

## 10-mL Retort

#165-00-1: 10-mL Retort with Thermostat (115V) #165-10-1: 10-mL Retort with Thermostat (230V)

## **Instruction Manual**

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#### **OFI Testing Equipment, Inc.**

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### Intro

The retort provides a means for separating and measuring the volumes of water, oil, and solids contained in a sample of drilling fluid. A known volume of sample is heated to vaporize the liquid components which are then condensed and collected in a graduated receiver tube. Liquid volumes are determined from reading the oil and water phases on the graduated receiver tube. The total volume of solids, both suspended and dissolved, is obtained by noting the difference of the total sample volume versus the final liquid volume collected. Calculations are necessary to determine the volume of suspended solids since any dissolved solids will be retained in the retort. Relative volumes of low-gravity solids and weight materials may also be calculated.

## **Specifications**

Size:  $7.5" \times 6.5" \times 11.5" (19 \times 17 \times 29 \text{ cm})$ 

Weight: 12 lb 5 Oz. (5.6 kg)

Maximum Temperature: 1000°F (537°C)

Temperature Control: Standard - Electronic Fixed Temperature Control

Cell Capacity: 10 mL

Heating Capacity: 350 Watts

Power Requirement: 115 Volt at 3 Amp or 230 Volt at 1.5 Amp 50/60 Hz

## Equipment

Sample Cup: 10 mL capacity

**Condenser:** Cools the water and oil vapors below their vaporization temperature prior to leaving the condenser chamber. 464 grams.

**Heating Element:** Raises the temperature of the sample above its vaporization point within API Specifications, without causing the solids to boil over. 350 watts.

**Thermostat:** Limits the temperature of the retort to  $930^{\circ} \pm 70^{\circ}$ F.

**Liquid Receiver:** Graduated cylinder or tube, transparent and inert to oil, water, or salt solutions and temperatures of up to 90°F (32°C).

Fine Steel Wool: No. 000 Steel Wool. Do not use liquid steel wool or coated steel wool substitutes.

**Grease:** Never-Seez®. Used for a thread seal and lubricant at high temperatures.

**T-handle Drill:** Used for cleaning the retort chamber and condenser passage.

**Pipe Cleaner:** Used for cleaning the retort chamber and condenser passage.

**Spatula:** Shaped to fit the inside dimensions of the sample cup.



## Components

included:	
#152-37	AC Power Cord (115 Volt)
#152-38	AC Power Cord (230 Volt)
#153-03	Graduate Brush, ½" × 8"
#153-18	Graduated Cylinder, 10 mL × .1 mL
#165-14-13	T-handle Drill
#165-31	Retort Chamber, 10 mL
#165-32	Condenser, 10 mL
#165-34	Spatula
#165-41	Corkscrew
#165-42	Steel Wool, Grade 000. Extra Fine, Package of 4 Pads
#165-43	Pipe Cleaner
#165-44-2	Anti Seize Compound, Silver, 7g Pouch
#165-46	Clip for Condenser
#280-00	Wetting Agent, 1 oz.

#### **Replacement Parts:**

#122-074-1	Euro 5 Amn 5 mm x 20 mm (115 \/olt)
	Fuse, 5 Amp, 5 mm × 20 mm (115 Volt)
#122-073-1	Fuse, 3 Amp, 5 mm × 20 mm (230 Volt)
#130-79-53-0990	Thermostat (115 Volt)
#130-79-54-0990	Thermostat (230 Volt)
#164-10	Insulator Block, Top
#164-11	Insulator Block, Bottom
#164-12	Insulator Block, Cover
#164-19-1	Retort Adapter
#165-00-10	Thermocouple, Type J
#165-14-005	Insulation Material
#165-35	Heating Element (115 Volt)
#165-36	Heating Element (230 Volt)
#165-45	Neon Lamp, Red

#### Optional:

#165-05	Receiver Tube, 10 mL, 0 - 10 mL × .1 mL, 0 - 100% × 1%, with
	Certificate

#165-00-1-SP Spare Parts Kit, 115 Volt

Part Number	Description	Quantity
#122-074-1	Fuse, 5 Amp, 5 mm × 20 mm	5
#153-03	Brush, Graduate ½" × 8"	1
#153-18	Graduated Cylinder, 10 mL × 1/10 mL	1
#165-05	Receiver Tube, 10 mL, 0 - 10 mL × .1 mL, 0 - 100% × 1%, with Certificate	2
#165-33	Lid for 10-mL Sample Cup	1
#165-34	Spatula	1
#165-41	Corkscrew	1
#165-42	Steel Wool, Grade 000. Extra Fine, Package of 4 Pads	16
#165-43	Pipe Cleaner	6
#165-44-2	Anti Seize Compound, Silver, 7g Pouch	2
#280-00	Wetting Agent	1

