Methylene Blue Kit

Instruction Manual

Manual No. 209860, Revision E

Instrument No. 209679 & 209694

fann®
Fann Instrument Company

Methylene Blue Kit Instruction Manual

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Houston, Texas, USA

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Table of Contents

1 Introduction....4
1.1 Methylene Blue Capacity of Drilling Fluids....4
1.2 Methylene Blue Capacity of Binder in Foundry Sand....4
1.3 Document Conventions....5
2 Safety....7
3 Equipment and Reagents....9
4 Operation....10
4.1 Drilling Fluids Test Procedure....11
4.2 Binder in Foundry Sand Test Procedure....12
5 Equipment Care....15
6 Parts List....16
7 Warranty and Returns....17
7.1 Warranty....17
7.2 Returns....17

List of Figures

Figure 3-1 Methylene Blue Kit....9
Figure 4-1 Spot Test for End Point of Methylene Blue Titration....10
Figure 4-2 Percent Binder in Foundry Sand....14

List of Tables

Table 6-1 Methylene Blue Kit Parts List....16
Table 6-2 Required Reagent....16
Table 6-3 Optional Reagents....16

1 Introduction

Fann Instrument Company offers a complete Methylene Blue Kit, containing all the reagents and equipment required to perform the methylene blue test according to API Recommended Practice.

The methylene blue test determines the capacity of clay to absorb cations from a solution, and thereby predict how the clay will react. Clay may be a component of a drilling fluid, a binder in foundry sand, or a component in another application. This test is based on the property of clays known as cation-exchange capacity; clays can exchange some of their ions for the ions of certain other chemicals. The number of ions available for this exchange varies with different clays. For example, Western Bentonite, has more base exchange capacity than Southern Bentonite. However, only the reactive portions of clays are involved in the cation exchange process.

In this test, the clay particles are coated with methylene blue, giving them a distinct color until all ions available in the clay cation exchange process have been used. Excess methylene blue remains unaffected in solution around the particles and forms a blue-green tint that radiates from the darker spot, similar to a halo. Development of the halo shows that the total absorption capacity of the clay has been reached, and is noted as the endpoint.

1.1 Methylene Blue Capacity of Drilling Fluids

The methylene blue capacity of a drilling fluid indicates the amount of reactive clay (e.g., bentonite or drill solids). The methylene blue capacity provides an estimate of the total cation exchange capacity of the drilling fluid solids. Methylene blue capacity and cation exchange capacity are not necessarily equivalent. Usually, methylene blue capacity is less than the actual cation exchange capacity.

Drilling fluids frequently contain substances, in addition to reactive clays, that absorb methylene blue. Pretreating with hydrogen peroxide may be necessary to remove the effect of organic materials, such as lignosulfates, lignites, cellulosic polymers, or polyacrylates. Methylene blue is added to the pre-treated sample until saturation which results in a dye halo surrounding the solids. See Figure 4-1.

1.2 Methylene Blue Capacity of Binder in Foundry Sand

The methylene blue test is a simple, fast test for measuring the amount of effective binder in foundry molding sands. This test produces accurate, reproducible results in 10 to 15 minutes.

Only reactive portions of clay participate in the cation exchange process. Finely ground seacoal, wood flour, or cereal does not absorb methylene blue.

The cation exchange capacity of clays is destroyed when clays are exposed to high temperature. The degree of destruction is depends on the temperature and time that the clay is exposed. A decrease in cation exchange capacity is accompanied by a proportional decrease in bonding capacity. The altered portion of the clay cannot be rehydrated, and bonding capacity cannot be restored. Since clay is the only ingredient in a sand mix with cation-exchange capacity, a precise measurement of this property is also a measure of the amount of effective binder in the sand.

Methylene blue is added to this sample until saturation, noted by the dye halo surrounding the solids suspension. See Figure 4-1.

1.3 Document Conventions

The following icons are used as necessary in this instruction manual.

NOTE. Notes emphasize additional information that may be useful to the reader.

CAUTION. Describes a situation or practice that requires operator awareness or action in order to avoid undesirable consequences.

MANDATORY ACTION. Gives directions that, if not observed, could result in loss of data or in damage to equipment.

WARNING! Describes an unsafe condition or practice that if not corrected, could result in personal injury or threat to health.

ELECTRICITY WARNING! Alerts the operator that there is risk of electric shock.

