

HYDROGEN PEROXIDE IN WORKPLACE ATMOSPHERES

Method No: ID-126-SG

Matrix: Air

OSHA Standard: 1 ppm TWA

Validation Level: 0.5 ppm to 2 ppm

Collection Procedure: TiOSO_4 in aidget fritted-glass impinger

Recommended Air Volume: 100 liters at 1 Lpm

Analytical Procedure: Differential Pulse Polarography

Determination Limit or Reliable Quantitation Limit: 0.10 ppm

Precision: $CV_1 = 0.0261$

Method Classification: New/Interim

Approved by:

Draft Subject
to Revision

HYDROGEN PEROXIDE

1. Introduction

1.1. Scope

- 1.1.1. This method describes the sampling of hydrogen peroxide using TiOSO_4 and the analysis of hydrogen peroxide by differential pulse polarography.

1.2. Advantages and Disadvantages

- 1.2.1. The analytical method is simple and specific.
- 1.2.2. The $\text{TiOSO}_4\text{-H}_2\text{O}_2$ complex is stable for over seven weeks. (See the H_2O_2 backup report).
- 1.2.3. The collection of hydrogen peroxide can be monitored by observing the clear TiOSO_4 solution change to a yellow color when the $\text{TiOSO}_4\text{-H}_2\text{O}_2$ complex forms.
- 1.2.4. The method has better sensitivity than the colorimetric method (7.1.) and has fewer interferences (See the H_2O_2 backup report).

1.3. Principle (7.4.)

- 1.3.1. The sample is collected using a Nidget fritted-glass bubbler containing 15 mL TiOSO_4 .
- 1.3.2. The sample is analyzed for H_2O_2 by differential pulse polarography at a dropping mercury electrode. The current (in uA) of known standards are plotted against the concentrations of the standards to quantitate the H_2O_2 .

2. Range and Detection Limit

2.1. The detection limit is 0.10 ppm for a 100 L air sample. The working analytical range is 5 to 100 ug.

3. Precision and Accuracy

3.1. Eighteen samples were spiked at three levels corresponding to levels of 1/2, 1, and 2 times the PEL. The CV₁ [pooled] for the three levels is 0.0261.

4. Interferences

4.1. Very high levels of strong oxidants and reductants will interfere with the analysis. See the H₂O₂ backup report.

5. Sampling

5.1. Apparatus

5.1.1. An air sampling pump capable of operating at a sampling rate of 1.0 Lpm. The pump must be properly calibrated so that the volume of air sampled can be determined accurately from the flow rate and time.

5.1.2. Midget fritted-glass bubbler.

5.1.3. 0.00115 M TiOSO₄ collection solution.

5.2. Procedure

5.2.1. Sampling is done in accordance with current instructions contained in OSHA directives to the industrial hygienist.

5.2.2. The sample is collected in a midget fritted-glass bubbler containing 10 to 15 mL of 0.00115 M TiOSO₄ solution (6.2.3.) using a flow rate of 1.0 liter per minute. A 100 liter air sample is recommended.

- 5.2.3. Ship to the laboratory as soon as possible. Do not use metal cap liners in the vial caps and tape the lids shut. Send one blank with every 10 samples.

6. Analytical Procedure

6.1. Apparatus

- 6.1.1. 25 mL class A burette with Teflon stopcock.
- 6.1.2. Glass volumetric pipettes.
- 6.1.3. Micropipettes with tips.
- 6.1.4. 125 mL Erlenmeyer flask.
- 6.1.5. Polarographic analyzer - Model 374 or 504 manufactured by Princeton Applied Research (PAR) or equivalent.
- 6.1.6. Static mercury drop electrode - PAR 303 or equivalent.
- 6.1.7. 15 mL glass or polyethylene polarographic cells.
- 6.1.8. Nitrogen purification apparatus.

- 6.2. Reagents - All chemicals should be ACS reagent grade or equivalent, and the dilution water must be deionized.