#165-10-1-SP Spare Parts Kit, 230 Volt

Part Number	Description	Quantity
#122-073-1	Fuse, 3 Amp, 5 mm × 20 mm	5
#153-03	Brush, Graduate ½" × 8"	1
#153-18	Graduated Cylinder, 10 mL × 1/10 mL	1
#165-05	Receiver Tube, 10 mL, 0 - 10 mL × .1 mL, 0 - 100% × 1%, with Certificate	2
#165-33	Lid for 10-mL Sample Cup	1
#165-34	Spatula	1
#165-41	Corkscrew	1
#165-42	Steel Wool, Grade 000. Extra Fine, Package of 4 Pads	16
#165-43	Pipe Cleaner	6
#165-44-2	Anti Seize Compound, Silver, 7g Pouch	2
#280-00	Wetting Agent	1



Spare parts listings are intended to be used as a guide only as individual usage will vary. Running a retort test twice a day will require more parts than running a test once a week.

## Safety

The following guidelines should be followed to ensure safe operation of retorts.

It is imperative that the laboratory technician be thoroughly familiar with the proper operation and potential hazards of operating a retort. Improper or negligent operation can result in severe burns and other injuries.

- 1. The retort case will get very hot during the test and can cause severe burns.
- 2. The retort chamber and tube will get very hot and can cause severe burns.
- 3. Clean and dry the retort chamber and condenser, especially the inside of the mud sample cup, lid, and the condenser passage (spout). Clean the sample cup threads with a wire brush. The spatula, corkscrew tip, or a knife may be used to dislodge solids inside the sample cup.

Make sure the spout and the hole in the lid of the mud sample chamber are absolutely free of obstructions.

- 4. The assembly should be cooled to below 100°F (37.8°C).
- 5. Visually inspect the threads on the retort for any sign of damage before use.
- 6. Change out the steel wool after every test to prevent solids from building up.
- 7. Retorts used offshore should be changed out every 6 months for examination and cleaning.
- 8. Make sure the electrical source is fused and grounded. Ensure the power cord is in good condition and the connector has a proper ground connection.
- 9. Pipe cleaners should be used to scrape out any residue out of the spout leading to the condenser. An optional T-handle drill (#165-14-13) may be used to scrape any carbon deposits that may build up inside the tube. If the retort is used daily, the T-handle drill should be used at least once a week.



10. DANGER: The 10 mL and 20 mL Retorts are not designed for cuttings analysis. Cuttings can clog these devices and create an explosive hazard of super-heated gasses. For cuttings analysis, use the 50 mL Retort.





### **Quick Start**

- 1. Connect the retort to an electrical outlet and let it start heating up.
- 2. Pour the test sample through a Marsh Funnel screen to remove large particles.
- 3. Mix the sample thoroughly, ending up with a slow speed for 2 3 minutes to remove any trapped air.
- 4. Record the sample temperature and ensure it is within 10 degrees of the sample when density was measured.
- 5. Use the T-handle drill to clear the spout of any obstructions.
- 6. Pack steel wool into the expansion chamber.
- 7. Slowly fill the retort mud cup to the top.
- 8. Place the lid on the mud cup. Some of the liquid should extrude from the hole in the lid.
- 9. Wipe away any excess liquid from the cup and the threads.
- 10. Lubricate the mud cup threads with thread lubricant.
- 11. Hand tighten the mud cup onto the expansion chamber.
- 12. Attach the expansion chamber tube to the condenser.
- 13. Carefully place the retort chamber inside the heater block taking care to not get burned.
- 14. Place a drop of wetting agent inside the glass receiver tube.
- 15. Attach the glass receiver tube to the clip on the condenser and let it hang off the side of the table.
- 16. Observe the liquid exiting the condenser and continue heating for ten minutes after the flow stops.
- 17. Remove the glass receiver and allow it to cool. Record the amount of water, oil, and solids.
- 18. Unplug the retort.
- 19. Remove the retort chamber and condenser and allow them to air cool.
- 20. Using the corkscrew, remove the steel wool from the expansion chamber as soon as it is cool enough to handle.
- 21. Use the T-handle drill to clear the spout of any obstructions.

## Operation



- Collect a representative sample of drilling fluid and pour it through a marsh funnel screen to remove any lost circulation material, large cuttings, or debris.
- 2. Mix the drilling fluid sample thoroughly to ensure it is homogeneous. Ensure that no air is injected into the sample.
- 3. If the sample contains gas or air bubbles, add 2 to 3 drops of a defoaming agent for every 300 mL of sample fluid. Stir slowly for 2 to 3 minutes to release any entrained gasses.

