

**SCC**  
SOCIETY OF CHEMISTS



## PAR ROUND ROBIN TEST

### TEST PROCEDURE

#### DETERMINATION OF PERACETIC ACID

#### DETERMINATION OF PERACETIC ACID

##### 1. SCOPE AND FIELD OF APPLICATION

This method is suitable for the determination of the peracetic acid in peracetic acid products containing a excess of hydrogen peroxide (up to a ten fold) and in treatment solutions prepared from these products.

The range covered by this method is about 10 – 60 g/kg peracetic acid.

##### 2. PRINCIPLE

The sample is diluted in a solution of potassium iodide and a organic solvent at -10°C. The liberated iodine is titrated with sodium thiosulphate solution. Using these conditions the hydrogen peroxide reacts very slowly with the iodide and the peracetic acid determination is made without significant interference.

##### 3. REAGENTS

All reagents must be of analytical reagent grade unless otherwise stated, and water of an equivalent purity must be used in the method.

Sodium thiosulphate solution  $c(\text{Na}_2\text{S}_2\text{O}_3) = 0.1\text{N}$

Use a commercial available, certified titration solution. Don't use titration solutions older than 4 weeks

Organic solvent: Ethanediol or Methanol.

Carbon dioxide – solid, pelleted.

If carbon dioxide for cooling is not available, another equipment for cooling the reaction mixture to -10 °C must be used.

Potassium iodide solution  $c = 10/\text{g}$

Dissolve 10g of potassium iodide in 1 litre of water. Store in a brown glass bottle.


Discard the solution if it becomes colored.

##### 4. APPARATUS

Thermometer: capable of reading temperature to -20°C.

Weighing bottle, glass or polythene with a respective capacity.

Conical flask 500 ml

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Burette of respective volume and accuracy, or titration dosimat

## 5. PROCEDURE

**CAUTION: SAFETY SPECTACLES MUST BE WORN WHEN USING THIS METHOD.**

**Note: For the round robin test seven separate determinations with seven weight ins must be performed.**

Using a measuring cylinder, add 50 ml of ethanediol or methanol and 50 ml of the potassium iodide solution (10 g/L) to a 500 ml conical flask.

Cool the solution to a temperature of approximately  $-10^{\circ}\text{C}$  by the addition of solid carbon dioxide pellets or with another suitable available equipment.  
For example pre cool the solution in a suitable plastic container and store it in a freezer below  $-10^{\circ}\text{C}$ .

Weigh 0.75 g (to  $\pm 0.001\text{g}$ ) in the weighing bottle.

Transfer the sample to the cooled titration flask and titrate rapidly with sodium thiosulphate solution 0.1N until the solution turns colourless.

Please note:

Because of the hydrogen peroxide content the solution will becomes yellow again. It is therefore very important to titrate very rapidly and to fix the end point of the titration by the first decolorisation.


## 6. CALCULATION OF RESULT

1 ml of 0.1 N sodium thiosulphate solution is equivalent to 3.803 mg peracetic acid ( $\text{CH}_3\text{CO}_3\text{H}$ ).

$$\text{PAA (g/kg)} = \frac{A \text{ (mL)} \times 3.803}{m \text{ (g)}}$$

Where:

PAA content of peracetic acid in the product in g/kg  
A volume of sodium thiosulphate solution consumed in the titration, in mL  
m exact weight in of the sample, in g

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## Appendix

### HAZARD ASSESSMENT

This assessment supersedes any CAUTION notes in the text of the method, which may have been written before current legislation was in place. The information is supplied to third parties in good faith, but does not absolve them from their responsibility for carrying out their own risk assessment.

### MINIMUM CONTROL MEASURES FOR LABORATORY WORK

The work shall be carried out in accordance with current Disinfection Technology Team rules and guidelines for laboratory work. Use at sites which may not comply with these rules will require and additional assessment of any extra risks that this may cause.

### CHEMICAL HAZARDS

CHEMICAL	CATEGORY OF DANGER
Peracetic acid	Corrosive, Oxidiser
Sodium thiosulphate	Unclassified
Ethanediol	Corrosive
Methanol	Toxic, Flammable
CO <sub>2</sub> (solid)	Causes burns
Potassium iodide	Unclassified

### OVERALL CONTROL MEASURES

Use fume cupboard for open handling of peracetic acid, ethanediol and methanol. Keep peracetic acid away from combustible materials. Use suitable gloves and goggles or faceshield to BSPrEN 166 (BS2092 c or d) for pouring peracetic acid from containers. Wear gloves when handling corrosive materials. Keep flammable materials away from naked flames. Otherwise use standard laboratory precautions.

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**PAR ROUND ROBIN TEST**

**TEST PROCEDURE (REVISION 1)**

**DETERMINATION OF  
HYDROGEN PEROXIDE**

**DETERMINATION OF HYDROGEN PEROXIDE**

**1. SCOPE**

This method is suitable for the determination of the hydrogen peroxide content in peracetic acid containing solutions.