- 6.2.1. 0.0575 M Titanium Oxy sulfate: Add 4.6 g TiOSO_4 , 20 g $(\text{NH}_4)_2\text{SO}_4$, and 100 mL concentrated H_2SO_4 to a 500 mL beaker. See precautions in 6.3.1. Heat gradually for several minutes until the chemicals are dissolved. Cool the mixture to room temperature and pour carefully into about 350 mL deionized water in a 500 mL volumetric flask. Filter the solution through an HA filter to remove any particulates, and dilute to 500 mL. The solution should be stable for 6 months.

- 6.2.2. 0.00575 M Titanium Oxysulfate: Make a 1-10 dilution of the 0.0575 M TiOSO_4 stock solution by adding 10 mL of the stock solution (6.2.1.) to a 100 mL volumetric flask and diluting to volume with deionized water. This solution should be made fresh monthly.
- 6.2.3. 0.00115 M Titanium Oxysulfate: Make a 1-50 dilution of the stock TiOSO_4 solution (6.2.1.) by adding 2 mL of the stock to a 100 mL volumetric flask and diluting to volume with deionized water. This solution should be made fresh monthly.
- 6.2.4. Supporting Electrolyte: Add 53 g $(\text{NH}_4)_2\text{SO}_4$, 36 g EDTA, and 75 mL of 28.2 M $(\text{NH}_4)\text{OH}$ to about 500 mL deionized water in a 1000 mL volumetric flask. Let cool, then dilute to 1000 mL with D.I. water.
- 6.2.5. 4 M Sulfuric Acid: Slowly add 112 mL H_2SO_4 to about 500 mL deionized water in a 1 L volumetric flask, stir and let cool. See precautions in 6.3.1. Dilute to 1 L with deionized water.
- 6.2.6. Starch indicator solution: To 5 g starch add a little cold water and grind in a mortar to a thin paste. Scrape into 1 L of boiling distilled water, stir, and let the covered solution settle overnight. Decant the clear supernate into a brown bottle and preserve with 4 g zinc chloride.
- 6.2.7. 0.1 M Sodium Thiosulfate: Add 24.82 g $\text{Na}_2\text{S}_2\text{O}_3 \cdot \text{H}_2\text{O}$ to about 500 mL deionized water in a 1000 mL volumetric flask and let dissolve. Dilute to volume with deionized water. Add two or three mL chloroform to minimize bacterial decomposition.
- 1 M Ammonium Molybdate: Add 20.6 g $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$ to about 50 mL deionized water in a 100 mL volumetric flask and dissolve. Dilute to volume with deionized water. Store in glass.

6.2.9. 1 M Potassium Iodide: Add 33.2 g of KI crystals to 100 mL deionized water, dissolve, and dilute to 1 L. Store in a brown bottle.

6.3. Precautions

6.3.1. When handling mercury, hydrogen peroxide, or sulfuric acid, gloves and safety glasses must be worn. Extreme care must be observed to avoid splashing or spilling on skin. Add sulfuric acid to water very carefully and never add water to sulfuric acid. Sulfuric acid gives off a great deal of heat when added to water and can splutter or boil violently. To prevent the heat from shattering the volumetric flask, place the flask in a cool water bath and add the sulfuric acid a little at a time.

6.3.2. Refer to the polarographic instruction manual for instrumental safety precautions (7.2., section I-1, and 7.3. section I-1).

6.4. Sample Preparation

6.4.1. Open the collection vial and measure the sample volume using a graduated cylinder. Take an aliquot of sample and transfer to a 15 mL polarographic cell. The sample aliquot size will depend on the intensity of the color of the collecting solution. If the sample is very yellow, use a 1 mL aliquot of sample and add 4.0 mL of the 0.00115 M TiOSO_4 (6.2.3.). If the sample is colorless, use a 5 mL aliquot.

6.4.2. Add 5 mL of the supporting electrolyte (5.2.4.) to give a total volume of 10 mL and analyze by differential pulse polarography.

