the turbidity, i.e. the liquid is clearer
- The height of the liquid of a test sample at the endpoint is compared to endpoints of the known standards to obtain a final result.
- Results are reported in in parts per million of silica (ppm) in reference to the concentration of the silica solutions used to calibrate the cylinder.
- Results obtained in the fashion are also commonly referred to as Jackson Turbidity units (JTU)

Limitations of the Historical Method

- There are many issues that could affect the consistency and reliability of the results when using the Jackson Turbidity method. Examples include:
  - Variability in determining endpoints between operators
  - Calibration inconsistencies between labs and operators- Each lab left to develop their own calibrations
  - Consistency in diatomaceous earth used to make standards- No certified diatomaceous earth
  - Inconsistencies in test set up -
    - Differences in candle flames resulting from larger/ smaller candle
    - Differences in distance between flame and cylinder
- The use of the candle flame as the light source and the human eye as the detector presents drawbacks for analyzing samples with very fine suspended particles, color, and low turbidity.
  - The combination of the limitations of the human eye and the characteristic of the light source (candle) lead to problems standardizing this test method and producing repeatable results.

Modern Approach- better instruments

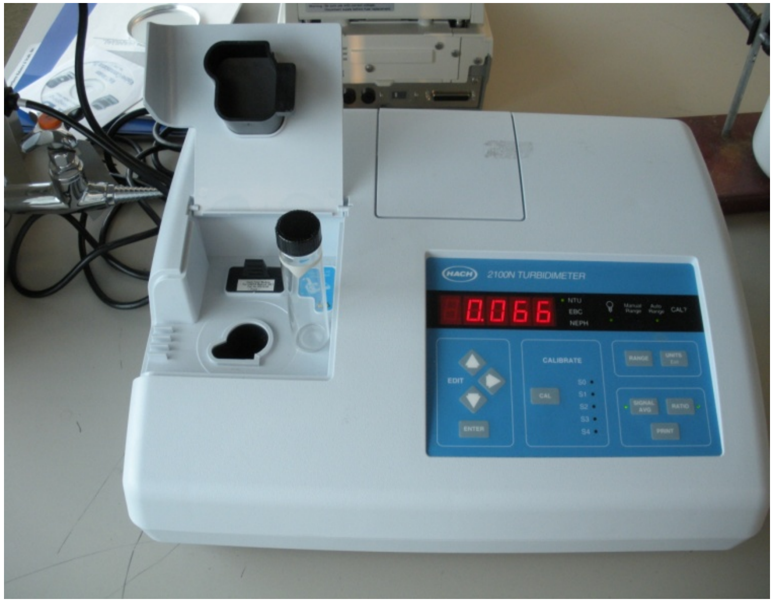
- Modern testing equipment which use a fixed light source are known as Nephelometer.
- Nephlometers make use of a electronically powered light source to provide stability and consistency
  - light is reproduced at the same wavelength at all times
- These instruments detect the light scattered in the liquid at a fixed angle from the incident beam/ light source
  - usually measured at 90^o or 180^o
- Modern techniques utilize stable, traceable standards for turbidity
  - Typically Formazine (Formazin) or stabilized Formazine
  - These are commercially available making standardization across labs much easier
  - Result are reported Nephelometric Turbidity Units (NTU’s)

Modern Instrument- Nephelometer

- There are different types for different applications, not all are suitable for expected turbidity levels of chromite sand or other mineral aggregates
- For low level turbidity measurements, such as evaluation of drinking waters, a portable turbidity meter with only a single 90° detector may be suitable, while higher turbidity samples (>100 NTU) may require dilution to be analyzed on this instrument .
  - Dilutions can introduce error and turbidity does not always produced a linear response
- Other instruments utilize multiple detectors. A common set up is the three detector system (traditional 90° detector, forward-scatter detector, and transmitted light detector) which helps reduce the effects of color differences in turbidity measurements.
  - This style of instrument is more suited to chromite turbidity applications, does not require dilution for samples under 4000 NTU, and is easy to standardize with readily available standards.
  - A Hach 2100 N laboratory turbidity meter is recommended for the above reasons

Examples of Modern Nephelometers

Portable style with single 90° detector- Accurate between 0-100 NTU without need for dilution of sample



Benchtop model with multiple detector arrangement and accurate between 0-4000 NTU

Comparison of Turbidity Units

- The Nephelometric Turbidity Unit (NTU), Formazine Turbidity Unit (FTU), Formazine Attenuation Unit (FAU), Formazine Nephelometric Unit- FNU -are all units based on the use of primary Formazine suspensions standards, but are not necessarily equivalent and do not correlate well to each other due to differences in measurement parameters
  - For instance NTU and FAU are both calibrated with the same standards but NTU are measured using a 90 degree incident white light source while the FAU is measured at 180 decrees with an infrared light source.
  - There is no universal conversion between ppm/ JTU and NTU

