



Method 1.15 - Official Methods: clarifier mud insoluble solids

1. Rationale

This method is applicable to clarifier mud samples and is used to obtain data for cane payment purposes.

2. Principle

The clarifier mud sample collected is prepared and by the addition of Celite 577, the amount of insoluble solids is determined and is used to correct the mass of clarifier mud.

3. Apparatus

- 3.1 Squat beakers (100 cm³, 250 cm³)
- 3.2 Light duty balance
- 3.3 Buchner funnel (110 mm ϕ)
- 3.4 Buchner flask
- 3.5 Drying oven at 105°C
- 3.6 Dessicator
- 3.7 Filter paper, Whatman No. 6 or equivalent (150 mm ϕ), dried at 105°C for 3 hours and stored in a dessicator
- 3.8 Stirring rod (150 mm \times 6 mm)

4. Reagents

- 4.1 Filter aid - Celite 577 (dried and stored in a dessicator)

Celite is an inert powder and inhalation may cause asbestosis of the lungs. Wear a dust mask during use.

5. Procedure

Zero the balance.

Place a 250 cm³ beaker, a 100 cm³ beaker and a filter paper on the balance pan.

Add approximately 12 g of filter aid to the 250 cm³ beaker and approximately 4 g of filter aid to the 100 cm³ beaker.

Note the total mass to the nearest 0.01g.

Place the 250 cm³ squat beaker on the bench top.

Agitate the composite sample and rapidly transfer 150 g to the 250 cm³ beaker.

Return the 250 cm³ beaker plus contents to the balance pan and note the total mass (which again includes the mass of the filter paper, 100 cm³ beaker, etc.) to the nearest 0.01g.

Connect the vacuum to the Buchner flask and adjust the vacuum to give a differential pressure of no more than 3 kPa.

Transfer the filter paper to the Buchner funnel and wet it with water so that the paper fits tightly around the sides of the funnel.

Precoat the filter paper with the 4 g of filter aid held in the 100 cm³ beaker. This is done by adding 30 cm³ water to the filter aid in the beaker and then pouring the slurry rapidly from the beaker directly onto the filter paper. Pouring must be executed so that the slurry is spread evenly over the whole area of the paper. Rinse all the filter aid adhering to the beaker into the funnel.

Stir the contents of the 250 cm³ beaker so that the filter aid and mud are well mixed.

Filter the sample by pouring it slowly down a glass rod onto the precoated filter paper, taking care not to flood the filter aid surface with sample, *i.e.* pour the sample in at a rate lower than the drainage rate of the filter; this is important to ensure quick filtration. This initial filtration should be done at a differential pressure of 3 kPa. During filtration the beaker contents must be stirred occasionally and visual checks must be made on the clarity of the filtrate to ensure that no by-passing of filter aid or suspended solids occurs.

Rinse the beaker with well distilled water and pour the rinsings into the funnel.

Increase the vacuum to a differential pressure of about 50 kPa and wash the filter with ten 30cm³ aliquots of distilled water, allowing the filter to drain between additions. Finally allow the filter to drain for 5 minutes under vacuum.

Release the vacuum and transfer the filter paper and contents quantitatively back to the 250 cm³ beaker. Care must be taken to ensure that no filter aid remains adhering to the sides of the funnel. Do not compress the filter paper in the 250 cm³ beaker.

Dry the 250 cm³ beaker and contents and the 100 cm³ beaker at 105°C for 4 hours.

Cool in a dessicator for 30 minutes and weigh. Record the mass to the nearest 0.01g.

6. Calculations

Where re-routing of clarifier mud is partial or intermittent (within a week) the insoluble solids determination is as follows:

$$\text{Insoluble solids \% press mud} = \frac{(M_3 - M_1)}{(M_2 - M_1)} \times 100$$

where M_1 ≡ mass of beaker and filter aid (g)
 M_2 ≡ mass of beaker and sample before drying (g)
 M_3 ≡ mass of beaker and sample after drying (g)

For total and continuous re-routing of clarifier mud the determination is as follows:

The tons insoluble solids in clarifier mud is taken to be the same as the tons insoluble solids in mixed juice.

$$\text{Insoluble solids \% mud} = \frac{\text{tons insoluble solids in mixed juice} \times 100}{\text{tons mud}}$$

7. References

Brokensha, MA, (2000). *Official Methods for the massing, sampling, analysis and calculation for clarifier mud returned to diffuser/milling tandems*. SASA Council, ADM620/MAB/cp.