6.5. Standard Preparation

- 6.5.1. A hydrogen peroxide stock solution is prepared by placing 2 mL of 30 % H_2O_2 in a 500 mL volumetric flask and diluting to volume with deionized water. This is approximately 1200 $\mu\text{g/mL}$ H_2O_2 .
- 6.5.2. A hydrogen peroxide standard solution is prepared by placing 1 mL of the H_2O_2 stock (6.5.) in a 100 mL volumetric flask and diluting to volume with deionized water. This is approximately 12 $\mu\text{g/mL}$ H_2O_2 .
- 6.5.3. Prepare a series of standards in the analytical range of 6 to 48 μg by making the following serial dilutions. Add to the polarographic cell the appropriate aliquots of the H_2O_2 standard solution (6.5.2.) and aliquots of deionized water using the calibrated micropipettes. Add 1 mL of the 0.00575 M TiOSO_4 (6.2.2.) and 5 mL of the supporting electrolyte (6.2.4.) to make a total volume of 10 mL.

Stock Solution (<u>conc</u>)	Aliquot H_2O_2 (<u>mL</u>)	Aliquot D.I. H_2O (<u>mL</u>)	Final Standard (<u>ug</u>)
12	4.0	0.0	48
12	3.0	1.0	36
12	2.0	2.0	24
12	1.0	3.0	12
12	0.5	3.5	6

6.6. Analysis

- 6.6.1. Turn on the polarograph, Model 384 and 303 and allow to warm up for at least 30 minutes.
- 6.6.2. Analyze the standards and samples by differential pulse polarography using the following instrumental conditions:

Initial Potential	-0.820 V
Final Potential	-1.020 V
Purge Time	300 sec
Scan Increment	2 mV
Replications	1
Drop Time	0.5 seconds
Peak Location	Yes
Peak Potential	-0.940 V
Date	as needed

The method is stored as Method No. 2 in the PAR Model 384.

- 6.6.3. Prepare the samples and working standard solutions as described in sections 6.4. and 6.5.
- 6.6.4. Purge each standard and sample for 5 minutes with pre-purified nitrogen.
- 6.6.5. Analyze the reagent blank, standards, and the samples. A standard should be re-analyzed after every 4 or 5 samples.
- 6.6.6. Record the peak current and potential for each standard and sample in the laboratory notebook. The differential pulse polarogram of hydrogen peroxide gives a peak at approximately -0.940 V.
- 6.6.7. If any of the samples have enough hydrogen peroxide to be over the PEL, the 1200 ug/mL stock (5.5.1.) must be standardized against the 0.1 M sodium thiosulfate (6.2.7.) before a standard curve is prepared. See 6.6.9. through 6.6.12.
- 6.6.8. Use any available least square regression program to plot a calibration curve of peak current vs. concentration (ppm, ppb, or total ug) of the standards.

6.6.9. To standardize the H_2O_2 stock solution, transfer the following solutions to a 125 mL Erlenmeyer flask.

1. 10 mL stock 1200 $\mu\text{g/mL}$ H_2O_2 (6.5.1.)

2. 10 mL 2M H_2SO_4 (6.2.5.)

3. 6 mL 1M KI (6.2.9.)

4. 3 drops 1M $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}$ (6.2.3.)

5. 20 mL D.I. water

6.6.10. The solution is titrated to a very faint yellow with 0.1 M $\text{Na}_2\text{S}_2\text{O}_3$ (6.2.7.) and then 1 mL starch solution (6.2.6.) is added to produce a blue color. The titration is continued until the solution becomes colorless.

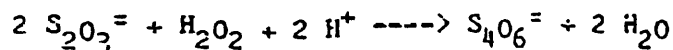
6.6.11. The total amount of $\text{Na}_2\text{S}_2\text{O}_3$ required to reach the endpoint is determined (about 10 mL) and recorded.

6.6.12. Calculate the concentration of the 1200 $\mu\text{g/mL}$ H_2O_2 stock, the 12 $\mu\text{g/mL}$ standard, and the actual concentrations of the standards to be used in the standard curve.

6.7. Calculations

6.7.1. Subtract the initial volume of sodium thiosulfate from the volume at the endpoint. This is the total volume of $\text{Na}_2\text{S}_2\text{O}_3$ used.