NTU ≠ JTU or ppm

Sample Preparation for Turbidity Measurement

Comparison of Agitation Methods

- An experiment was conducted to demonstrate the variation associated with agitating the chromite sand and water by hand
- 12 operators were given the same sample of chromite sand and the same testing procedure and the were asked to test the turbidity
- First the operator was asked to perform the procedure by shaking the sand and water by hand
- Next the operator was asked to use a mechanical device to agitate the sand and water
  - A Burrell Wrist Action Shaker was used for this experiment
  - The shaker mimics the action of a human wrist

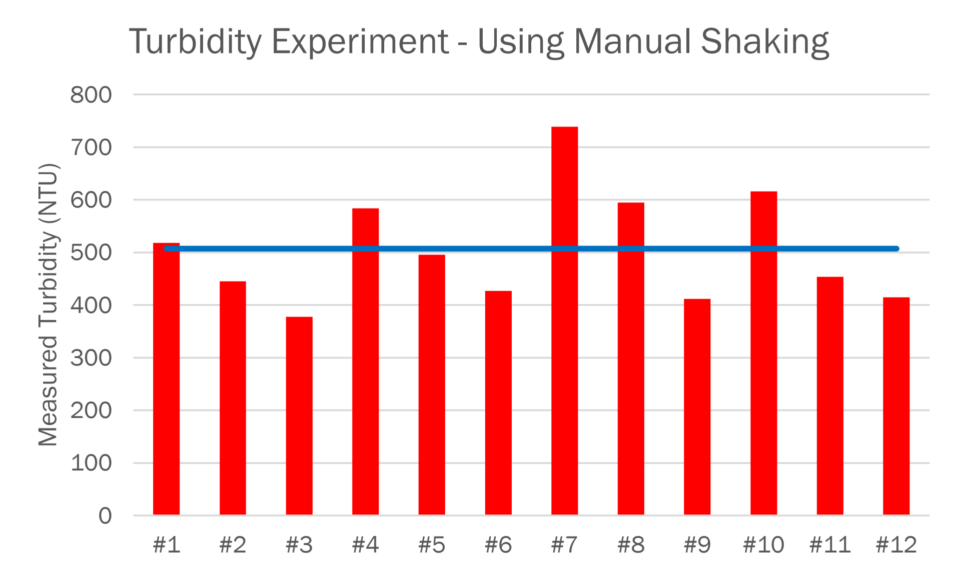
Turbidity Results in NTU

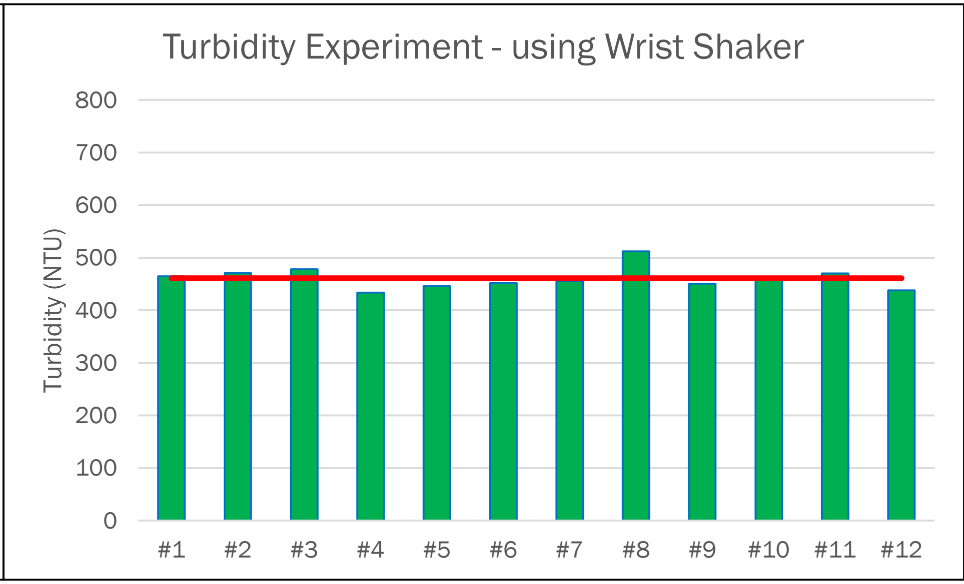
1. Manual Shaking

- Average = 507 NTU
- Standard Deviation = 107.3

2. Using Mechanical Shaker

- Average = 461 NTU
- Standard Deviation = 20.8





The standard deviation is improved greatly by using a standard , reproducible method of agitation

