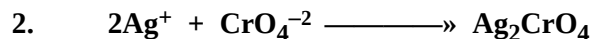
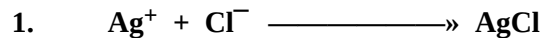


Water Based Fluids

This test measures the total soluble Chloride ion concentrations in the mud *Filtrate*. These Chlorides can come from Sodium Chloride, Calcium Chloride or Potassium Chloride. For the titration to work correctly, the pH of the filtrate needs to be slightly basic, around pH = 8.3, etc. Two chemical reactions take place simultaneously during the titration procedure.



The first reaction, the formation of Silver Chloride, accounts for the appearance of the white specs or milky appearance during the titration. The formation of Silver Chromate, which is red in color, will not start until all of the Chloride Ions are tied up as Silver Chloride. The Silver Nitrate will then react with the chromate from the Potassium Chromate Indicator to form Silver Chromate. So, for the above two reactions to occur, the filtrate needs to be weakly basic. (pH = 8.3). High pH will precipitate out Silver Oxide.

EQUIPMENT:

- #153-26 Titration Dish, polyethylene
- #153-28 Stirring Rod, polyethylene
- #153-34 Pipette, 1 ml x 1/100 ml, glass
- #153-40 Pipette, 10 ml x 1/10 ml, glass

Reagents:

- #206-01 Distilled Water, 8 oz
- #215-00 *Potassium Chromate Indicator sol'n, 2 oz (UN3082)
- #220-00 Phenolphthalein Indicator sol'n, 2 oz
- #230-08 Sulfuric Acid, 0.02N (N/50), 8 oz
- #265-02 Silver Nitrate sol'n, 0.001G, 0.0282N, 16 oz
- #265-06 Silver Nitrate sol'n, 0.01G, 0.282N, 8 oz
- #285-00 Calcium Carbonate, precipitated. 35 grams

Case:

- #144-35 Multi Kit Case, Diagonal design, Stainless Steel

PROCEDURE - Water Based Muds:

1. Pipette one or more milliliters (cm³) of filtrate into the titration dish.
2. Add 2 - 3 drops of Phenolphthalein Indicator sol'n to the filtrate.
If a pink color appears - Titrate with N/50 Sulfuric acid until the color is discharged.
If the filtrate is deeply colored - Add an additional 2 ml of N/50 Sulfuric Acid and stir.
 Then add 1 gram of Calcium Carbonate and stir.
3. Add 25 - 50 ml of distilled water. If any water other than distilled water is used, the chlorides in it must be accounted for by titration before adding the filtrate to it.
4. Add 5 - 10 drops of Potassium Chromate Indicator sol'n.
5. Add Silver Nitrate sol'n drop-by-drop from the pipette while stirring continuously with the stirring rod, until the color changes from yellow to orange-red and persists for 30 seconds. Record the number of milliliters (cm³) of Silver Nitrate sol'n required to reach the end point.
 - A. If the chloride ion concentration of the filtrate exceeds 10,000 milli grams (mg/l) per liter use the 0.1 gram chloride-ion per cubic centimeter (0.282N) strength solution.
 - B. If over 10 milliliters (cm³) of Silver Nitrate is used, repeat the test with a smaller amount of filtrate.

CALCULATION:

$$\text{Chloride, mg/L} = \frac{*1000 \times \text{mls of Silver Nitrate titrated}}{\text{Filtrate Sample, mls}}$$

To Convert Units:

$$\text{Chloride, ppm} = \frac{\text{Chloride, mg/L}}{\text{Specific Gravity of Filtrate}}$$

$$\text{Salt (NaCl), mg/L} = (1.65) \text{ Chloride, mg/L}$$

* Use 1000 as the multiplier for 0.0282N (.001gram) Chloride Ion solution, and 10,000 for the 0.282N (.01 gram) AgNO₃

REMARKS:

1. Avoid contact with Silver Nitrate and wash immediately with water if Silver Nitrate gets on skin or clothing.
2. The end point of the reaction is when the Silver Chromate is first formed and it is reddish in color. When using the weak, or 0.001 gram sol'n, the end point is approached very gradually. Therefore the formation of the Silver Chromate can be seen by a color change from yellow to orange-red. If the strong (.01gram) solution is used the end point is reached much more rapidly and may go from yellow to red. As soon as the red color is seen, the titration procedure is complete.
3. White lumps of Silver Chloride form when titrating high concentrations of salt. This should not be taken for the end point of the titration.