Since:



Then:

$$M \text{ Na}_2\text{S}_2\text{O}_3 \times V \text{ Na}_2\text{S}_2\text{O}_3 = 2 \times M \text{ H}_2\text{O}_2 \times V \text{ H}_2\text{O}_2$$

or:

$$0.1 \times \text{mL Na}_2\text{S}_2\text{O}_3 \text{ used} = M \text{ H}_2\text{O}_2 \times 2 \times 10 \text{ mL, and:}$$

$$\text{mmoles H}_2\text{O}_2 = \text{mmoles Na}_2\text{S}_2\text{O}_3 \times 1/2 \text{ then:}$$

$$\begin{aligned} \mu\text{g H}_2\text{O}_2 &= \text{mmoles Na}_2\text{S}_2\text{O}_3 \times 1/2 \times 34 \\ &= \text{mmoles Na}_2\text{S}_2\text{O}_3 \times 17 \end{aligned}$$

6.7.2. The weight of H_2O_2 in a sample aliquot is determined from the calibration curve. The total weight of H_2O_2 is calculated from the equation:

$$\mu\text{g H}_2\text{O}_2 = \frac{(\text{alic. } \mu\text{g} - \text{blank alic.})(\text{sample vol. mL})}{(\text{sample aliquot vol. mL})}$$

6.7.3. The concentration of H_2O_2 is calculated in $\mu\text{g/L}$, converted to $\mu\text{g/m}^3$, and then to ppm.

$$\mu\text{g H}_2\text{O}_2/\text{liters sampled} = \mu\text{g/m}^3 \quad \text{and;}$$

$$\text{ppm H}_2\text{O}_2 = \mu\text{g/m}^3 \times 24.45/34 = \mu\text{g/m}^3 \times 0.719$$

7. References

- 7.1. Hydrogen Peroxide Colorimetric Method, Method No: VI-6, Last Revised on January 26, 1978.
- 7.2. Instruction Manuals, Polarographic Analyzer, Model 374 and Hanging Mercury Drop Electrode Model 303, Princeton Applied Research, Princeton, NJ.
- 7.3. Polarographic Instruction Manual, Model 384, Princeton Applied Research, Princeton, NJ.

- 7.4. Boto, K.G., and Williams, L.F.G., *Analytica Chimica Acta*, Vol. 85, pp 179-183 (1976).

BUD

Backup Data Report

Substance: Hydrogen PeroxideOSHA-PEL: 1.0 ppm TWA

Chemical used

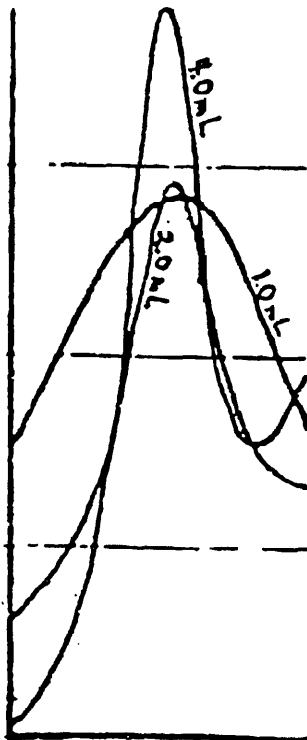
for validation: Hydrogen Peroxide, Analytical Reagent, 30 %, Mallinckrodt.

1. Procedure

The general procedure used is described in the OSHA Sampling and Analytical Method (SAM) for hydrogen peroxide. Instrumental analysis was done by Carl Cook (See Reference 8.1). This method replaces the colorimetric method (8.2).

2. Analysis

The analysis of hydrogen peroxide is by differential pulse polarography (DPP), see Reference 8.1. 5.0 mL of the supporting electrolyte and 5.0 mL of the sample or standard solution is placed in a 10 mL sample cell. The sample or standard must be in 5.0 mL 0.00115 M TiOSO_4 . This gives a much sharper and larger peak than 4 or less mL of the 0.00115 M TiOSO_4 as can be seen from the diagram below.



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3. Generation

Hydrogen peroxide was generated by adding 25 mL of 30 % hydrogen peroxide to a flask and heating the flask while bubbling N_2 through the solution at a rate of 1 LPM. The hydrogen peroxide was collected in 15 mL of $TiSO_4$ in a midjet fritted glass bubbler.

