



## **Methylene Blue Test Kit**

168-00 (115 Volt) 168-00-1 (230 Volt)

## **Instruction Manual**

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#### Intro

The Methylene Blue capacity test of a drilling fluid provides an indication of the amount of reactive clays (Montmorillonite) present as determined by the Methylene Blue test (MBT). The Methylene Blue capacity gives an estimate of the total cation exchange capacity (CEC) of the solids in the drilling fluid. Shale cuttings may be characterized and estimations may be made regarding the effects on hole stability and various mud making properties. The test measures the amount of Methylene Blue dye that is adsorbed by the clays suspended in the solution, which in turn provides an indication of the clays base exchange capacity.

The test procedure begins by diluting a sample of the drilling fluid. Hydrogen Peroxide is then added to the sample to remove organic materials, such as polymers and thinning agents which will react with the Methylene Blue dye and give erroneous results. The solution is then heated and Methylene Blue dye is added incrementally to the test solution. After each addition of the dye, a drop of the suspension is added to a circle of filter paper. The addition of the dye, followed by placing a drop of the solution on the filter media, is repeated until a blue ring or halo is seen surrounding the dyed solids on the paper. This represents the saturation point of the solids in the fluid. The Methylene Blue capacity is defined as the number of mL of Methylene Blue solution added per mL of the test solution. Bentonite content may be obtained in pounds per barrel by multiplying the Methylene Blue capacity results by 5 (kg/m<sup>3</sup> multiply by 14.25).

## Components

- #134-36-1 Red Knob for Bleeder Valve
- #140-56 Filter Paper, Whatman Grade 1, 12.5 cm Package of 100
- #144-36 Case
- #153-14 Graduated Cylinder, 50 mL × 1 mL, Glass
- #153-29 Syringe, 2cc, Glass-Tip
- #153-34 Pipet, 1 mL × 1/100 mL, Glass
- #153-40 Pipet, 10 mL × 1/10 mL, Glass
- #153-41 Pipet Aid, Safety Bulb
- #153-50 Erlenmeyer Flask, 250 mL
- #163-27 Medium Clip
- #163-28 Large Clip, Qty: 2
- #168-03 Hot Plate, 120 Volt
- #168-03-1 Hot Plate, 230 Volt
- #168-04 Stirring Rod, Glass, 6", Qty: 2
- #200-03 Methylene Blue Solution, 1 mL 0.01 ME, 16 oz., 500 mL
- #200-11 Hydrogen Peroxide, 3%, 8 oz, 250 mL
- #206-01 Deionized Water, 8 oz, 250 mL
- #230-13 \*Sulfuric Acid, 5N, 8 oz, 250 mL, UN #2796

168-00-1-SP	Spare Part Kit	
Part No.	Description	Qty
140-56	Filter Paper, 12.5 cm, Whatman Grade 1, 100/box	5
153-14	Graduated Cylinder, Glass, 50 mL × 1 mL	2
153-29	Syringe, Glass Tip, 2 cc	2
153-40	Pipet, Glass, 10 mL × <b>¼</b> mL	2
153-41	Pipette Aid	1
153-50	Erlenmeyer Flask, Glass, 250 mL	2
168-04	Stirring Rod, Glass, 6 inch	2
200-03	Methylene Blue Solution, 1 mL = 0.01 ME, 16 oz (500 mL)	6
200-09-1	Hydrogen Peroxide, 3%, 1 Gallon	1
206-01	Deionized Water, 8 oz (250 mL)	6
230-13	*Sulfuric Acid, 5N, 8 oz (250 mL) UN 2796	1
*Hazardous S	Shipping Item	

## Safety

#### **Hot Plate**

- 1. Make sure the hot plate is rated for the proper voltage before plugging it in.
- 2. Make sure the electrical cord is in good condition and equipped with a grounding plug.
- 3. Do not leave the hot plate unattended while heating.
- 4. Use caution while handling hot flasks and other laboratory containers.

