



# Standard Test Method for Peroxide Number of Aviation Turbine Fuels<sup>1</sup>

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*This standard has been approved for use by agencies of the Department of Defense.*

## 1. Scope

1.1 This test method covers the determination of the peroxide content of aviation turbine fuels.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific precautionary statements, see 6.3, 6.6, 8.2, and Annex A1.

## 2. Referenced Documents

### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

D 4057 Practice for Manual Sampling of Petroleum and Petroleum Products<sup>3</sup>

### 2.2 Other Standards:

CRC Report No. 559 Determination of the Hydroperoxide Potential of Jet Fuels<sup>4</sup>

4500-C1 B. Iodometric Method I—Standard Methods for the Examination of Water and Wastewater<sup>5</sup>

## 3. Summary of Test Method

3.1 A quantity of sample dissolved in 1,1,2-trichloro-1,2,2-trifluoroethane is contacted with aqueous potassium iodide solution. The peroxides present are reduced by the potassium iodide. An equivalent amount of iodine is liberated, which is titrated with sodium thiosulfate solution. The results are calculated as milligrams per kilogram (ppm) of peroxide.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.05 on Properties of Fuels, Petroleum Coke and Carbon Material.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 05.02.

<sup>4</sup> Available from the Coordinating Research Council, Inc., 219 Perimeter Center Parkway, Atlanta, GA 30346.

<sup>5</sup> Published by the American Health Assoc., the American Water Works Assoc. and Water Environment Federation. Available from American Public Health Publication Sales, P. O. Box 753, Waldorf, MD 20604-0753.

## 4. Significance and Use

4.1 The magnitude of the peroxide number is an indication of the quantity of oxidizing constituents present. Deterioration of turbine fuel results in the formation of peroxides and other oxygen-carrying compounds. The peroxide number measures those compounds that will oxidize potassium iodide.

4.2 The determination of the peroxide number of aviation turbine fuels is significant because of the adverse effect of peroxides upon certain elastomers in the fuel systems.

## 5. Apparatus

5.1 *Iodine Number Flask*, 250 mL, glass-stoppered.

## 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>6</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

6.3 *Acetic Acid Solution*—Mix 4 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) with 996 mL of glacial acetic acid (CH<sub>3</sub>COOH (**Warning**—Poison Corrosive. Combustible can be fatal if swallowed. Causes severe burns. Harmful if inhaled. See A1.2)).

6.4 1,1,2-Trichloro-1,2,2 Trifluoroethane (**Warning**—See A1.1)

6.5 *Potassium Dichromate Solution, Standard (0.1 N)*—ACS reagent grade. Dissolve 2.452 g of the dried potassium

<sup>6</sup> "Reagent Chemicals, American Chemical Society Specifications," American Chemical Society, Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

dichromate ( $K_2Cr_2O_7$ ) in water and dilute to 500 mL in a volumetric flask. This solution is 0.1 *N*. As an alternative, the lab may use commercially prepared solution.

6.6 *Potassium Dichromate Solution, Standard (0.01 N)* (**Warning**—Avoid contact with eyes and skin and avoid breathing of dust)—Dilute 100 mL of 0.1 *N*  $K_2Cr_2O_7$  solution with water to 1000 mL in a volumetric flask.

6.7 *Potassium Iodide Solution*—Dissolve 120 g of potassium iodide (KI) in 100 mL of water. Larger quantities of solution may be prepared, provided the concentration of KI in water is equivalent. Discharge any color from this solution by placing 1 mL of KI solution, 50 mL of water, and 5 mL of starch solution in a 300-mL flask and blanketing with nitrogen or carbon dioxide. If a blue color develops, add 0.005 *N*  $Na_2S_2O_3$  solution from a microburet until color just disappears. Add a sufficient quantity of  $Na_2S_2O_3$  solution, thus determined, to the main KI solution to convert all free iodine to iodide. When properly prepared, 1 mL of KI solution should not turn blue when starch solution is added, but with starch plus one drop of 0.01 *N*  $K_2Cr_2O_7$  solution plus two drops of HCl, the blue color should develop. Store this solution under chloroform.

6.8 *Sodium Thiosulfate Solution, Standard (0.1 N)*—Dissolve 12.5 g of sodium thiosulfate ( $Na_2S_2O_3 \cdot 5H_2O$ ) plus 0.1 g of sodium carbonate ( $Na_2CO_3$ ) in 500 mL of water (the  $Na_2CO_3$  is added to stabilize the  $Na_2S_2O_3$  solution). Let this solution stand a week or more before using. As an alternative, the lab may use a commercially prepared solution. Standardize against 0.1 *N*  $K_2Cr_2O_7$  solution, using any appropriate technique, such as the one indicated in 4500-C1 B. Standardize at intervals frequent enough to detect changes of 0.0005 in normality.

6.9 *Sodium Thiosulfate Solution, Standard (0.005 N)*—Prepare a twenty-fold dilution of the 0.1 *N*  $Na_2S_2O_3$  solution prepared in 6.8, using a volumetric flask. One way to accomplish this is to dilute 100 mL of 0.1 *N*  $Na_2S_2O_3$  solution with water to 2000 mL in a volumetric flask. Prepare this solution fresh with restandardized 0.1 *N*  $Na_2S_2O_3$  solution when any change of 0.0005 or over in normality is detected.

