

BOA 0-52/11

A METHYLENE BLUE TEST FOR BENTONITE

A1.1. DESCRIPTION

The methylene blue capacity is an estimate of the cation exchange capacity of bentonite. The true cation exchange capacity of the bentonite will probably be somewhat higher than the methylene blue capacity. A well dispersed sample of the bentonite is titrated with methylene blue solution until a "blue halo" appears around a drop of dyed bentonite solids on filter paper.

A1.2.2 EQUIPMENT

1. REAGENTS

- a. Methylene blue solution ($C_{16}H_{18}N_3SCL$) MB 319,85 (0,5%) from methylene blue "MERCK NR" 6040.
- b. Tetrasodium pyrophosphate $Na_4P_2O_7 \times 2H_2O$ (saturated solution - distilled water - Sulfuric acid (H_2SO_4 , 5N solution) - Standard bentonite sample (S&B Quality Control Code number "80018 Wyoming").

2. APPARATUS

- a. Balance sensitivity 0.0001 g.
- b. Conical Erlenmeyer Flask 250 ml.
- c. Stirring glass rod.
- d. Pipette 10 cm^3 .
- e. Graduated cylinder 50 cm^3 .
- f. Whatman No 1 or Schleicher & Schue (589²) 25 mm filter paper.

g. Burette : 50 cm³.

h. Beaker 1000 cm³.

i. Magnetic stirrer with stirring bar (coated).

82%

80%

95%

45%

55%

A1.3. PREPARATION OF METHYLENE - BLUE SOLUTION (0,5%)

5 g methylene blue solution MERCK NR 6040 is dissolved in 600 ml of warm distilled H₂O. After 24 hours the solution is transferred to a 1000 ml beaker. The undissolved residue is boiled in approximately 100 ml H₂O. After cooling the solution is passed from a filter paper to check if the methylene - blue is completely dissolved. Add the solution to the beaker. Add distilled water up to 1000 ml. Stir with a glass - rod until homogeneity is established, at room temperature. The solution is kept in a dark bottle, away from the light and is used only for a month.

A1.4. PROCEDURE

105°C/overnight

a. Accurately weigh 0,500 g of dried bentonite and add to the Erlenmeyer Flask containing 5 cm³ saturated tetrasodium pyrophosphate solution and 50 cm³ distilled H₂O.

b. Stir well for 3 minutes and boil gently for 5 minutes.

c. Cool to room temperature and add 2 ml H₂SO₄ 5N.

d. Stir well for 30 sec.

e. Titrate the solution against Methylene blue solution (usually 80 -90% of the necessary volume of the solution is added at once). After each addition mix the contents of the flask for 1 minutes and remove one drop of the suspension with the stirring glass rod and place on the filter paper. The initial endpoint of the filtration is reached when dye appears as a faint blue ring or halo surrounding the dyed solids.

f. When the initial endpoint is detected, stir the flask for an additional one minute and place another drop on the test paper. If the blue ring is again evident the endpoint has been reached. If the ring does not

appear, continue as in Par(e) until a drop taken after stirring another minute shows the blue tint or halo.

A1.5 CALCULATIONS

Procedure A1.4 is repeated using the "internal" standard sample (Code No 80018).

$$G = V_1(\text{ml}) \cdot 100 / V_2(\text{ml})$$

G : Methylene Blue Value - (% std. Wyoming sample)

$V_1(\text{ml})$: METHYL. BLUE ABSORBED BY THE UNKNOWN SAMPLE

$V_2(\text{ml})$: METHYL. BLUE ABSORBED BY THE WYOMING SAMPLE

% MONTMORILLONITE (approx.) = $G \times 0.68$

Std \rightarrow 29 ml

$$35 \text{ ml} / 29 \times 100 = X \cdot 0.68 = \text{Mont \%}$$

$$\frac{35.5}{29} \times 100 =$$

83%