STANDARD METHOD FOR DETERMINATION OF GLYCOLS IN DRILLING FLUID FILTRATES

I. SUMMARY:

A colorimetric method is used to determine the glycol concentration in drilling fluid filtrates. The glycol is extracted into dichloromethane using a blue complexing agent. The resulting blue color of the dichloromethane is compared to standard solutions and the concentration determined.

II. EQUIPMENT AND MATERIALS:

A. Eppendorf Model 3190 Fixed – Volume Pipette, 100 ml
B. Pipette tips, 10 – 100 ml range
C. Test Solution Vials
D. Color Wheel for viewing samples and holding vials
E. Box of Kemwipes

III. PROCEDURE:

A. Obtain a mL or more of filtrate
B. Filling the pipette. (For concentrations between 5 and 10% by volume see "Note" below.)
    1. Attach pipette tip to pipette.
    2. Press the yellow control button (at the top of the pipette) down to the first stop.
    3. Hold the pipette vertically and immerse tip about 3 mm into the filtrate.
    4. Let the yellow control button rise slowly to fill the tip with liquid.
    5. Slide the tip out of the filtrate along the wall of the container.
    6. Wipe off any droplets on the outside of the tip with a Kemwipe.

C. Open a Test Solution Vial and dispense the filtrate.
    1. Hold the pipette tip at an angle against the inside of the vial, but not into the liquid.
    2. Press the yellow control button slowly down to the first stop and wait about 3 seconds.
    3. Press the button on down to the second stop to empty the tip completely.
    4. Hold the button down and slide the tip up along the wall of the vial and remove.
    5. Let the yellow control button glide back to its rest position.
    6. Eject the tip by pressing the tip ejector button.

D. Cap the vial and shake for 1 minute.

E. Allow vial to rest for 2 – 3 minutes while the phases separate.

F. Place vial in the top of the color-wheel viewer and best match its color to one of the standard solutions. Tilt the color-wheel forward and view the % (by Volume) glycol in the aqueous phase on the scale which is visible through the small hole on top the color-wheel. Record.

Note: For glycol concentrations between 5 and 10% by volume, the filtrate must be diluted 50/50 with DI water. Using the pipette techniques described in Part III B, proceed with the steps listed below and then return to Part III f. The % volume glycol estimated in Part G must be doubled to reflect a concentration between 5 and 10%.

1. Draw 3 pipette volumes of DI water and add to a clean, dry vial or other suitable container.
2. Draw 1 pipette volume of filtrate and discard.
3. Follow with 3 pipette volumes of filtrate added to the vial containing the 3 volumes of DI water.
4. Draw 1 volume of the 50/50 filtrate/DI water mixture and discard.
5. Draw 1 final volume of the 50/50 filtrate DI/water mixture and proceed to step III C.

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