HOT SURFACE! Alerts the operator that there is a hot surface and that there is risk of getting burned if the surface is touched.

EXPLOSION RISK! Alerts the operator that there is risk of explosion.

2 Safety

Safe laboratory practices and procedures should be observed while using the methylene blue kit.

The chemicals in this kit may cause hazard to the user's health by direct contact, inhalation, or ingestion. Explosion and fire hazards may also exist.

Read all warnings, precautions, and hazard classifications (fire, health, and reactivity) on the container labels. Refer to each MSDS for detailed information on handling, reactivity, storage, and other safety information. If personal contact or an environmental accident occurs, follow the counteractive measures written on the label or MSDS.

Never pipette any chemical by mouth. Always use a pipette filler/dispenser (P/N 206730).

Methylene blue solution is an irritant. Avoid prolonged with skin or contact with eyes. Do NOT inhale vapors. Do not take internally. Wear approriate safety equipment. If contacted, flush with water for 15 minutes.

Hydrogen peroxide is a strong oxidizer. Avoid its contact with organic materials. Avoid contact with skin and eyes. It may cause skin irritation or burns. Do not inhale vapors. Wear appropriate safety equipment. If contacted, flush with water for at least 15 minutes.

Sulfuric acid is corrosive and may cause chemical burns. Avoid contact with skin or eyes. Do not inhale vapors or take internally. Wear appropriate safety equipment. If contacted, flush with water for at least 15 minutes. Get medical attention.

Make sure the electrical cord on the hot plate is in good condition and equipped with a grounding plug.

Read and follow the operating instructions for the hot plate. Do not leave it unattended while heating.

Use caution when handling hot flasks or other hot laboratory containers. Wear appropriate hand protection.

3 Equipment and Reagents

The Methylene Blue Kit contains the materials (except sulfuric acid) that are required to test the cation-exchange capacity of drilling fluid solids and clays, or foundry sand binder clays:

- Methylene Blue solution, reagent grade
- Hydrogen Peroxide, 3% solution
- Sulfuric Acid, 5N (not included; sold separately)
- Syringe, 5 ml
- Erlenmeyer Flask, 250 ml
- Graduated Cylinder, 50 ml
- Stirring Rod
- Pipettes, 1 ml and 10 ml
- Hot Plate
- Filter Paper, API, 5-in. (12.5 cm) diameter

The Methylene Blue Kit is available with a 115V or 230 V hot plate:

- Methylene Blue Kit, 115V, Part No. 209679
- Methylene Blue Test Kit, 230V, Part No. 209694

Figure 3-1 Methylene Blue Kit

4 Operation

The Methylene Blue Kit is used in the following test procedures:

- Methylene Blue Capacity of Drilling Fluids
- Percent Binder in Foundry Sand

Refer to Figure 4-1 to assist in determining the methylene blue endpoint.

