

TITLE: DETERMINATION OF HYDROGEN PEROXIDE AND PERACETIC ACID IN SOLUTIONS	PAGE 1 OF 3 PAGES
Enviro Tech Chemical Services, Inc.	Chemicals Division

PURPOSE

To determine hydrogen peroxide and peracetic acid content of a peracetic acid sample at equilibrium.

SCOPE AND APPLICATION

This procedure is applicable to peracetic acid solutions from 1% to 35% concentration by weight.

DEFINITIONS

PAA = Peracetic acid

H₂O₂ = Hydrogen peroxide

INTERFERENCES

The method is widely applicable because organics do not interfere.

EQUIPMENT

Analytical balance

250 mL Erlenmeyer flasks

Automatic titrator Dosimat 665 or equivalent

Magnetic stirrer

REAGENTS AND MATERIALS

(See the Critical Reagent List for approved suppliers)

<u>Chemical</u>	<u>Formula</u>	<u>Concentration</u>
Ceric (IV) Sulfate	Ce(SO ₄) ₂	0.1 N
Sulfuric Acid	H ₂ SO ₄	1 N
Ferriin Indicator Solution	Ferrous Phenanthroline	
Sodium Thiosulfate	Na ₂ S ₂ O ₃	0.1 N
Potassium Iodide	KI	2.5 N
Starch Indicator - stabilized with salicylic acid or HgCl ₂		1%

SAFETY PRECAUTIONS

Peracetic acid is a strong oxidizer and corrosive. Handling of 35% PAA needs to be done in a hood. Avoid contact of PAA 35% with organic material. Spills need to be diluted with water immediately.

Wear gloves, safety glasses, face shield and lab coat.

CALIBRATION

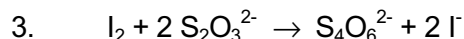
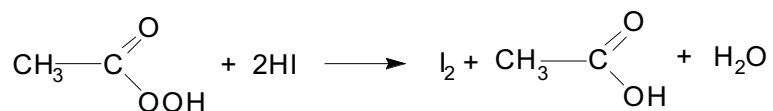
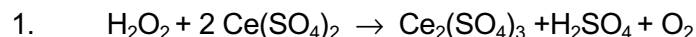
Check that Ceric (IV) sulfate and Sodium thiosulfate have been recently standardized or are fresh. Note the date of standardization or initial use. Dosimat should be calibrated annually. See calibration procedures if necessary.

SAMPLE HANDLING AND PRESERVATION

Peracetic acid requires several days to reach equilibrium. It also degrades in concentration over time. Material should be kept cool.

CHEMICAL BACKGROUND

The hydrogen peroxide content is determined by an oxidation reduction titration with ceric sulfate, according to equation 1. After the endpoint of this titration has been reached, an excess of potassium iodide is added to the solution. The hydroiodic acid formed in acidic media reacts with peracetic acid to liberate iodine, according to equations 2. A standard solution of sodium thiosulfate is used to titrate the liberated iodine, as shown in equation 3. The endpoint of this titration is used to calculate the peracetic acid content.



PROCEDURE

1. Weigh to the nearest of 0.0001 g an amount (m) of sample according to the assumed concentration of PAA as presented bellow.

PAA concentration	sample weight
[%]	[g]
35	0.2
15	0.2
5	0.5
1-2	0.5

2. Transfer the weighed amount into a 250 mL Erlenmeyer flask containing 50 mL of 1 N ice-cooled sulfuric acid. D.I. ice is important, do not avoid using ice !
3. Add 2 drops of ferroin indicator and titrate with 0.1 N ceric sulfate until disappearance of salmon color and appearance of light blue color.
4. Add 10 mL of 2.5 N KI solution. Allow red color to develop for several minutes.
5. Titrate with 0.1 N sodium thiosulfate.
6. When iodine color lightens to brown-yellow, add a few mL starch solution to produce purple color.
7. Add additional thiosulfate until solution turns salmon color for 15 seconds.
8. Record volumes of ceric sulfate and sodium thiosulfate.

CALCULATIONS

$$\%H_2O_2 = \frac{V_1 \times N_1 \times \text{meq}H_2O_2 \times F \times 100}{m}$$

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If $N_1=0.1$ and $F=1$,

$$\text{Then } \%H_2O_2 = \frac{0.17 \times V_1}{m}$$

$$\%PAA = \frac{V_2 \times N_2 \times \text{meqPAA} \times F \times 100}{m}$$

If $N_2=0.1$ and $F=1$,

$$\text{Then } \%PAA = \frac{0.38 \times V_2}{m}$$

Example: $\frac{26.117 \text{ ml } 0.1 \text{ N ceric sulfate} \times 0.17}{0.20 \text{ g sample}} = 22.2\% H_2O_2$

$$\frac{7.895 \text{ ml } 0.1 \text{ N sodium thiosulfate} \times 0.38}{0.20 \text{ g sample}} = 15.0\% PAA$$

Note:

$\%H_2O_2$ = concentration of H_2O_2 in weight percent

$\%PAA$ = concentration of PAA in weight percent

V_1 = volume of $Ce(SO_4)_2$ solution consumed for the H_2O_2 titration [mL]

N_1 = normality of $Ce(SO_4)_2$

$\text{meq}_{H_2O_2}$ = milliequivalent of H_2O_2 - The molecular weight of H_2O_2 divided by the number of electrons exchanged in the oxidation-reduction reaction, divided by 1000.

$\text{meq}_{H_2O_2} = \frac{34\text{g/mol}}{2e^- (1000)} = 0.017\text{g } H_2O_2 / \text{milliequivalent}$
or in this method g/mL of 1 N titrant

V_2 = volume of $Na_2S_2O_3$ consumed for the PAA titration [mL]

N_2 = normality of $Na_2S_2O_3$ solution

meq_{PAA} = milliequivalent of PAA - The molecular weight of PAA divided by the number of electrons exchanged in the oxidation-reduction reaction, divided by 1000.

$\text{meq}_{PAA} = \frac{76\text{g/mol}}{2e^- (1000)} = 0.038\text{g PAA/milliequivalent}$
or in this method g/mL of 1 N titrant

F = dilution factor (1 for undiluted samples)

m = amount of sample [g]

ENVIRONMENTAL PRECAUTIONS

Do not discharge the titrated sample in the sink without neutralization pH 6.5 to 7.5.

REFERENCES

Greenspan, F & Mackellar, Anal. Chem. 20,1061 (1948) FMC. Technical Bulletin 4, peracetic acid, 35%, page 10.