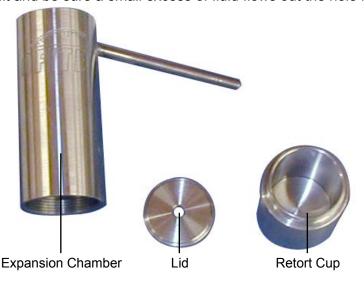
Air or gas entrapment in the sample will result in erroneously high solids content.

Do not test fluids that may contain formates. The formates will crystalize and block the spout.

- 4. Record the sample temperature. It should be within 10°F (5.5°C) of the temperature at which mud density was determined.
- 5. Use the T-handle drill to clear the spout of any obstructions.



- 6. Pack steel wool into the expansion chamber to approximately \(^{3}\)/<sub>6</sub> inches (4.76 mm) above the threads. As determined from experience, use only enough steel wool to prevent a boiling over of solids into the liquid receiver.
- 7. Using a clean syringe, fill the retort cup slowly with the non-aerated sample in order to avoid air entrapment. Lightly tap the side of the cup to expel any air and place the lid onto the cup. Rotate the lid to obtain a proper fit and be sure a small excess of fluid flows out the hole in the lid.



- 8. Lubricate the threads on the sample cup with a light coat of thread lubricant. This will prevent vapor loss through the threads and will also facilitate disassembly of the equipment at the end of the test.
- 9. Carefully hand tighten the retort cup onto the expansion chamber.



- 10. Apply thread lubricant to the threads on the expansion chamber arm and carefully connect the assembly to the condenser. Be careful not to overtighten and strip out the threads in the condenser. Place the chamber into the heating jacket and close the insulating lid.
- 11. Place a clean, dry liquid receiver under the condenser discharge tube. The length of this receiver may require that it be angled out from the retort or supported off the edge of the worktable. If the condenser clip is supporting the receiver, be careful not to break the lip of the glass receiver on the condenser.

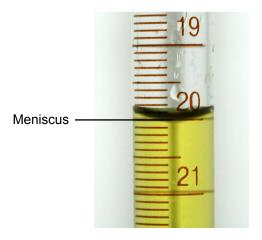
12. Connect the retort to an electrical outlet to turn it on. Observe the liquid exiting the condenser. Continue heating for ten minutes beyond the time that no more condensate is being collected.

If whole mud boils over into the receiver tube, the test must be rerun. Pack the retort body with a larger amount of steel wool and rerun the test. Allow it to run a minimum of 45 minutes. The light on the front of the retort will turn on while the heaters are engaged.

- 13. Remove the liquid receiver and allow it to cool. After it has cooled to ambient temperature, read and record the volumes (or volume percentage) of the following:
  - a. total liquid volume
  - b. oil volume
  - c. water volume

A drop of Wetting Agent will improve the meniscus for easier reading.

Always read volumes from the lowest point on the meniscus curve inside the receiver.



If an emulsion interface is present between the oil and water phases, heating the interface may break the emulsion. One way to do this is to remove the retort assembly from the heating jacket by grasping the condenser. Carefully heat the receiver along the emulsion band by gently touching the receiver for short intervals with the hot retort chamber. Avoid boiling the liquid. After the emulsion interface is broken, allow the receiver to cool and read the water volume at the lowest point of the meniscus.







- 14. Turn off the retort and allow it to cool prior to cleaning. Do not use cold water to try to rapidly cool down the chamber.
- 15. Use the T-handle drill to clear the spout of any obstructions.





The OFITE Retort will recover nearly 100% of refined oil that is being tested. However, if the drilling fluid is made up of crude oil, calibration runs may not be accurate, as the known percentage of crude oil in solution will need to be predetermined. Recovery of some crude oil may be as low as 60% due to impurities in the fluid, so crude oil is a poor indicator of the accuracy of the instrument.

## **Calculations**

The measured volumes (mL) of oil and water are converted into volume percents based on the volume of whole mud in the retort cup.

Volume Percent (%) Oil = 
$$V_0 = \frac{100 \text{ (Oil Volume Collected, mL)}}{\text{Sample Volume, mL}}$$

Volume Percent (%) Water = 
$$V_w = \frac{100 \text{ (Water Volume Collected, mL)}}{\text{Sample Volume, mL}}$$

Volume Percent (%) Solids = 
$$V_S = 100 - (V_O + V_W)$$



The volume percent solids include both suspended solids (weight material, etc.) and dissolved materials (for example salts). This volume percent will represent total suspended solids only if the mud is an untreated, freshwater mud.

