



AEROSPACE RECOMMENDED PRACTICE

ARP5991™

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Test Method for the Determination of Water Concentration in Polyol Ester and Diester Aerospace Lubricants by Coulometric Karl Fischer Titration

RATIONALE

ARP5991 has been reaffirmed to comply with the SAE five-year review policy.

1. SCOPE:

The test method describes the procedure for the direct determination of water concentration in polyol ester and diester based aerospace lubricants by the commercially available automated coulometric Karl Fischer titration instrument. The method was validated to cover the water concentration range of 150 to 3500 µg/g. The method may also be suitable for the determination of water concentrations outside this range and for other classes of fluids, however, the precision statement shall not be applicable for such uses.

1.1 Purpose:

Knowledge of the water concentration in polyol and diester aerospace lubricants is required since the presence of excess amounts of water can lead to hydrolysis of the lubricant, resulting in corrosion and wear of the lubricated components and degradation of the lubricant. Significant variability observed in inter-laboratory Karl Fischer test results for these lubricants highlighted the need for a standardized Karl Fischer test procedure, specifically suited for these lubricants.

2. APPLICABLE DOCUMENTS:

The following publications form a part of this document to the extent specified herein. The latest issue of SAE publications shall apply. The applicable issue of other publications shall be the issue in effect on the date of the purchase order. In the event of conflict between the text of this document and references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

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Documents

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2.1 ASTM Publications:

Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.

ASTM D 6304-98 Standard Test Method for the Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

ASTM D 6300-99 Standard Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

3. TEST REQUIREMENTS:

3.1 Outline of Method:

An accurately weighed aliquot of the fluid sample is injected into the titration vessel of a coulometric Karl Fischer apparatus in which iodine for the Karl Fischer reaction is generated coulometrically at the anode. Upon the completion of the titration of the water in the sample, the electrometric end point sensor detects excess iodine and the titration is terminated. The quantity of water is determined by the integration of the total amount of current consumed by the reaction, which, based on the stoichiometry of the reaction, is proportional to the molar concentration of water in the sample.

4. APPARATUS:

4.1 Coulometric Karl Fischer Apparatus (using electrometric end point):

A number of automated coulometric Karl Fischer apparatus consisting of a titration cell, platinum electrodes, magnetic stirrer and a control unit are commercially available. The instructions for the operation of these devices are provided by the respective manufacturers and are not discussed in this document.

4.2 Syringes:

Fine glass syringes with luer fittings and hypodermic needles are recommended. Disposable plastic precision syringes can be used for routine sample analysis but not for the validation. The bores of the needles shall be kept as small as possible, but large enough to avoid problems arising from excessive back pressure or blocking while obtaining a sample. Suggested syringe sizes are as follows:

- 4.2.1 Ten microliters fine glass syringe graduated for readings to the nearest 0.01 μl or better. This syringe is used in the standardization step.

- 4.2.2 One and five ml capacities, accurate to the nearest 0.01 ml. A quality gas-tight glass syringe with a TFE fluorocarbon plunger and luer fitting is recommended. Disposable plastic precision syringes can be used.

NOTE: Rinse all glass syringes and needles with anhydrous acetone (or equivalent) after cleaning. Then dry in an oven at 100 °C and store immediately in desiccators.

4.3 Balance:

An analytical balance with a resolution of 0.0001g.

5. REAGENTS/MATERIALS:

Reagent grade chemicals, conforming to the specifications of the Committee on Analytical Reagents of The American Chemical Society shall be utilized in all tests, unless indicated otherwise.

5.1 Karl Fischer Reagent:

Standard commercially available reagents for coulometric Karl Fischer titration.

5.2 Cathode Solution:

Standard, commercially available cathode Karl Fischer solution.

5.3 1-Octanol:

ACS grade, >99% pure, <1% mass percent of water, <0.4% mass percent of evaporation residue. (See Appendix A for the preparation of water saturated 1-Octanol.)

5.4 Water:

Reagent grade water as defined by Type IV of ASTM D 1193.

5.5 Xylene:

Reagent grade, <0.05% water.

5.6 Acetone:

Reagent grade, <0.05% water.

6. PREPARATION OF APPARATUS:

- 6.1 Follow the manufacturer's direction for the preparation and operation of the titration apparatus.

TABLE 1 - Test Sample Size Based on Water Concentration

Expected water concentration ($\mu\text{g/g}$)	Sample size (g)	Water titrated (μg)
100 to 500	2.0	200 to 1000
500 to 1000	1.0	500 to 1000
> 1000	0.5	> 500

- 6.2 Turn on the apparatus and start the magnetic stirrer for a smooth stirring action avoiding the formation of a vortex. Allow the residual moisture in the titration vessel to be titrated until the end point is reached and the background current (or background titration rate) is constant and less than the value recommended by the manufacturer.

NOTE: High background current for a prolonged period may be due to moisture on the inside wall of the titration vessel. Gentle swirling of the vessel will wash the inside with electrolyte. Keep the titrator on to allow stabilization to a low background current.

7. VALIDATION:

- 7.1 Validate the operation of the apparatus/reagent system by injecting a 10 μl sample of water saturated 1-octanol (see Appendix A) or 1000 μg or 1 μl of water in the titration vessel as outlined in 8.3.1 below for the test sample. Suggested intervals for the validation are each time the apparatus is turned on, and initially with fresh reagent.

- 7.1.1 The water concentration of the water saturated 1-Octanol should be within 47,300 $\mu\text{g/g} \pm 1000 \mu\text{g/g}$. The result of 1000 μg or 1 μl injection of water should be within 1000 $\mu\text{g} \pm 10 \mu\text{g}$.

NOTE: If the fluid being analyzed is suspected of interfering with the titration, a water-spiked sample of the fluid can be utilized for the validation as outlined in the Appendix A, Section A.9.

8. PROCEDURE:

In case of referee samples, requirements outlined in Appendix B are to be applied in addition to the following:

- 8.1 Examine the fluid sample for the presence of free water droplets or emulsified water as evidenced by a hazy to milky appearance of the fluid. If free water droplets are observed, withdraw sample for titration avoiding free water droplets as per 8.3.1. If emulsified water is observed, shake the sample bottle vigorously by hand followed by ultrasonication in a bath at high frequency for fifteen minutes, followed by shaking by hand before withdrawing a sample for titration as per 8.3.1.