Proposed Standard Procedure – Turbidity

MTI suggested improvements to historical turbidity methods

- Use a Wrist Action Shaker to significantly reduce the variability in shaking between operators while still achieving an aggressive shake
- Use a Hach 2100 N (or similar) Laboratory Nephelometric Testing Unit
    - Alternative instrument should use a white light source and measures at 90^{o} to the incident beam
- Measure and report turbidity values in NTU

Equipment for Turbidity Testing

Before beginning any testing or activity in a laboratory setting, please take the time to evaluate any risks associated with the task to make sure the testing can be done safely. Also ensure the necessary personal protective equipment (PPE) is used.

- 500 ml polycarbonate Erlenmeyer flask with screw top and cap
  - (from Thermo Fisher/ Nalgene or equivalent) –
  - polycarbonate flasks have been found to reduce the risk associated with glass flasks cracking or breaking.
- Plexiglas/ Lexan shield between operator and shaker to prevent any contact between moving instrument and flask and operator
- Burrell – Wrist Action Shaker (amplitude lever set to 10)
- Hach 2100N laboratory Turbidity Meter (calibrated quarterly with Hach Stab Cal standard solutions of 0, 20, 200, 1000 and 4000 NTU as per manufacturer's instructions)
- Turbidity Free water (Distilled/ De-ionized Water)
- Drying oven capable of 110°C
- Scale (Capable of accurately measuring at least 250 grams)

Turbidity Testing Procedure

*Before beginning test, inspect the equipment and lab ware for any damage or wear and review laboratory safety practices to ensure the task can be done safely.

1. Dry a sufficient amount of chromite sand to a constant weight in a drying oven (110°C)
2. Weigh 250 grams distilled water into a beaker and add to 500 ml polycarbonate flask
3. Weigh 50g dry chromite sand in a weigh boat and add to 500 ml polycarbonate flask
4. Place polycarbonate flask into Grip of Wrist Action Shaker and tighten until secure (do not over tighten or risk cracking the neck of the flask) and shake for 1 minute on maximum amplitude (separate lever on side on shaker, set to “7”).
5. Place shield in from of shaker arm in such a manner to prevent anyone from being able to come in contact with the moving arm of the shaker during movement (see Image 1 in Appendix A)
6. Turn Wrist Action Shaker on.
7. After shaking 1 minute, turn shaker off.
8. Remove shield from in front of shaker and carefully remove polycarbonate flask from shaker grips.
9. Allow Flask to stand for 30 seconds, after settling seconds pour off turbid water into beaker.
10. Carefully pour water from beaker into clean, dry 30 ml Hach sample vial, secure cap
11. Wipe vial with cloth, apply silicone oil to vials as necessary, and insert into Hach instrument
12. Make sure ratio and signal averaging modes are active (lights will be green on these keys), and make sure the units are NTU
13. Record first value displayed by Hach, as the numbers fluctuate due to settling while in instrument

Appendix A- Images

Image 1: Plexiglas shield in front of shaker

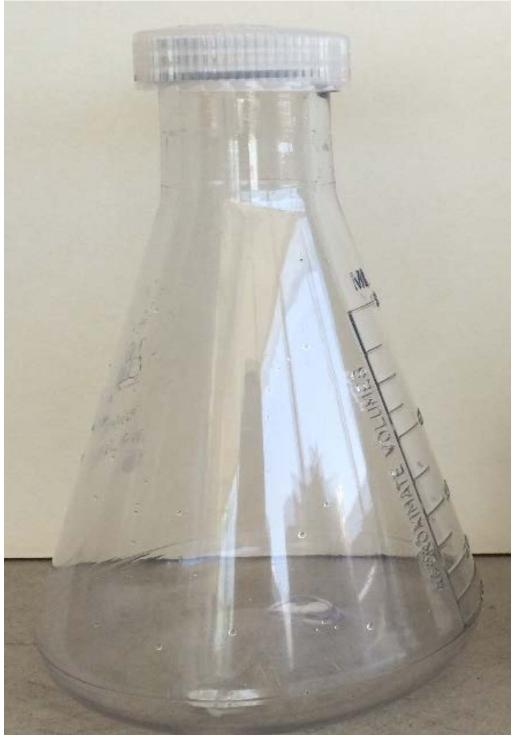


Image 2: Polycarbonate 500 ml Erlenmeyer Flasks

Appendix A- Images

Image 3: Hach 2100 N turbidity meter

Image 4: Burrell Wrist Action Shaker