4. Collection Efficiency

Hydrogen peroxide was generated for 40 minutes, and while the 1st impinger collected 500 ug/mL H_2O_2 (about 60 times the PEL), the 2nd impinger showed no hydrogen peroxide collected. This means that at levels below 60 times the PEL there is 100 % collection efficiency.

5. Storage Stability

To assess the stability of hydrogen peroxide in $TiOSO_4$, a time study was conducted at the 0.5, 1.0, and 2.0 PEL level.

On 1/9/84, 24 samples were prepared for analysis over a two month period to determine the storage stability. Assuming that 100 L of air were taken in 15 mL $TiOSO_4$, there would be 75 ug H_2O_2 found in a sample at 1/2 the PEL, 150 ug at the PEL, and 300 ug at 2 times the PEL. Eight samples were prepared at each level and contained 15 mL of 0.00115 M $TiOSO_4$, plus the spiked H_2O_2 concentration. Table I gives the results of the stability study.

Table I Stability Study Data

Day	ug found	ug expected	f/t
1	75.	75.	1.000
1	176.	150.	1.173
1	347.	300.	1.157
4	76.	75.	1.113
4	141.	150.	0.940
4	295.	300.	0.983
3	74.9	75.	0.999
2	149.	150.	0.993
8	300.	300.	1.000
15	91.5	75.	1.220
15	183.	150.	1.220
15	367.	300.	1.223
51	76.3	75.	1.017
51	150.	150.	1.000
51	377.	300.	1.259

From the results it can be seen that hydrogen peroxide is stable in $TiOSO_4$ for 51 days, or almost 2 months. One problem that was noticed was that although the hydrogen peroxide- $TiOSO_4$ complex is stable for two months, the $TiOSO_4$ stock solution (0.05775 M) and subsequent diluted solutions of the 0.05775 M $TiOSO_4$ stock solution are not stable. A comparison of the 0.05775 M QC stock solution and the 0.05775 M Laboratory stock solution showed significant differences. The QC stock was 12 months old and the lab stock was 3 months old. When analyzed by the colorimetric method, samples spiked with

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96 ug in the QC stock showed 80 ug, whereas samples spiked with 96 ug and collected in the lab stock showed 96 ug. The standards were made using the lab stock TiOSO_4 . When the samples were analyzed by DPP, all samples showed 96 ug. This is due either to the fact that the QC stock solution is 9 months older than the lab stock solution or differences in solution preparation. This points out another problem with the colorimetric analysis. The results indicate that age and/or makeup of the TiOSO_4 solutions are not as important when the DPP method is used.

6. Interferences

Table II shows the effects of different interferences on the analysis of hydrogen peroxide. 96 ug of hydrogen peroxide was placed in a 10 mL sample cell along with different levels of interferent. From Table II it can be seen that the only serious interferent with the DPP method is KMnO_4 which will also prevent the analysis of hydrogen peroxide using the colorimetric method. Additionally, KI does not effect the analysis of hydrogen peroxide by DPP but does prevent the analysis of hydrogen peroxide using the colorimetric method.

Table II Effect of Interferent on the Analysis of Hydrogen Peroxide

ug H_2O_2 Added	Interferent Added	H_2O_2 /Interferent ratio	uA	Peak location (V)
96	0.4 ppm SnCl_2	1:02	2.66	-.948
96	20 ppm K_2CrO_4	1:1	2.41	-.950
96	0.2 ppm KMnO_4 *	1:01	—	—
96	0.3 ppm $\text{NH}_2\text{OH} \cdot \text{HCl}$	1:04	1.84	-.948
96	2860 ppm $\text{Na}_2\text{S}_2\text{O}_3$	1:150	2.17	-.950
96	10 ppm Cr_2O_3	1:5	2.43	-.948
96	33200 ppm KI *	1:1800	2.09	-.950
96	20 ppm $\text{K}_2\text{S}_2\text{O}_8$	1:1	2.26	-.950

* These were highly colored and would not allow analysis by the colorimetric method.

7. Precision and Accuracy

The last day of the study was on day 51, the results from day 51 are tabulated below.