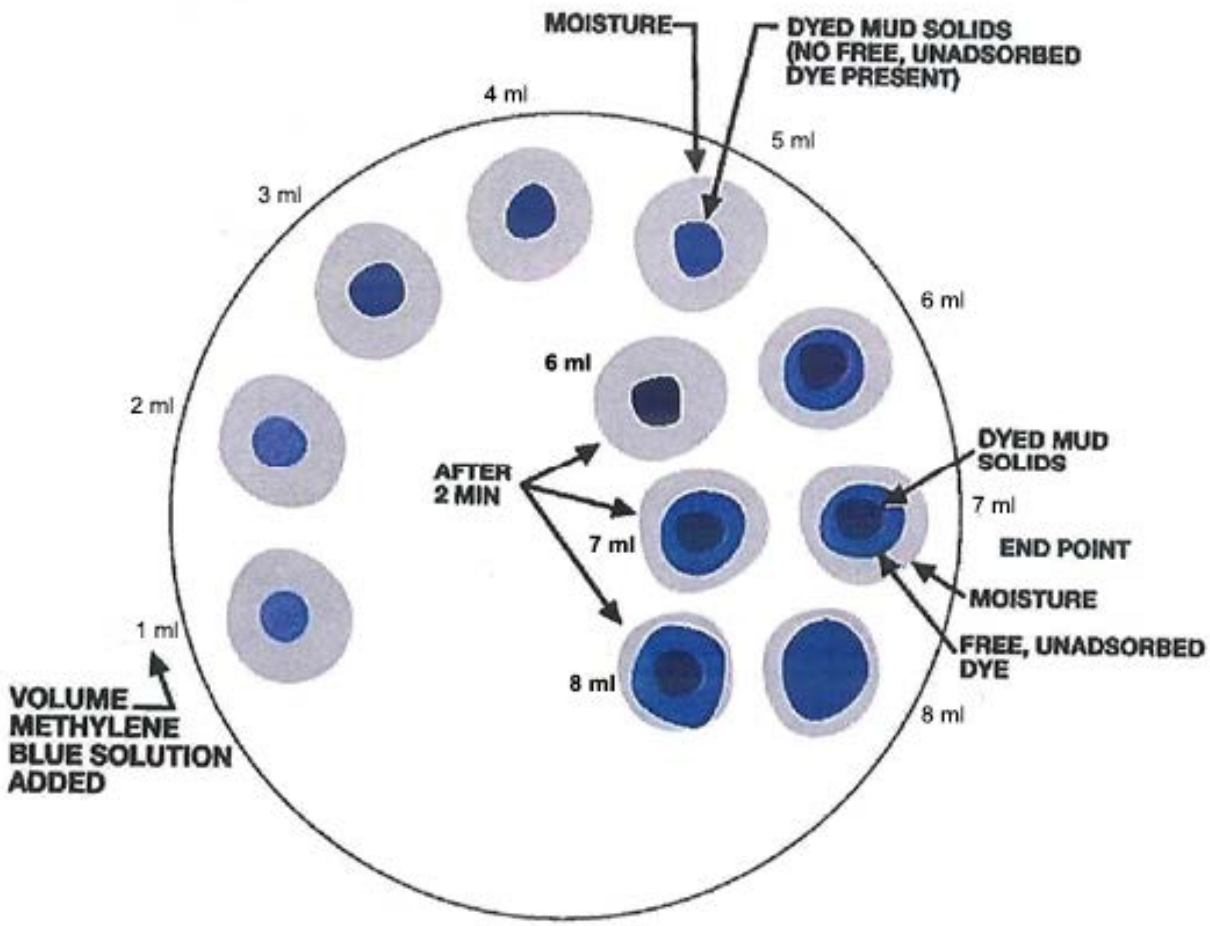

Figure 4-1 Spot Test for End Point of Methylene Blue Titration

4.1 Drilling Fluids Test Procedure

1. Pipette 10 ml of deionized water into the Erlenmeyer flask.
2. Use the syringe to accurately measure 2 ml of freshly agitated drilling fluid. Transfer this portion into the flask.

If less than 2 ml of methylene blue is required to reach endpoint, increase the volume of drilling fluid sample. If more than 10 ml of methylene blue solution is required, then decrease the sample volume

3. Add 15 ml of 3% hydrogen peroxide and 0.5 ml of 5N sulfuric acid to the flask.
4. Boil this mixture on the hotplate for 10 minutes. Do not boil to dryness.
5. Add deionized water to dilute this mixture to approximately 50 ml.
6. Add methylene blue in increments of 0.5 ml and swirl the flask after each addition for about 30 seconds.

If the approximate amount of methylene blue necessary to reach endpoint is known, then larger increments can be added at the beginning of the titration.

7. Dip a stirring rod into the flask while the solids are suspended, and then remove it. Place a drop of the liquid on the filter paper. The initial endpoint is reached when the dye appears as a blue ring surrounding the dyed solids (Figure 4-1).
8. When the blue ring spreading from the spot appears, shake the flask an additional 2 minutes. Place another drop on the filter paper. If the blue ring appears again, the final endpoint has been reached. If the blue ring does not appear, continue the titration until a drop taken after two minutes shows the blue tint.

9. Report methylene blue capacity of the drilling fluid as follows:

$$Methylene\ Blue\ Capacity = \frac{methylene\ blue, ml}{drilling\ \ fluid, ml}$$

Bentonite, lb/bbl Drilling Fluid = 5 x Methylene Blue Capacity

Bentonite, kg/m^3 Drilling Fluid = 2.85 x Methylene Blue Capacity

4.2 Binder in Foundry Sand Test Procedure

1. Pipette 50 ml of deionized water into the Erlenmeyer flask.
2. Obtain a representative sample of foundry sand.
3. Accurately weigh 5 g of this foundry sand.

Make sure that the scale accuracy is 0.01 g or better.