Oil Based Fluids - Whole Mud Chloride

The whole mud chloride test procedure for oil muds is a titration procedure which measures the volume of a standard Silver Nitrate required to react with the Chloride ions to form insoluble Silver Chloride salts. The Chloride value reported for the whole mud may be assigned to the water phase of the solution up to the saturation point. The water soluble salt concentration indicates how effective an oil mud is in controlling shale through the "aqueous phase activity" concept. The water phase salinity value is also needed to adjust the retort value to obtain a corrected solids content for the oil mud. The test procedure uses the same sample as used in the whole mud Alkalinity test provided the sample is acidic ($\text{pH} < 7$).

Equipment - Oil Based Fluids:

#153-29-1	Syringe, glass tip, 5 ml
#153-51-1	Beaker, 400 ml, glass
#153-53	Magnetic Stirrer, with Stir Bars
Reagents:	
#206-02	Distilled Water, 16 oz
#215-02	Potassium Chromate sol'n, 8 oz (UN3082)
#220-01	Phenolphthalein Indicator sol'n, 8 oz
#230-10	Sulfuric Acid sol'n, N/10, 8 oz
#265-08	Silver Nitrate sol'n, 0.01g, 0.282N, 16 oz
#280-30	Arcosolv PNP, gal.

Procedure - Oil Based Fluids

Perform the whole mud alkalinity test first as follows:

1. Add 100 ml of Arcosolv PNP solvent to the 400 ml beaker.
2. Fill the 5 ml syringe with at least 3 mls of whole mud and discharge 2 ml into the beaker. Swirl the mixture until it is homogenous.
3. Add 200 ml of distilled water.
4. Add 15 drops of Phenolphthalein Indicator solution.
5. While stirring with the magnetic stirrer, slowly titrate with N/10 Sulfuric Acid until the pink color just disappears. Continue stirring for one more minute, and if no pink color reappears, discontinue stirring. It may be necessary to stop the stirring to allow separation of the two phases in order to more clearly see the color in the aqueous phase.
6. Let the sample stand for 5 minutes and if no pink color reappears, the Alkalinity end point has been reached. If the pink color returns, titrate a second time with the Sulfuric Acid solution. If the pink color still returns titrate a third time, but if the color returns after this third titration, call this the end point.
7. After running the Alkalinity test make sure the mixture to be titrated for Chlorides is acidic (pH < 7) by adding 10 - 20 drops of N/10 Sulfuric Acid.
8. Add *3.0 mls of Potassium Chromate Indicator solution.
9. While stirring with a Magnetic Stirrer, slowly titrate with 0.282N Silver Nitrate solution until a salmon pink color remains stable for at least one minute. It may be necessary to stop the stirring of the mixture and allow separation of the two phases to occur in order to more clearly see the color in the aqueous phase.
10. Note the total milliliters of 0.282N Silver Nitrate required to reach the end point to calculate the whole mud Chloride.

Calculation:

$$\text{Chloride, Whole Mud (Cl}_{om}\text{) Mg/l} = \frac{10,000 (\text{ml Silver Nitrate titrated})}{\text{ml of sample}}$$

*Quantity has been revised since the last API issue - "R.P. 13B-2", Third Edition, Dated Feb. 1998.

For more information, please contact us:

ExpotechUSA
10700 Rockley Road
Houston, Texas 77099
USA

281-496-0900 [voice]

281-496-0400 [fax]

E-mail: sales@expotechusa.com

Website: www.ExpotechUSA.com