# of Samples Analyzed	Concentration Expected	Concentration Found (ave)	CV_1
6	75.0	76.0	0.0312
6	150.0	150.0	0.0166
6	300.0	373.0	0.0231

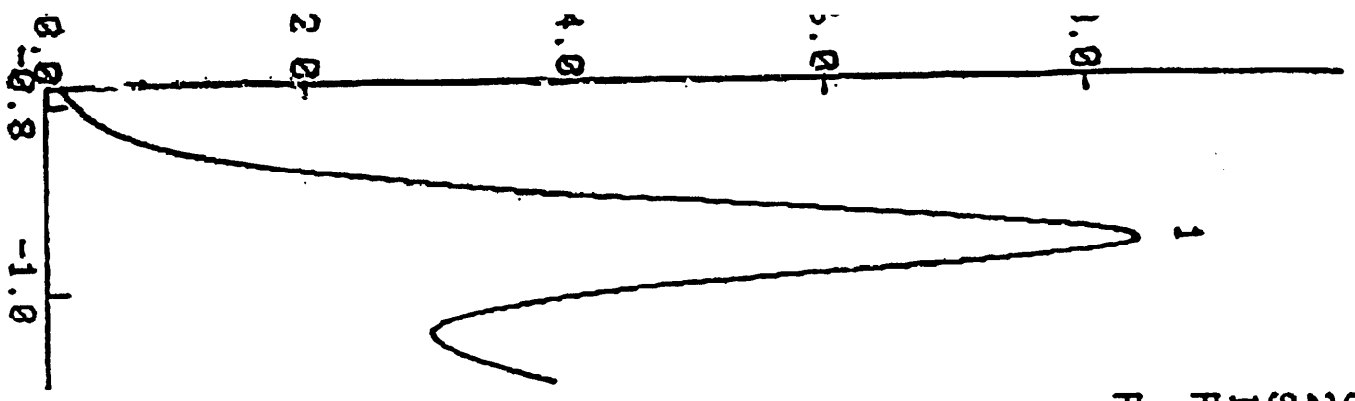
The CV_1 [pooled] for the three sets of samples was 0.0261. Six samples for each of the three different concentration ranges were used.

Appendix I contains typical polarograms of 120. ug and 75. ug H_2O_2 respectively in a 10 mL sample cell.

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8. References

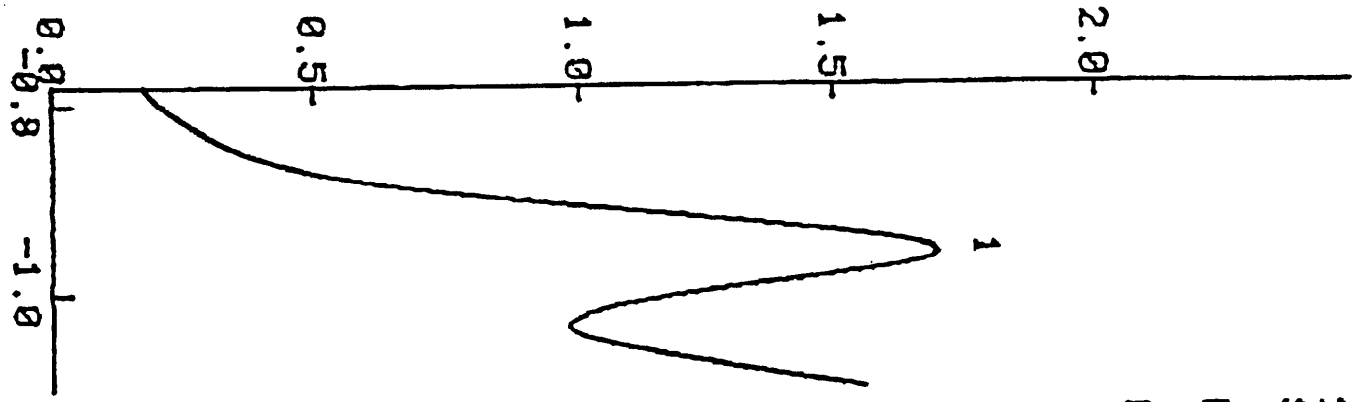
1. Hydrogen Peroxide in Workplace Atmospheres, Method No: ID-126-SG.
2. Hydrogen Peroxide Colorimetric Method, Method No: VI-6, Last Revised on January 26, 1978.