4. Place 5g sample in the Erlenmeyer flask with the water.
5. Shake or stir thoroughly for 10 to 15 minutes. As an option, boil the mixture for 5 minutes.
6. Add 1 ½ ml to 2 ml of 5N sulfuric acid and shake vigorously for 20 to 30 seconds.
7. Add methylene blue in 1 ml increments. After each addition shake the flask briefly. Using a stirring rod, place a drop of the fluid on the filter paper. Note the volume of methylene blue titrated. If the end point has not been reached, a purple circle will appear on the filter paper.

No color may appear on the filter paper after the first few milliliters are added because the methylene blue has been absorbed by the effective binder present. With experience, it may be possible at the beginning of the test to add several milliliters at a time until the endpoint is near. Use 1 ml increments near the endpoint.

8. Continue titrating until a blue-green halo appears around the purple circle on the filter paper. When the halo first appears, shake the solution for two minutes to make sure that all the methylene blue has been absorbed by the binder.

9. After two minutes, place another drop of liquid on the filter paper. If the halo is still present, the end point has been reached. If the halo disappears, add 1 ml methylene blue and repeat the filter paper test until the halo remains after two minutes of mixing.

10. Record the volume of methylene blue used to reach the endpoint. Read the percent binder in the sand from the graph in Figure 4-2.