6.10 *Starch Solution*—Make a paste of 6 g of arrowroot starch or soluble iodometric starch with cold water. Pour the paste into 1 L of boiling water. Add 20 g of potassium hydroxide (KOH), mix thoroughly, and allow to stand for approximately 2 h. Add 6 mL of glacial acetic acid. Mix thoroughly and then add sufficient HCl (sp gr 1.19) to adjust the pH value of the solution to 4.0. Store in a glass-stoppered bottle. Starch solution prepared in this manner can remain chemically stable for up to one year. As an alternative, the lab may use a commercially prepared solution (some are preserved with salicylic acid).

## 7. Sampling

7.1 Samples shall be taken in accordance with the procedures described in Practice D 4057.

## 8. Procedure

8.1 Select the appropriate weight of sample from the following table:

Estimated Peroxide Number, mg/kg	Sample Mass, g
0 to 10	50
11 to 30	35
31 to 50	25
51 to 80	10
81 to 100	5

8.2 Weigh the sample into a 250-mL iodine flask that has been flushed with nitrogen or carbon dioxide. Add 25 mL of 1,1,2-trichloro-1,2,2-trifluoroethane (**Warning**—See A1.1). Pass a vigorous flow of nitrogen or carbon dioxide through the solvent for at least 1 min; then, without stopping the gas flow, add 20 mL of acetic acid solution and reduce the flow of gas so that the rate is one bubble per second. Add 2 mL of KI solution and mix vigorously for  $30 \pm 1$  s. Set the flask aside to stand for  $5 \text{ min} \pm 3 \text{ s}$ .

8.3 At the end of the reaction period, add 100 mL of water and stop the gas flow. Add 5 mL of starch solution. Titrate with 0.005 *N*  $Na_2S_2O_3$  solution to the disappearance of the blue color.

NOTE 1—This end point is of the returning type. The end point intended in this method is the disappearance of the blue color for 30 s or longer.

8.4 Titrate a reagent blank just prior to each set of unknown determinations, following the same procedure as described in 8.1-8.3.

## 9. Calculation

9.1 Calculate the peroxide number as follows:

$$\text{Peroxide number, mg/kg} = [(A - B)N \times 1000 \times 8]/S \quad (1)$$

where:

*A* = millilitres of  $Na_2S_2O_3$  solution required for titration of the sample,

*B* = millilitres of  $Na_2S_2O_3$  solution required for titration of the blank,

*N* = normality of the  $Na_2S_2O_3$  solution, and

*S* = grams of sample used.

## 10. Precision and Bias

10.1 *Precision*—The precision of this test method, reported in 10.1.1, was originally determined by dissolving the sample in carbon tetrachloride. The precision when using 1,1,2-trichloro 1,2,2-trifluoroethane has been shown to match that of the original work and is reported in CRC Report No. 559.

NOTE 2—Prior to 1983, the test method called for the use of carbon tetrachloride and the precision was determined with carbon tetrachloride as the sample solvent. It has become the policy of most laboratories to avoid the use of carbon tetrachloride wherever possible because it is a carcinogen. 1,1,2-trichloro-1,2,2-trifluoroethane should now be used as per the procedure in Section 8 of this test method.

10.1.1 *Repeatability*—The difference between two successive test results, obtained by the same operator with the same

apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty:

$$r = 0.15X \quad (2)$$

where:

$X$  = the average of two test results.

10.1.2 *Reproducibility*—The reproducibility of the test method has not been determined because of the difficulty encountered in maintaining sample integrity when distributing

them to cooperative laboratories. Further work is being undertaken to come up with an acceptable reproducibility statement.

10.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this test method measuring the peroxide number of aviation turbines fuels, no statement on bias is being made.

## 11. Keywords

11.1 peroxide number

## ANNEX

### (Mandatory Information)

#### A1. PRECAUTIONARY STATEMENTS

##### A1.1 1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon TF)

A1.1.1 **Warning**—Inhalation of high concentrations may cause anaesthetic effects or asphyxiation. Repeated or prolonged skin contact will cause defatting and possible dermatitis. May produce toxic vapors if burned.

A1.1.1.1 Use with adequate ventilation.

A1.1.1.2 Keep container closed.

A1.1.1.3 Avoid prolonged breathing of vapor or spray mist.

A1.1.1.4 Avoid prolonged or repeated contact with skin.

A1.1.1.5 Can produce toxic vapors on contact with flames, hot glowing surfaces, or electric arcs.

##### A1.2 Acetic Acid (Glacial)

A1.2.1 **Warning**—Poison. Corrosive. Combustible. May be fatal if swallowed. Causes severe burns. Harmful if inhaled.

A1.2.1.1 Do not get in eyes, on skin, or on clothing.

A1.2.1.2 Do not breathe vapor, spray, or mist.

A1.2.1.3 Dilute by addition of acid to water.

A1.2.1.4 Keep away from heat and open flame.

A1.2.1.5 Keep in tightly closed container in approved acid storage cabinet.

A1.2.1.6 Keep cool.

A1.2.1.7 Loosen closure carefully when opening.

A1.2.1.8 Use with adequate ventilation.

A1.2.1.9 Keep container closed when not in use.

A1.2.1.10 Use protective clothing and goggles when handling.

A1.2.1.11 Wash thoroughly after handling.

##### A1.3 Potassium Dichromate

A1.3.1 **Warning**—Avoid contact with eyes and skin and avoid breathing of dust.

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