CURRENT X: 10^{-2} NA
 2-FEB-84 # 5
 SAMPLE DPP
 INITIAL E -0.780 V
 FINAL E -1.100 V

PEAK
 1 -0.954V
 8.560E2 NA

120.49 μ g H_2O_2



CURRENT X: 10^{-2} NA
 2-FEB-84 # 6
 SAMPLE DPP
 INITIAL E -0.780 V
 FINAL E -1.100 V

PEAK
 1 -0.958V
 1.738E2 NA

75.19 μ g H_2O_2

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HYDROGEN PEROXIDE

A

Matrix: Air

Method No: VI-6

OSHA Standard: 1.4 mg/m³

Method Classification:

Analytical Procedure: MFCB -
Colorimetric

Date: February 22, 1977

Detection Limit: 2 µg H₂O₂

Date Revised: January 26, 1978

1. Principle of the Method:

- 1.1 H₂O₂ vapor is collected in a midget fritted glass bubbler containing 15 ml of TiOSO₄ collecting solution.
- 1.2 A 5 ml aliquot of the collecting solution is transferred to a 2 cm cuvette and 10 ml of distilled water are added.
- 1.3 The absorbance of the solution at 410 nm is read and compared to the absorbance of standards.

2. Range and Detection Limit:

- 2.1 When using 2 cm cells and a 15 ml final volume, a 1% absorption or detection limit occurs at about 2 µg H₂O₂. For a 100 liter air sample this translates to a detection limit of about 0.06 mg H₂O₂/m³ or approximately 5 percent of the present TLV of 1.4 mg/m³. The yellow titanium - H₂O₂ complex is visually observed at 10 µg in the collecting solution or 0.1 mg/m³.
- 2.2 The range for this colorimetric method is useful from 2 µg H₂O₂ up to about 100 µg H₂O₂ which corresponds to 0.06 to 3.0 mg H₂O₂/m³ for a 100 liter air sample.

3. Precision and Accuracy:

The average percent error for the method is estimated as 2.9.

4. Advantages and Disadvantages:

- 4.1 The method is simple, specific, and sensitive. The Ti-H₂O₂ color complex is stable for over one week and insensitive to light and temperature. Semiquantitative visual field analysis is made possible by the spontaneous color complex formed by reaction of H₂O₂ with the collecting solution.

is:

5.1 Sampling Equipment:

5.1.1 A midget fritted glass bubbler.

5.1.2 An air-sampling pump capable of operating at a sampling rate of 0.5 lpm. The pump must be properly calibrated so that the volume of air sampled can be determined accurately from the flow rate and time.

5.1.3 Thermometer

5.1.4 Manometer

5.1.5 Stopwatch

5.2 Analytical Equipment:

5.2.1 Spectrophotometer set at 410 nm.

5.2.2 Matched cuvettes, 2 cm path length

5.2.3 Assorted glassware

6. Reagents:

All reagents must be analytical reagent grade or better.

6.1 A stock solution of titanium(IV) is prepared as follows:

4.6 g of TiOSO_4 , 20 g of $(\text{NH}_4)_2\text{SO}_4$, and 100 ml of concentrated H_2SO_4 are heated gradually for several minutes until the chemicals are dissolved. The mixture is cooled to room temperature, poured carefully into 350 ml H_2O , filtered through an HA filter to remove any trace of turbidity, and then diluted to 500 ml. A 1:50 dilution of this stock solution is the titanium reagent or collecting solution.

6.2 A standard H_2O_2 stock solution is prepared by placing 2 ml of 30 percent H_2O_2 in a 500 ml volumetric flask and adding distilled water to the mark. Two ml of this stock are diluted to 200 ml with distilled water. Aliquots of this solution are used as standards. The 10 ml aliquot is about 90 μg .

6.3 The solutions required for H_2O_2 standardization are:

6.3.1 4N H_2SO_4

6.3.2 1N KI

6.3.3 1N $(NH_4)_6Mo_7O_{24}$

6.3.4 0.1N $Na_2S_2O_3$

6.3.5 Starch solution prepared by adding 2 g soluble starch to 10 ml boiling water in which 1 g boric acid has been dissolved. This solution is boiled for 1 minute, cooled and stored in a stoppered bottle.