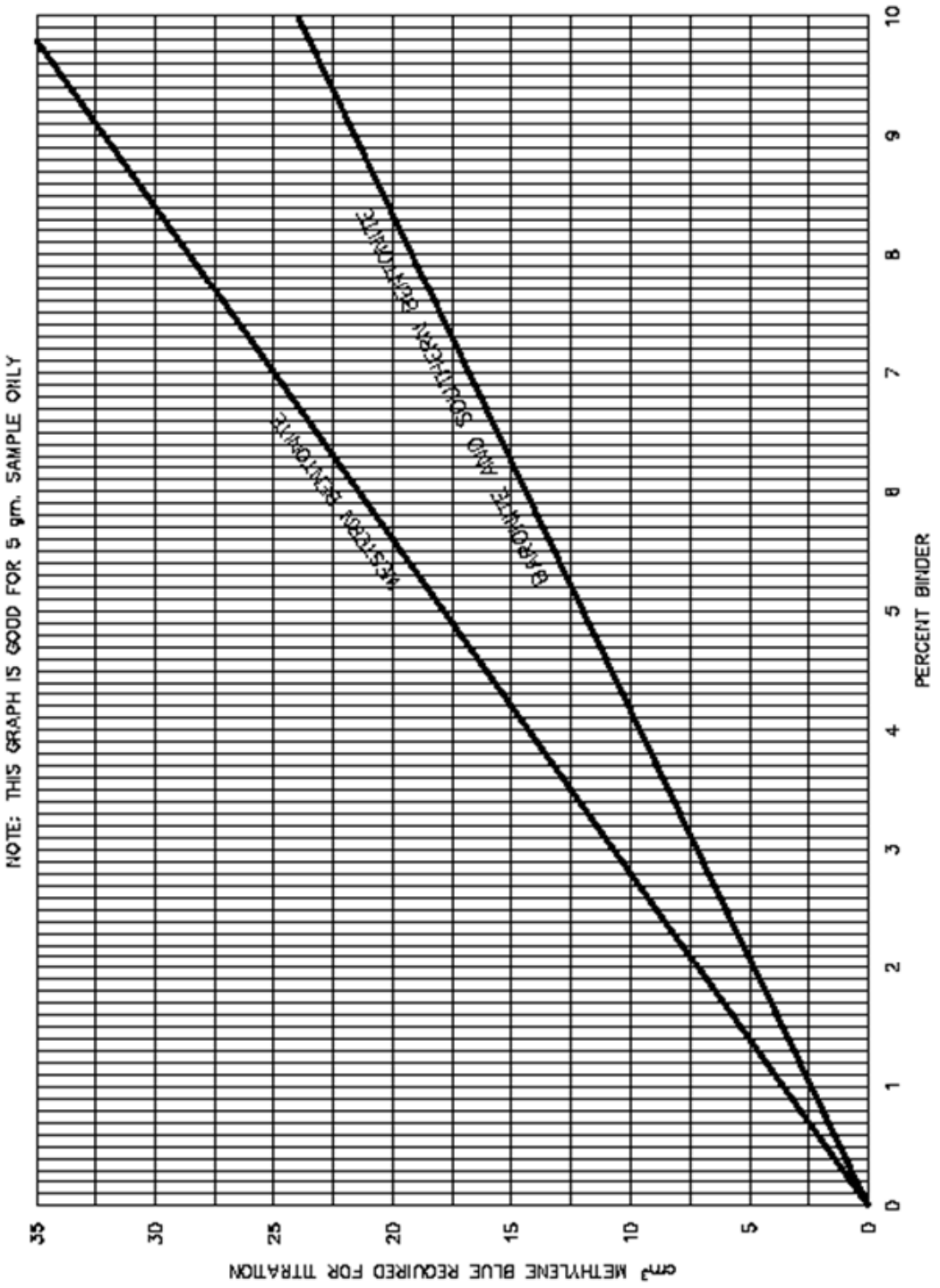

Figure 4-2 Percent Binder in Foundry Sand

5 Equipment Care

Methylene blue is a dye. If it dries on glassware or other laboratory equipment, methylene blue will leave a stain that is difficult or impossible to remove.

Avoid spilling methylene blue.

Immediately wash and thoroughly dry equipment and glassware after use.

Make sure that methylene blue bottles are capped tightly after testing ends.

Make sure that the hot plate is rated for the proper voltage before applying power.

6 Parts List

The Methylene Blue Kit is available with a 115V or 23V hotplate:

- Part No. 209679, Methylene Blue Kit, 115V
- Part No. 209694, Methylene Blue Kit, 230V

The part numbers for the items in the kit are listed in the following table.

Table 6-1 Methylene Blue Kit Parts List

Part No.	Description
205847	BOTTLE, 2 OZ, PLASTIC
210071	BULB, RUBBER
205867	CYLINDER, GRADUATED, GLASS, 50 ML, TD
206050	FILTER PAPER, 5-IN. DIAMETER (12.5 CM), 100/BOX
205914	FLASK, ERLENMEYER, 250 ML
205741	HOT PLATE, 115V, 325 WATT W/THERMOSTAT
205742	HOT PLATE, 230V, 325 WATT W/THERMOSTAT
209487	HYDROGEN PEROXIDE, 3%, 8 OZ
209687	METHYLENE BLUE, 3.2 g/L, 1ML = 0.01 meq, 8 OZ
205799	NEEDLE, LEUR-LOK SYRINGE
206026	PIPETTE, 1 ML
206029	PIPETTE, 10 ML
210070	PIPETTE DROPPER
206730	PUMP PIPETTE FILLER/DISPENSER
206030	STIRRING ROD, GLASS
205290	STOPPER, RUBBER, #6
205895	SYRINGE, LEUR-LOCK, GLASS, 5 ML
209944	WATER, DISTILLED, 8 OZ

Table 6-2 Required Reagent

Part No.	Description
209874	SULFURIC ACID, 5N, 8 OZ

Table 6-3 Optional Reagents

Part No.	Description
209688	METHYLENE BLUE, 3.2 g/L, 1ML = 0.01 meq, 16 OZ
209689	METHYLENB BLUE, 3.2 g/L, 1ML = 0.01 meq, 32 OZ
209690	METHYLENE BLUE, 3.2 g/L, 1ML = 0.01 meq, 1 GAL
209686	METHYLENE BLUE, 3.2 g/L, 1ML = 0.01 meq, 5 GAL
209692	METHYLENE BLUE, 4.5 g/L, 3.2 g/L, 1ML = 0.01 meq, 16 OZ
209693	METHYLENE BLUE, 4.5 g/L, 3.2 g/L, 1ML = 0.01 meq, 1 GAL

7 Warranty and Returns

7.1 Warranty

Fann Instrument Company warrants its products to be free from defects in material and workmanship for a period of 12 months from the time of shipment. If repair or adjustment is necessary, and has not been the result of abuse or misuse within the twelve-month period, please return, freight prepaid, and correction of the defect will be made without charge.

Out of warranty products will be repaired for a nominal charge.

Please refer to the accompanying warranty statement enclosed with the product.

7.2 Returns

For your protection, items being returned must be carefully packed to prevent damage in shipment and insured against possible damage or loss. Fann will not be responsible for damage resulting from careless or insufficient packing.

Before returning items for any reason, authorization must be obtained from Fann Instrument Company. When applying for authorization, please include information regarding the reason the items are to be returned.

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