PROCEDURE FOR DETERMINING IODIDE IN MUD FILTRATES

Equipment:

#153-36 Pipette, 2 ml x 1/10 ml, glass - 2 each
#153-38 Pipette, 5 ml x 1/10 ml, glass - 2 each
#153-40 Pipette, 10 ml x 1/10 ml, glass - 2 each
#153-50 Erlenmeyer Flask, 250 ml, glass - 2 each
#153-86 Rubber Stopper, no 6, for Flask - 2 each

Reagents:

#145-551 Starch Indicator sol'n, 2 oz
#230-13 Sulfuric Acid, 5N, 8 oz (UN2796)
order Urea sol'n, 8 oz
order Sodium Nitrite ($NaNO_2$), 100 g (0.05M sol'n - 0.86 g 0.05M $NaNO_2$ in 250 ml distilled water)
Must be prepared the same day as the analysis due to the possibility of oxidation to nitrate.
order Potassium Iodide (KI) sol'n, 10%, 16 oz
order Sodium Thiosulfate ($Na_2S_20_3$) sol'n, 0.0080 N, 16 oz

Optional:

#153-53-9 Magnetic Stirrer, 1500 rpm, 115 volt
#153-53-1 Stir Bar, 1" x 5/16"
#166-00 Balance, Portable, 100 x 0.01 g

Procedure:

1. With a 10 ml pipette transfer a 10 ml sample to a 250 ml Erlenmeyer Flask.
2. Using pipettes, add 5 ml of 0.05M Sodium Nitrite ($NaNO_2$) and 10 ml of the Urea solution. Dilute to approximately 100 ml with distilled water.
3. Repeat procedures 1 and 2 using 10 ml of distilled water instead of the sample. This is for the blank determination.
4. Add 5 ml of 5N Sulfuric Acid to both flasks and stopper immediately. Allow to stand with frequent swirling for 10 minutes. Do not shake and do not let the contents come into contact with the rubber stopper.

*5. Add 10 ml of 10% Potassium Iodide (KI) solution to the blank and observe a yellow color due to iodine. Titrate immediately with 0.008N Sodium Thiosulfate until *most* of the yellow color is discharged. Add about 1 ml of starch indicator solution and observe that a blue color is formed. Continue to titrate with the 0.008N Sod. Thiosulfate until the blue color just disappears. This is the end-point. Record the total number of milliliters of Sodium Thiosulfate used.

6. Add 10 ml of 10% Potassium Iodide to the sample and titrate as in step 5 to the end-point.

Calculation:

$$\textbf{Iodide, mg/L} = \frac{\textbf{1000 x (ml } Na_2S_20_3 \textbf{ from step 6 – ml } Na_2S_20_3 \textbf{ from step 5)}}{\textbf{ml of sample}}$$

* If the filtrate is colored or if a small concentration of iodide is being determined in the sample, the starch indicator may be added at the beginning of the titration.

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