7. OSHA Collection Procedure.

7.1 H_2O_2 in air is collected in a midget fritted glass bubbler containing 15 ml of titanium reagent as collecting solution.

7.2 Air is drawn through the bubbler at a rate of 0.5 liter per minute. A 100 liter sample is recommended.

7.3 The solutions are transferred to capped bottles for shipping. Vinyl or waterproof tape is used around the caps to prevent leakage during shipment. The tape is wrapped around the cap in the direction the cap is turned. Each cap is also sealed with an official OSHA seal.

7.4 With each batch of samples, one bottle containing collection solution, labeled as a blank, should be submitted.

7.5 The bottles in which samples are collected should be shipped in a suitable container, designed to prevent damage in transit.

8. Analytical Procedure.

8.1 Analysis of Samples and Standards

8.1.1 The volume of each sample received should be determined and recorded. A 5 ml aliquot is transferred to a cuvette and the volume of each adjusted to 15 ml with 10 ml deionized water.

8.1.2 Standard H_2O_2 solutions are made by placing 5 ml of titanium reagent in each of 6 cuvettes. Aliquots of the standard H_2O_2 solution are added to each and the total volume is adjusted to 15 ml with water.

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- 3 The absorbance of each sample, blank, and standard are determined at 410 nm with a spectrophotometer with 0.00 absorbance corresponding to a reagent blank.

8.2 Standardization of H_2O_2

- 8.2.1 The following solutions are transferred to a 125 ml erlenmyer flask.

1. 4 ml stock H_2O_2
2. 21 ml water
3. 10 ml 4N H_2SO_4
4. 6 ml 1N KI
5. 3 drops 1N $(NH_4)_2MoO_4$

- 8.2.2 The solution is titrated to a very faint yellow with 0.1N $Na_2S_2O_3$ and then 1 ml starch solution is added to produce a blue color. The titration is continued until the solution is colorless.

- 8.2.3 The total amount of $Na_2S_2O_3$ required to reach the colorless end point is determined.

8.3 Interferences:

Positive interference can be expected from any compound collected that liberates H_2O_2 on acid hydrolysis. Negative interferences are a function of the reactivity of H_2O_2 with other compounds present in the air sample.

9. Calculations:

- 9.1 A standard curve is plotted from the absorbance values obtained for the standard H_2O_2 solutions.

Typical values used for a curve are given:

Standard Aliquot	(410 nm)	$\mu g H_2O_2$
10 ml	0.150	90
4 ml	0.087	36
2 ml	0.049	18
1 ml	0.024	9
0.5 ml	0.012	4.5

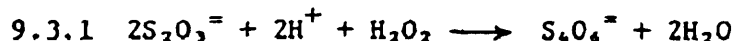
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The weight of H_2O_2 in a sample aliquot is determined from the calibration curve using the measured absorbance of the color developed by the sample aliquot.

The total weight of H_2O_2 in the sample is calculated by the equation

$$\mu g(H_2O_2) = \frac{(\text{aliquot } \mu g - \text{blank aliquot } \mu g) (\text{sample volume, ml})}{(\text{sample aliquot volume, ml})}$$

- 9.3 The following equation and calculations are used for standardization of 4 ml stock H_2O_2 standard.



- 9.3.2 The H_2O_2 normality is determined from the $S_2O_3^{2-}$ titrant volume, and the corresponding concentration of H_2O_2 is determined by the relationship /

$$, ppm(H_2O_2) = N(H_2O_2) \times 17.0 \times 1000.$$

- 9.3.3 The 4 ml standard solution weight is 1 percent of the 4 ml stock value since a 100:1 dilution was made.

- 9.4 The concentration of H_2O_2 in air is expressed in mg H_2O_2 per cubic meter of air.

$$mgH_2O_2/m^3 = \mu gH_2O_2/l$$

10. References:

- 10.1 Cohen, I., Purcell, T., Anal. Chem., 39, 131 (1967).
10.2 AOAC Methods of Analysis, 12th Ed. Washington D.C. 605 (1975).