

OGMCOAL - Crandall Canyon : Polymer Testing Procedure

From: Steve Christensen
To: OGMCOAL
Date: 12/6/2010 2:21 PM
Subject: Crandall Canyon : Polymer Testing Procedure
Attachments: Presence of Polymer Procedure Nov 2010.pdf

>>> "Marrelli, Dana" <dmarrelli@coalsource.com> 12/6/2010 10:18 AM >>>

Hello Steve and Kevin,

Here is the procedure for testing for residual polymer with the coagulant. Please feel free to call Randy at WaterSolve direct if you have any questions. Randy's number is (616) 575-8693.

Thank you,

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DETERMINATION OF THE PRESENCE OF POLYMER USING THE FLOCCULATION METHOD

1. PURPOSE

The purpose of this method is to provide a means of qualitatively determining the presence of a flocculent or coagulant within a solution, water sample, etc. Additionally, in cases where the polymer present within a sample is known, this method can be tentatively used as a quantitative measure.

2. PRINCIPLE

Slurry of kaolin clay is very easily flocculated or coagulated when either a flocculent or a coagulant is present. Therefore, for qualitative purposes, the sample being tested is mixed with kaolin clay slurry, and the effects are visually assessed by comparison to a blank. If polymer is present, significant coagulation or flocculation will be seen from the sample being tested as compared to that seen in the blank.

These principles can also be applied to the quantification of the concentration of polymer present within a sample. However, the exact product present within the sample must be known, and the water used to prepare all solutions must be similar in pH, hardness, etc. to the water present in the sample being tested. Standard solutions containing the known product are prepared at different concentrations. Each solution is then mixed with a kaolin clay slurry, and the settlement time of each is measured. A curve and the equation of the curve are then generated from the obtained results, the settlement time of the unknown sample is measured, and the concentration of the specific product in the unknown sample is calculated by substitution into the obtained equation.

3. PROCEDURE DESCRIPTION

3.1 Hazards and Safety Precautions

The information provided below is not a substitute for the MSDS but is supplementary to it. All users must have read and be familiar with the appropriate manufacturer's MSDS before using the chemicals listed below.

All unknown water samples and polymer solutions should be considered irritants to the skin and eyes.

Contact with calcium chloride powder may cause irritation to the skin, eyes, or respiratory tract.

General laboratory safety procedures should be followed.

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3.2 Apparatus and Reagents

Apparatus	Reagents
General Apparatus: 1) 100 mL Glass Mixing Cylinders with Stoppers 2) Syringes (as appropriate) 3) Three Place Top Loading Balance 4) Bottles with Caps (as appropriate) Additional Apparatus Required for Quantification Procedure: 1) Stopwatch 2) Graduated Cylinders [200 mL and 50 mL] 3) 400 mL Glass Beakers (2)	1) ACS Grade Calcium Chloride 2) Laboratory Grade Kaolin Clay

3.3 Procedures

Preparation of the Clay Slurry

1. Preparation of a 1% Calcium Chloride Solution
 - a. Determine how much 1% calcium chloride will be needed to perform the required testing. Please note that approximately 1.7 mL of a 1% calcium chloride solution is required for each test.
 - b. Calculate the required weight of calcium chloride needed to obtain the desired weight of 1% calcium chloride solution using the equation below.

$$W_1 = \frac{W_2 \times C_2}{C_1}$$

Where:

W_1 = Weight of Calcium Chloride Required to Prepare the Solution (g)

C_1 = Concentration of the Calcium Chloride Being Used (%)

W_2 = Desired Weight of 1% Calcium Chloride Solution (g)

C_2 = Concentration of Calcium Chloride Solution Required (1%)

- c. Tare an appropriately sized bottle on a three place top loading balance.
 - d. Accurately weigh out the calculated weight of calcium chloride required into the tared bottle. The accuracy of this weight should be ± 0.002 g. Add deionized water to the bottle to achieve the desired final solution weight. For example, if 100 g of 1% calcium chloride solution is desired, add 1.000 g of pure calcium chloride to a tared bottle, and add deionized water to achieve a final weight of 100.000 g.
2. Preparation of the Clay Slurry

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- a. Determine how much clay slurry will be needed to perform the required testing. Please note that 5 mL of slurry is needed for each test.
- b. Tare an appropriately sized bottle on a three place top loading balance.
- c. Into the tared bottle, weigh out 1 parts laboratory grade Kaolin clay and 3 part 1% calcium chloride solution.
- d. Cap the bottle, and shake vigorously until the contents are homogeneous.

Qualitative Determination of the Presence of Polymer in a Sample

1. Perform a blank as follows. To a 100 mL glass mixing cylinder, add 5 mL of the previously prepared clay slurry and 90 mL of water. Please note that the water used for this blank should be similar in quality to the water present in the sample which is to be tested with regard to hardness, pH, etc.
2. To a second 100 mL glass mixing cylinder, add 5 mL of the previously prepared clay slurry and 90 mL of the sample being tested.
3. Invert both cylinders three times, and visually assess whether flocculation or coagulation has occurred in the sample being tested by comparing the settlement rate and floc size of the clay in the sample cylinder to the settlement rate and floc size of the clay in the blank sample. Record observations.

Quantitative Determination of the Presence of a Specific Product in a Sample

1. A quantitative determination of the presence of polymer in a sample can only be performed with any accuracy in cases where the exact product present within a sample is known and when the quality of the water being used to prepare all solutions does not vary significantly from the quality of the water sample being tested.
2. Obtain a sample of the product which is known to be present within the sample to be tested.
3. For Liquid Dispersion or Emulsion Grade Products – Prepare a 0.5% standard stock solution using a sample of the product known to be present and water which is similar in quality to the water present in the sample to be tested.
 - a. Measure 200 mL of sample water in a graduated cylinder.
 - b. With a 1 mL syringe measure 1 mL of the product known to be present.
 - c. Cap the cylinder and mix vigorously for two minutes.
4. Prepare a series of standard solutions with various ppm from the stock solution.
5. Measure the settlement time of each solution.

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- a. To a 50 mL glass graduated cylinder with topper, add 5 mL of the previously prepared clay slurry and 90 mL of the sample with a predetermined ppm.
 - b. Invert the cylinder three times, and using a stopwatch, measure the time taken for the mudline formed by the flocculated clay to travel from the 50 mL mark on the cylinder to the 40 mL mark on the cylinder. The stopwatch is to be started after the third inversion and stopped when the mudline reaches the 40 mL. This constitutes the settlement time, in seconds, given by the known ppm standard.
 - c. Repeat Steps a and b for the various known ppm standard, and for the water sample being tested.
6. Determine the quantity of the specific product in the sample being tested.
- a. Using the obtained settlement times for the standards, plot a graph of settlement time vs. concentration.
 - b. Fit this data with the best fit curve, and obtain the equation of the generated curve from the software.
 - c. Substitute the obtained settlement time from the unknown sample for Y in the obtained equation. Solve this equation for X. The resulting value obtained for X is the concentration of the specific product, in mg/L, within the sample.