The range covered by this method is 5-200 g/kg hydrogen peroxide.

**2. PRINCIPLE**

The sample is dissolved in diluted sulphuric acid and cooled with ice. The hydrogen peroxide is titrated with ceric sulphate solution using ferroin as indicator.

**3. REAGENTS**

All reagents must be of analytical reagent grade unless otherwise stated, and water of equivalent purity must be used in the method.

Ceric sulphate solution c ( $\text{Ce}(\text{SO}_4)_2$ ) = 0.1N

Use a commercial available, certified titration solution. Don't use titration solutions older than 4 weeks.

Diluted sulphuric acid solution (1 + 19)

Carefully and slowly add 50 ml of sulphuric acid (d 1.84 kg/L) to 950 ml of water in a 2 litre beaker with continuous stirring and cooling.

**CAUTION: SAFETY GOGGLES MUST BE WORN WHEN HANDLING CONCENTRATED SULPHURIC ACID.**

Ferroin indicator solution

Dissolve 0.1740 g of ferrous sulphate heptahydrate in 25 ml of water. Add 0.3712 g of 1,10-phenanthroline mono-hydrate to the solution and stir to dissolve.

**4. Apparatus**

Weighing bottles, glass or polythene 1 – 5ml capacity.

Thermometer: capable of reading temperature to 0 °C

Conical flask 500 mL

Burette of respective volume and accuracy, or automatic titration equipment

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**PAR ROUND ROBIN TEST  
TEST PROCEDURE (REVISION 1)**

**DETERMINATION OF  
HYDROGEN PEROXIDE**

**5. PROCEDURE**

**CAUTION: SAFETY SPECTACLES MUST BE WORN WHEN  
USING THIS METHOD**

**Note: For the round robin test seven separate determinations with seven weight ins must be performed.**

Weigh 0.25 g (to  $\pm 0.001$ g) in the weighing bottle. Note the exact weight in for the calculation.

Measure 150 ml of sulphuric acid solution (1+ 19) into a 500 ml conical flask and add sufficient crushed ice to maintain a temperature of less than 10°C during the titration. When the temperature of the solution is below 10°C add about ten drops of ferroin indicator solution and tritrate dropwise with ceric sulphate solution (0.1N) until the colour changes to a blue.

Add the weighing bottle containing the sample to the cold solution and swirl to mix. Titrate rapidly with ceric sulphate solution (0.1N) to the same blue colour.

Note the consumed ceric sulphate solution (= A (ml)).

**6. CALCULATION**

1 mL of the 0.1 N ceric sulphate solution is equivalent to 1.701 mg hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>)

$$\text{H}_2\text{O}_2 \text{ (g/kg)} = \frac{\text{A (mL)} \times 1.701}{\text{m (g)}}$$

Where:

H<sub>2</sub>O<sub>2</sub> content of hydrogen peroxide in the product in g/kg  
A volume of ceric sulphate solution consumed in the titration, in mL  
m exact weight in of the sample, in g



## PAR ROUND ROBIN TEST

### TEST PROCEDURE (REVISION 1)

#### DETERMINATION OF HYDROGEN PEROXIDE

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Add the weighing bottle containing the sample to the cold solution and swirl to mix. Titrate rapidly with ceric sulphate solution (0.1N) to the same blue colour.

Note the consumed ceric sulphate solution (= A (ml)).


#### 6. CALCULATION

1 mL of the 0.1 N ceric sulphate solution is equivalent to 1.701 mg hydrogen peroxide ( $\text{H}_2\text{O}_2$ )

$$\text{H}_2\text{O}_2 \text{ (g/kg)} = \frac{\text{A (mL)} \times 1.701}{\text{m (g)}}$$

Where:

$\text{H}_2\text{O}_2$  content of hydrogen peroxide in the product in g/kg  
A volume of ceric sulphate solution consumed in the titration, in mL  
m exact weight in of the sample, in g

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#### CHEMICAL HAZARDS

CHEMICAL	CATEGORY OF DANGER
Peracetic acid	Corrosive, Oxidiser
Ceric sulphate	Irritant
Sulphuric acid	Corrosive
Sulphuric acid (1 + 9)	Irritant
1,10 Phenanthroline hydrate	Toxic

#### OVERALL CONTROL MEASURES

Use commercially available ready prepared ferroin indicator solution. Use fume cupboard for open handling of peracetic acid, keep away from combustible materials. Wear Marigold Suregrip gloves and goggles or faceshield to BSPrEN 166 (BS2092 c or d) for pouring peracetic acid from containers and for diluting concentrated sulphuric acid. Carry out Ceric sulphate titration in a fume cupboard. Wear gloves when handling corrosive materials. Otherwise use standard laboratory precautions.