#### Chemicals

The chemicals used in this kit (Methylene Blue solution, Hydrogen Peroxide 3%, and Sulfuric Acid 5N), are hazardous to your health. Avoid direct contact, inhalation, and ingestion. Keep away from fire and other heat sources.

Read all warnings, precautions, and hazard classifications (flammability, health, and reactivity) on the container label.

For in depth information on handling, reactivity with other substances, storage, and other safety related information, refer to the "Material Safety Data Sheet" (MSDS) for each chemical. If personal contact or an environmental accident occur, use the counteractive measures outlined on the MSDS.

As preventive measures:

- 1. Never pipette any chemical by mouth. Always use a pipette aid (#153-42 or equivalent) to load chemicals into a pipette.
- 2. Avoid contact with skin. Wear impervious, protective clothing, including boots, apron, gloves, lab coat or coveralls, as appropriate, to prevent skin contact.
- 3. Do not inhale vapors or take internally.
- 4. Use chemical safety goggles and/or full face shield where splashing is possible. Maintain an eye wash fountain and quick drench facilities in the work area.
- 5. Always pour acid into water and not water into acid.
- 6. Have feet completely covered. No sandals in the laboratory.
- 7. Always wear a lab coat when in the laboratory.
- 8. Have and keep adequately maintained an Eye Wash Station. Be sure everybody knows how to use it.
- 9. Get a good night of sleep before running any laboratory tests.
- 10. At the end of the day, unplug all unnecessary power cables.

#### Procedure



1. With the syringe, add 2.0 mL of drilling fluid to the Erlenmeyer flask. Air or gas entrained in the drilling fluid must be removed prior to injection.

Vigorously stir the drilling fluid to break the gel and quickly draw the mud into the syringe. Then slowly discharge the syringe back into the drilling fluid keeping the tip submerged. Again draw the fluid into the syringe and deliver exactly 2.0 mL of fluid to the flask.

If less than 2.0 mL or more than 10.0 mL of methylene blue solution will be required, the volume of drilling fluid sample may be increased or decreased to a more convenient size.

- 2. Add 10 mL of deionized water to the Erlenmeyer flask.
- 3. Add 15 mL of 3% Hydrogen Peroxide to the flask.
- 4. Add 0.5 mL of 5N Sulfuric Acid to the mixture.
- 5. Using the hot plate, boil the sample gently for 10 minutes. Do not allow it to boil to dryness.
- 6. Turn off the hot plate.
- 7. Dilute the mixture to about 50 mL using deionized water.
- 8. Add Methylene Blue solution to the flask in increments of 0.5 mL.

If the approximate amount of Methylene Blue dye is known from previous testing, then larger increments of the Methylene Blue dye may be used at the beginning of the procedure.

- 9. After each addition of Methylene Blue solution, swirl the contents of the flask by hand for about 30 seconds.
- 10. While the solids are still suspended, insert the glass stirring rod into the solution.
- 11. Place the tip of the stirring rod on the filter paper, which will deposit a drop of solids and dye.

12. Observe the drop on the filter paper.

- a. If you see a blue/turquoise ring around the dyed solids, swirl the flask for an additional 2 minutes and place another drop on the filter paper. If you see the blue/turquoise ring again, you have reached the final endpoint. If you do not see the blue/turquoise ring the second time, return to step 8.
- b. If you do not see the blue/turquoise ring, return to step 8.



### Calculation

Methylene Blue Capacity = <u>Methylene Blue (mL)</u> Drilling Fluid (mL)

The Methylene Blue capacity may also be reported as pounds per barrel of equivalent Bentonite, based on bentonite with a cation exchange capacity of 70 meq / 100 grams.

Bentonite equivalent (lb/bbl) =  $\frac{5 \text{ (Methylene Blue mL)}}{\text{Drilling Fluid (mL)}}$ 

Bentonite equivalent (kg/m<sup>3</sup>) = 2.85 × Bentonite equivalent (lb/bbl)

## Maintenance

Methylene Blue is a dye, and if allowed to dry on glassware or other laboratory equipment, will cause a stain that is difficult or impossible to remove.