To find the volume percent (%) of suspended solids and relate them to the relative volumes of low-gravity solids and weighting materials, an accurate mud weight and Chloride concentration must be known.

$$V_{SS} = V_S - V_W \frac{\text{Chloride Concentration, mg/L}}{1,680,000 - 1.21 (C_S)}$$

 $V_{SS}$  = Volume Percent (%) Suspended Solids  $C_S$  = Chloride Concentration, mg/L

Volume percent (%) Low-Gravity solids,  $V_{\rm lg}$ , are calculated as follows:

$$V_{lg} = \frac{1}{P_b - P_{lg}} [100 P_f + (P_b - P_f) V_{SS} - 12 W_m - (P_f - P_O) V_O]$$

V<sub>Ig</sub> = Volume percent (%) low-gravity solids.

W<sub>m</sub> = Mud Weight, pounds per gallon

P<sub>f</sub> = Density of filtrate, grams per cubic meter

P<sub>b</sub> = Density of weighting material, grams per cubic meter

P<sub>lg</sub> = Density of low gravity solids, grams per cubic meter (use 2.6 if unknown)

P = Density of oil, grams per cubic meter (use 0.84 if unknown)

Volume percent (%) weighting material (V<sub>b</sub>) is calculated as follows:

$$V_b = V_{SS} - V_{Ig}$$

Concentrations of low gravity solids, weighting material and suspended solids may be calculated as:

$$C_{lg} = 3.49 (P_{lg}) \times (V_{lg})$$
  
 $C_{b} = 3.49 (P_{b}) \times (V_{b})$   
 $C_{SS} = C_{lg} + C_{b}$ 

Where:

 $C_{lg}$  = Low gravity concentration, pounds per barrel  $C_{b}$  = Weighting material concentration, pounds per barrel  $C_{SS}$  = Suspended solids concentration, pounds per barrel

### Maintenance

#### **Calibration of Retort Mud Cups:**

- 1. Record the temperature of the retort cup and lid to the nearest 0.5°C(1°F).
- 2. Tare an electronic balance to zero and place the clean and empty retort cup and lid on the balance. Record the weight.
- 3. Fill the retort cup with deionized water. Place the lid on top of the mud cup and ensure that a small excess of liquid comes out of the hole.
- 4. Wipe excess liquid from the mud cup and lid. Avoid wicking out water from the cup.
- 5. Place the filled retort and lid on the previously tared balance and record the mass of water to the nearest gram.
- Calculate the volume of the retort (V<sub>rc</sub>) using the density of water at ambient temperature.
   (See Density Temperature Chart on page 14)

$$V_{rc} = \frac{Mass \text{ of water, (g)}}{Water \text{ density, (g/cm}^3)}$$

#### **Calibration Check for Temperature:**

- 1. Remove retort chamber assembly from heating chamber.
- 2. Obtain a thermometer capable of 1200°F (516°C) (#154-25) and position the sensing probe inside the retort adapter block when the retort is turned fully on.
- 3. Let temperature stabalize. The temperature should read in the range of  $930^{\circ}\text{F} \pm 70^{\circ}\text{F}$  ( $498^{\circ}\text{C} \pm 21^{\circ}\text{C}$ ).

#### Cleaning:

Clean the expansion chamber tube thoroughly with the T-handle drill (#165-14-13) after every test. Over time the tube can completely fill with carbon deposits.



## **Appendix**Density-Temperature Chart

Density of Water as a Function of Temperature			
°C	°F	Density g/cm <sup>3</sup>	
15.0	59.0	0.9991	
15.5	59.9	0.9991	
16.0	60.8	0.9990	
16.5	61.7	0.9989	
17.0	62.6	0.9988	
17.5	63.5	0.9987	
18.0	64.4	0.9986	
18.5	65.3	0.9985	
19.0	66.2	0.9984	
19.5	67.1	0.9983	
20.0	68.0	0.9982	
20.5	68.9	0.9981	
21.0	69.8	0.9980	
21.5	70.7	0.9979	
22.0	71.6	0.9977	
22.5	72.5 73.4	0.9976 0.9975	
23.5	74.3	0.9973	
24.0	75.2	0.9974	
	76.1		
24.5		0.9971	
25.0	77.0	0.9970	
25.5	77.9	0.9969	
26.0	78.8	0.9968	
26.5	79.7	0.9966	
27.0	80.6	0.9965	
27.5	81.5	0.9964	
28.0	82.4	0.9962	
28.5	83.3	0.9961	
29.0	84.2	0.9959	
29.5	85.1	0.9958	
30.0	86.0	0.9956	
30.5	86.9	0.9955	
31.0	87.8	0.9953	
31.5	88.7	0.9952	
32.0	89.6	0.9950	
32.5	90.5	0.9949	
33.0	91.4	0.9947	
33.5	92.3	0.9945	
34.0	93.2	0.9944	
34.5	94.1	0.9942	
35.0	95.0	0.9940	

## 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 <a href="mailto:techservice@ofite.com">techservice@ofite.com</a>.