



AEROSPACE RECOMMENDED PRACTICE

ARP5991™

REV. B

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Superseding ARP5991A

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

RATIONALE

The test method in ARP5991 is not likely to evolve or change, so the E-34 Committee has decided to stabilize the Aerospace Recommended Practice.

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1. SCOPE

The test method describes the procedure for the direct determination of water concentration in polyol ester and diester based aerospace lubricants by commercially available automated coulometric Karl Fischer titration instruments. 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 to assess the possibility of water corrosion of lubricant system components. The presence of excess amounts of water may also result in hydrolysis of the lubricant, resulting in corrosion and wear of the lubricated components and degradation of the lubricant properties. 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.

2.1 ASTM Publications

Available from ASTM International, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959, Tel: 610-832-9585, www.astm.org.

ASTM D6304 Standard Test Method for the Determination of Water in Petroleum Products, Lubricating Oils, and Additives by Coulometric Karl Fischer Titration

ASTM D6300 Standard Practice for Determination of Precision and Bias Data for Use in Test Methods for Petroleum Products and Lubricants

ASTM D1193 Standard Specification for Reagent Water

3. TEST REQUIREMENTS

3.1.1 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.

4.1.1 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:

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

4.1.3 One and five mL capacities, accurate to the nearest 0.01 mL. A quality gas-tight glass syringe with a PTFE 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 a desiccator.

4.2 Balance

An analytical balance with a resolution of 0.0001 g.

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.