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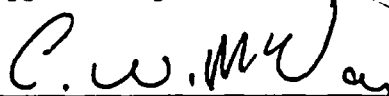
WEST VALLEY NUCLEAR SERVICES, CO., INC.

ANALYTICAL CHEMISTRY METHOD
ANALYTICAL AND PROCESS CHEMISTRY

ACM-H202-2101, Rev. 1
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DETERMINATION OF HYDROGEN PEROXIDE
IN AQUEOUS SOLUTIONS

Approved by:



C. W. McVay, Manager
Analytical & Process Chemistry

Part I

1.0 PURPOSE

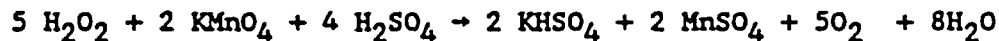
This method is for the determination of hydrogen peroxide in aqueous solutions, such as those used in off-gas scrubbers.

2.0 APPLICATION

This method can be used on solutions free of other oxidizing or reducing materials and clear enough to see the end point of the titration. A range of 1-50 mg can be determined in the sample aliquot.

3.0 DISCUSSION

Potassium permanganate reacts with hydrogen peroxide forming a colorless solution by the following reaction:



This reaction is normally slow to begin. The presence of manganese ions, however, acts as a catalyst and allows the titration to be done without delay.

The end point of the titration is observed when a pink color persists from a slight excess of permanganate.

4.0 REFERENCES

4.1 Scotts Standard Methods of Chemical Analysis, Wilfed W. Scott, 5th Edition, D. VanNostrand, Co.

Part II

5.0 EQUIPMENT

5.1 Volumetric glassware.

> 5.2 25 mL buret.

6.0 REAGENTS AND STANDARDS

> 6.1 Prepare stock solution per ACP 8.1. Potassium permanganate (0.1N): Dissolve 3.16 g KMnO₄ in deionized water and dilute to 1 l in a volumetric flask.

> 6.2 Sulfuric acid solution (1:4): Add 10 ml H₂SO₄ slowly to 40 ml H₂O with stirring in a 250 mL Erlenmyer flask.

6.3 Potassium oxalate.

6.4 Manganous sulfate.

6.5 Standardization of potassium permanganate

6.5.1 The normality of the potassium permanganate solution is verified by titrating a solution of potassium oxalate. This should be done for each new batch of permanganate and weekly as a check of the stability of the solution.

> 6.5.2 Weigh 0.2 g of potassium oxalate, weighed to the nearest 0.1 mg. Dissolve this in about 100 ml of H₂O and add 10 ml H₂SO₄ (1 : 4). Add a small scoop of MnSO₄ (about 10-50 mg) and titrate with 0.1 N KMnO₄ solution to a persistent pink color.

6.5.3 Determine a blank by titrating 100 ml of water with the H₂SO₄ and MnSO₄ added, as above. Subtract this from all titrations to obtain the correct ml of KMnO₄ in the calculations.

6.5.4 Calculate the normality of the permanganate solution as follows:

$$N = \frac{\text{mg potassium oxalate}}{92 \times (\text{ml KMnO}_4)}$$

7.0 SAFETY PRECAUTIONS

7.1 When making sulfuric acid solutions, always add the acid slowly to the water to avoid localized boiling.

7.2 Disposable skin-dex gloves should be worn when handling permanganate and peroxide solutions.

7.3 Standard lab safety precautions as stated in ACP 7.2.

8.0 RECORDS

8.1 All measurement data and sample identification shall be recorded on the worksheet (Attachment A). The final result shall be recorded on the analytical request sheet (PRD 5.0).

9.0 CALIBRATION AND CONTROL

> The calibration of the potassium permanganate solution is verified weekly, when applicable by 6.5 of this procedure and recorded in standard logbook and on the worksheets. A standard hydrogen peroxide solution shall be analyzed prior to the start of daily hydrogen peroxide sample analysis to verify the method is in control. If results are outside the established limits more than twice in succession, the potassium permanganate solution must be restandardized and the QC sample repeated. If this does not correct the problem, a review of the method by the Analytical Chemistry Manager will be required. A Quality Assurance log book and control chart will be used to record all standard checks per ACP 8.2.

10.0 PROCEDURE

> Transfer a suitable aliquot of the test solution (0.1 mL for off-gas surge, to 0.5 mL for other samples) to a 250 mL Erlenmyer flask. Add approximately 100 ml H₂O, 10 ml H₂SO₄ (1 : 4) and a small scoop of MnSO₄. Titrate with the permanganate solution until the end point is reached.

11.0 CALCULATION

$$\% \text{ H}_2\text{O}_2 = \frac{\text{ml KMnO}_4 \times N \times 1.701}{\text{ml of sample aliquot}}$$

12.0 ATTACHMENT

12.1 Attachment A - Hydrogen peroxide worksheet.

ATTACHMENT A

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HYDROGEN PEROXIDE WORKSHEET

SAMPLE NAME _____ LOG NUMBER _____

SPECIAL INSTRUCTIONS _____

N - mg POTASSIUM OXALATE / 92 x mL KMnO₄

NORMALITY OF KMnO₄ _____

PERCENT - mL x N x 1.701 / mL OF SAMPLE

SAMPLE ID							
SAMPLE VOLUME							
mL END							
mL START							
mL TITRATED							
BLANK							
mL TITRATED - BLANK							
NORMALITY KMnO ₄							
PERCENT H ₂ O ₂							

Calibrated Equipment used: _____

ANALYST _____
APPROVED _____

DATE _____
DATE _____