- 1. Avoid spilling Methylene Blue.
- 2. Thoroughly wash and dry all laboratory equipment and glassware immediately after use.
- 3. Make sure methylene blue bottles are closed tightly after use.

## Appendix

Spot Tests for End Point of Methylene Blue Titration



\*Free dye detected immediately after adding the 6<sup>th</sup> mL is adsorbed after 2 minutes and indicates that the final end point has not yet been reached.

# Warranty and Return Policy

#### Warranty:

OFI Testing Equipment, Inc. (OFITE) warrants that the products shall be free from liens and defects in title, and shall conform in all respects to the terms of the sales order and the specifications applicable to the products. All products shall be furnished subject to OFITE's standard manufacturing variations and practices. Unless the warranty period is otherwise extended in writing, the following warranty shall apply: if, at any time prior to twelve (12) months from the date of invoice, the products, or any part thereof, do not conform to these warranties or to the specifications applicable thereto, and OFITE is so notified in writing upon discovery, OFITE shall promptly repair or replace the defective products. Notwithstanding the foregoing, OFITE's warranty obligations shall not extend to any use by the buyer of the products in conditions more severe than OFITE's recommendations, nor to any defects which were visually observable by the buyer but which are not promptly brought to OFITE's attention.

In the event that the buyer has purchased installation and commissioning services on applicable products, the above warranty shall extend for an additional period of twelve (12) months from the date of the original warranty expiration for such products.

In the event that OFITE is requested to provide customized research and development for the buyer, OFITE shall use its best efforts but makes no guarantees to the buyer that any products will be provided.

OFITE makes no other warranties or guarantees to the buyer, either express or implied, and the warranties provided in this clause shall be exclusive of any other warranties including ANY IMPLIED OR STATUTORY WARRANTIES OF FITNESS FOR PURPOSE, MERCHANTABILITY, AND OTHER STATUTORY REMEDIES WHICH ARE WAIVED.

This limited warranty does not cover any losses or damages that occur as a result of:

- Improper installation or maintenance of the products
- Misuse
- Neglect
- Adjustment by non-authorized sources
- Improper environment
- Excessive or inadequate heating or air conditioning or electrical power failures, surges, or other irregularities
- Equipment, products, or material not manufactured by OFITE
- Firmware or hardware that have been modified or altered by a third party
- Consumable parts (bearings, accessories, etc.)

#### **Returns and Repairs:**

Items being returned must be carefully packaged to prevent damage in shipment and insured against possible damage or loss. OFITE will not be responsible for equipment damaged due to insufficient packaging.

Any non-defective items returned to OFITE within ninety (90) days of invoice are subject to a 15% restocking fee. Items returned must be received by OFITE in original condition for it to be accepted. Reagents and special order items will not be accepted for return or refund.

OFITE employs experienced personnel to service and repair equipment manufactured by us, as well as other companies. To help expedite the repair process, please include a repair form with all equipment sent to OFITE for repair. Be sure to include your name, company name, phone number, email address, detailed description of work to be done, purchase order number, and a shipping address for returning the equipment. All repairs performed as "repair as needed" are subject to the ninety (90) day limited warranty. All "Certified Repairs" are subject to the twelve (12) month limited warranty.

Returns and potential warranty repairs require a Return Material Authorization (RMA) number. An RMA form is available from your sales or service representative.

Please ship all equipment (with the RMA number for returns or warranty repairs) to the following address:

OFI Testing Equipment, Inc. Attn: Repair Department 11302 Steeplecrest Dr. Houston, TX 77065 USA

OFITE also offers competitive service contracts for repairing and/or maintaining your lab equipment, including equipment from other manufacturers. For more information about our technical support and repair services, please contact <u>techservice@ofite.com</u>.