Potassium & Potassium Chloride Kit
Centrifuge Method: > 5000 Milligrams per Liter KCl (1% KCl)

Part No. 285-09

Instruction Manual
Updated 6/1/2017
Ver. 3.0

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Potassium ion is used in drilling fluids to aid in the stabilization of shales and to control swelling clays. The accurate determination of the potassium ion content is necessary to control the inhibition characteristics of the drilling fluid. This procedure is used to measure the potassium ion content in mud filtrates at levels above 5000 milligrams per liter or 3.5 pounds per barrel KCl. The procedure is most accurate in the range between 10,000 mg/L (2%) and 40,000 mg/L (8%) KCl and is suspect below 10,000 mg/L (2%) KCl. Potassium is precipitated in a centrifuge tube as the perchlorate salt, and then the precipitate volume is measured. The potassium ion content is then read from a prepared standard curve.

**Components**

#151-50   Carrying Case  
#151-52   Pluck Foam Set for Carrying Case  
#153-21   10-mL-Centrifuge Tube; Kolmer; Qty: 4  
#153-25-2 Hand-Crank Centrifuge for 15-mL Tubes; 4-Place Head and Shields  
#153-38   Glass Pipet; 5 mL × 1/10 mL; Qty: 2  
#206-01   Deionized Water; 8 oz.; Qty: 2  
#285-11   Potassium Chloride Solution; 4 oz.  
#285-13   *Sodium Perchlorate; (UN #3139); 8 oz.; Qty: 2
Procedure

Generating a Calibration Curve

1. A standard calibration curve is required for each type of centrifuge. Each curve should consist of at least three points (3.5, 10.5, and 17.5 pounds per barrel) to create an accurate graph.

2. Prepare the samples by using the standard potassium chloride solution where 0.5 mL is equivalent to 3.5 pounds per barrel KCl.

   If standard potassium chloride solution is not readily available, it can be made by mixing 14.0 g of KCl powder with 100 mL of deionized water.

   $3.5 \text{ ppb KCl} = 1\% \text{ KCl} = 5000 \text{ mg/L} = 0.5 \text{ mL standard KCl solution.}$

   Therefore:

   a. To obtain 3.5 ppb equivalent KCl or 1% KCl use 0.5 mL of standard potassium chloride.

   b. To obtain 10.5 ppb equivalent KCl or 3% KCl use 1.5 mL of standard potassium chloride.

   c. To obtain 17.5 ppb equivalent KCl or 5% KCl use 2.5 mL of standard potassium chloride

3. Dilute the samples in the centrifuge tube to the 7.0 mL mark with deionized water and agitate the mixture.

4. Add 3 mL of standard sodium perchlorate solution to each centrifuge tube and do not agitate.

5. Centrifuge at a constant speed of approximately 1,800 revolutions per minute for one minute and read the precipitate volume immediately. It will be necessary to counterbalance the centrifuge tube with another tube and 10 mL of liquid of the same weight.

   To obtain a fairly constant 1,800 RPM, determine how many revolutions the rotor makes with each cycle of the crank. Turn the crank very slowly and count the number of revolutions of the rotor head during one cycle of the crank. Divide 1,800 by the number of revolutions of the rotor head to determine the required number of cycles of the crank.

   For example, if the rotor head makes 15 revolutions in one cycle of the crank, then 120 cycles of the crank are required to achieve 1,800 revolutions of the rotor head ($1,800 / 15 = 120$). Therefore, the crank must be cycled 120 times per minute to achieve 1,800 RPM. At this rate, in 5 seconds, the crank must be cycled 10 times ($120 / 60 \times 5$). Record this interval for future reference.
6. Clean the centrifuge tube immediately after each use.

7. Plot the volume of precipitate in milliliters versus pounds per barrel of KCl or milliliters of precipitate versus percent (%) potassium chloride using rectangular graph paper. See the example below.
Procedure

Sample Testing

1. Measure the appropriate volume of filtrate into the centrifuge tube. See the table below for the volume:

<table>
<thead>
<tr>
<th>KCl Concentration Range (kg/m³)</th>
<th>K+ (mg/L)</th>
<th>Filtrate Volume to Use (mL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10 - 50</td>
<td>3.5 - 18</td>
<td>5,250 - 27,000</td>
</tr>
<tr>
<td>50 - 100</td>
<td>18 - 35</td>
<td>27,000 - 52,500</td>
</tr>
<tr>
<td>100 - 200</td>
<td>35 - 70</td>
<td>52,500 - 105,000</td>
</tr>
<tr>
<td>over 200</td>
<td>over 70</td>
<td>over 105,000</td>
</tr>
</tbody>
</table>

2. Dilute to 7.0 mL if necessary with deionized water in the tube and agitate the mixture.

3. Add 3.0 mL of standard sodium perchlorate solution and do not agitate. If potassium is present precipitation occurs at once.

4. Centrifuge at a constant speed of approximately 1,800 RPM for one minute. Read the precipitate volume immediately and then record it.

5. Add 2 to 3 drops of the sodium perchlorate solution to the tube. If a precipitate still forms, the total amount of potassium has not been measured. Refer to the table above and use the next smaller filtrate volume. Repeat the procedure in items 1 - 5 above.

6. Determine the potassium chloride concentration by comparing the precipitate volume measured with the standard calibration curve as prepared above. Report the potassium concentration as pounds per barrel KCl. The potassium concentration may also be reported as milligrams per liter potassium ion. If the filtrate potassium chloride concentration from the standard calibration curve exceeds an 18 pounds per barrel reading, accuracy of the results is reduced. For more accurate results, use the next smaller filtrate volume as noted in the table above, and repeat the steps 1 through 5.
Procedure

High Potassium Chloride Concentrations

If the potassium concentration of the filtrate is greater than 35,000 mg/L (7%) KCl, dilution may be accomplished as follows:

1. Pipette 2.0, 3.0 or 4.0 mL of filtrate into the centrifuge tube and dilute with deionized water to the 7.0 mL mark.

2. Correct the results by multiplying % KCl by 7, divided by the volume of filtrate used (2.0, 3.0 or 4.0 mL)

High Potassium Chloride Concentrations

\[ \text{KCl in Filtrate (kg/m}^3\text{)} = \frac{\text{Value from Standard Curve (mg/L) } \times 7}{\text{Filtrate Used (mL)}} \]

\[ \text{K}^+ \text{ in Filtrate (mg/L) } = 1,500 \text{ (KCl in filtrate, lb/bbl)} \]

Calculations

Remarks

1. The potassium ion test must be made with the same centrifuge used to construct the standard curve. Turn the centrifuge at a constant speed of 1,800 RPM.

2. Sodium and potassium perchlorates are explosive in the dry state if heated to a high temperature or allowed to contact organic reducing agents. The perchlorates are not hazardous if kept water wet. They will decompose harmlessly if dispersed in a bucket of water.

3. Salts, lignosulfonates, polymers, clay and pH do not interfere with the test.

4. The precipitate may become slightly discolored in high lignosulfonate concentrations, but the test is not adversely affected.

5. Only the Kolmer-type clinical centrifuge tubes are satisfactory for the test.

6. The calibration curve does not need to be remade for every potassium determination, however the validity of the curve should be rechecked regularly by re-running at least one standard.

7. A new standard graph must be prepared if a different model of centrifuge is used.
Warranty and Return Policy

Warranty:

OFITE 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.

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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

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