Determination of Peracetic Acid (PAA) and Hydrogen Peroxide (H₂O₂) in Water

Concentration Range of 0.01 to 35% (Titration)

Equipment and Reagents:

Digital Titrator – Hach PN 5953062

Delivery Tubes for Digital Titrator – Hach PN 1720500

Flask, Erlenmeyer, 125-mL – Hach PN 50543

Sulfuric Acid Standard Solution, 5.25 N – Hach PN 244932

Ferroin Indicator Solution – Hach PN 181233

Ceric Standard Solution Titration Cartridge, 0.5 N – Hach PN 2270701

Sulfite 1 Reagent Powder Pillows, pk/100 – Hach PN 220399

0.2 N Sodium Thiosulfate Digital Titrator Cartridge – Hach PN 2267501

2.0 N Sodium Thiosulfate Digital Titrator Cartridge – Hach PN 1440101

Titration Procedure:

H2O2 Titration

Cool Deionized Water and Sulfuric Acid Solution to ~4°C before beginning test

- (1) Select the sample volume from *Table 1* which corresponds to the expected H_2O_2 and PAA concentration.
- (2) Insert a clean delivery tube into the Ceric Standard Solution Titration Cartridge and attach the cartridge to the titrator body. See *General Description, Step by Step* in the Digital Titrator manual for more information.
- (3) Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.
- (4) Pour approximately 75 mLs of cold DI water into the 125-mL Erlenmeyer flask.
- (5) Use a graduated cylinder or pipet to measure the sample volume from *Table 1*. Transfer the sample to the 125-mL Erlenmeyer flask.
- (6) Add 5 drops of the cold 5.25 N H2SO4 to the DI water. Add one drop of the Ferroin Indicator Solution to the DI water, swirl to mix.
- (7) Place the delivery tip into the solution. While titrating with Ceric Standard Solution, swirl the flask until the solution color changes from orange to a pale blue. Record the number of digits required and calculate: Digits Required x Digit Multiplier = $\% H_2O_2$. Do not discard titrated sample, this sample is used in the PAA titration below.

PAA Titration:

- (1) Replace the Ceric Standard Solution Titrant Cartridge with the 0.2 N Sodium Thiosulfate Cartridge or the 2.0 N Sodium Thiosulfate Cartridge. See *Table 1* to choose which cartridge to use based on the expected PAA concentration. If you don't want to change out the titrant cartridges each time during the titration, you can use two Digital Titrators to perform this titration.
- (2) Add the contents of one Sulfite 1 Reagent powder pillow to the 125-mL Erlenmeyer flask and swirl to mix. The solution will turn dark brownish purple.
- Place the delivery tip into the solution. While titrating with Sodium Thiosulfate Solution, swirl the flask until the solution color changes from brown to the original orange color from the H_2O_2 titration. Record the number of digits required and calculate: Digits Required x Digit Multiplier = % or mg/L PAA.



Table 1

Expected Sample Concentration for H ₂ O ₂	Titration Cartridge Ceric Standard Solution (N)	Sample Volume (mL)	Digit Multiplier
2 - 10%	0.5	0.2	0.0053
0.5 - 2%	0.5	0.5	0.00212
0.1 - 0.5%	0.5	1.0	0.00106
0.05 - 0.1%	0.5	2.0	0.00053
0.01 - 0.05%	0.5	5.0	0.000212

Expected Sample	Titration Cartridge Sodium	Sample Volume (mL)	Digit Multiplier
Concentration for PAA	Thiosulfate (N)		
5 - 35%	2.0	0.2	0.0475
2 - 10%	2.0	0.5	0.0190
1 - 5%	2.0	1.0	0.0095
0.5 - 1%	2.0	2.0	0.00475
0.2 - 0.5%	2.0	5.0	0.0019
0.1 - 0.2%	2.0	10	0.00095
1000 - 5000 mg/L	0.2	1	9.5
500 - 2500 mg/L	0.2	2	4.75
200 - 1000 mg/L	0.2	5	1.9
100 - 500 mg/L	0.2	10	0.95

Note: To convert % to mg/L, multiply the % value by 10,000 to get mg/L values. To convert mg/L to %, divide the mg/L value by 10,000 to get % values.

Safety Precautions:

Peracetic acid is a strong oxidizer and corrosive. Handling of 35% PAA needs to be done in a hood. Wear gloves, safety glasses, face shield and lab coat. Spills need to be diluted with water immediately. Avoid contact of 35% PAA with organic material.

Standardization of Ceric Solution:

The normality of the Ceric Solution will decrease over time. Before use, verify the normality with the following procedure. This standardization should be done monthly.

- 1) Use a graduated cylinder or pipet to measure 50 mL of deionized water into a 125-mL Erlenmeyer flask.
- 2) Add 5 mL of 19.2 N Sulfuric Acid Standard Solution (Hach PN 203832). Swirl to mix.
- 3) Insert a clean delivery tube into a Ceric Standard Titration Cartridge.
- 4) Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.
- 5) Place the delivery tube tip into the solution. While swirling the flask, add 200 digits of Ceric Standard Solution.
- 6) Insert a clean delivery tube into a 0.2 N Sodium Thiosulfate Titration Cartridge.
- 7) Hold the Digital Titrator with the cartridge tip pointing up. Turn the delivery knob to until a few drops of titrant are expelled. Reset the counter to zero and wipe the tip.
- 8) Place the delivery tube tip into the solution. While swirling the flask, titrate with the sodium thiosulfate from an intense yellow color to a faint yellow color. Record the number of digits required. This step should require about 400-450 digits of titrant.
- 9) Add one drop of Ferroin Indicator Solution, swirl to mix. The solution will turn a faint blue.
- 10) Continue titrating the Ceric Standard Solution (using the 0.2 Sodium Thiosulfate Titration Cartridge) from faint blue to orange color. Record the number of digits required.
- 11) Calculate the correction factor:

Correction Factor = Digits Required / 500

12) Multiply the % H_2O_2 from *Step 7* in the H2O2 titration procedure by the correction factor to obtain the correct H_2O_2 concentration.



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