

SEMI P8-90

METHOD FOR DETERMINATION OF WATER IN PHOTORESIST

1 Scope

1.1 This method is intended for the application of the Karl Fischer titration to the determination of water in photoresists. An electrometric method is used for determining the endpoint. The procedure is applicable to most resists that have polymer and solvent systems fully compatible with Karl Fischer reagent and diluent. The resist manufacturer should be consulted to determine if this procedure can be used with the resist in question.

2 Applicable Documents

2.1 ASTM Standard¹

ASTM E203 — Water by the Karl Fischer Method

2.2 Reagent Chemicals — Sixth Edition, American Chemical Society Specifications

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

3 Apparatus

3.1 Automatic titrator, equipped for Karl Fischer titrations. The titrator should be equipped with a titration vessel with a port for sample entry, desiccated vent tube, and a platinum versus platinum electrode. Mechanical stirring of the titration solvent should be provided for. The manufacturer's suggested operating procedure should be followed, and the endpoint timer set for 20 seconds.

3.2 Hypodermic syringes with lock tip, 0.25 mL and 10 mL.

3.3 Syringe needles, 5 to 8 cm length, 20 and 22 gauge.

3.4 Lint-free tissue.

3.5 Analytical balance, capable of weighing to 0.1 mg.

3.6 Reagents

3.6.1 Distilled water.

3.6.2 Methanol, ACS Reagent Grade.

3.6.3 Karl Fischer reagent with a titer of 3.0 (± 5.0) mg of water per ml of titrant. Typical preparation involves a one-to-one dilution of stabilized Karl Fischer reagent with Karl Fischer diluent from the same supplier.

4 Procedure

4.1 *Standardization of Karl Fischer Reagent* — The Karl Fischer reagent should be standardized immediately before its use in water determinations.

4.1.1 Install Karl Fischer reagent in the titrator and flush all lines of the titration equipment at least three times.

4.1.2 Fill the titration vessel with enough methanol to cover the electrodes, and seal.

4.1.3 Titrate the methanol automatically with the Karl Fischer reagent to the endpoint (blank).

4.1.4 Draw 0.1 mL of distilled water into a 0.25 mL syringe equipped with a 22 gauge needle. Draw the water from the needle into the syringe by inverting the syringe (needle up) and drawing the plunger downward an additional 1 to 2 cm.

4.1.5 Wipe the needle with lint-free tissue.

¹ ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA; Telephone: 610.832.9585, Fax: 610.832.9555, <http://www.astm.org>

- 4.1.6 Weigh the syringe assembly to the nearest 0.1 mg (W_1).
- 4.1.7 Inject the water into the titration vessel.
- 4.1.8 Titrate to the same endpoint used for the methanol blank.
- 4.1.9 Reweigh the syringe assembly to the nearest 0.1 mg (W_2).
- 4.1.10 Record the number of milliliters of titrant consumed (V_1).
- 4.1.11 Calculate the Karl Fischer factor (F):

$$F = \frac{\text{mg water injected}}{\text{mL KF titrant}} = \frac{W_1 - W_2}{V_1} \quad (1)$$

4.1.12 Repeat the standardizing titration twice and calculate the mean value for F from the three values (which should fall within a range of 0.02).

4.2 Determination of Water

- 4.2.1 Fill the titration vessel with enough methanol to cover the electrodes, and seal.
- 4.2.2 Titrate the methanol automatically with the Karl Fischer reagent to the endpoint (blank).
- 4.2.3 Flush a dry 10 mL syringe fitted with a 20 gauge needle several times with the sample to be tested. Fill the syringe with 3 to 5 mL of photoresist sample.
- 4.2.4 Wipe the needle with lint-free tissue.
- 4.2.5 Weigh the syringe assembly to the nearest 0.1 mg (W_3).
- 4.2.6 Inject the sample into the titration vessel.
- 4.2.7 Titrate to the same endpoint used for the methanol blank.
- 4.2.8 Reweigh the syringe assembly to the nearest 0.1 mg (W_4).
- 4.2.9 Record the number of milliliters of titrant used (V_2).
- 4.2.10 Calculate the water content of the sample.

$$\begin{aligned} \text{Weight \% H}_2\text{O} &= \frac{\text{mL KF titrant} \times F}{\text{sample weight in mg}} \times 100 \\ &= \frac{V_2 \times F}{W_3 - W_4} \times 100 \end{aligned} \quad (2)$$

4.3 *Remarks* — Photoresists which contain interfering compounds (such as aldehydes and ketones) may require modification of the solvent or titration conditions. For further information, see § 3.4 of ASTM E203. For some negative (polyisoprene) photoresists, solubility can be improved by substituting ethylene glycol as the solvent for methanol.

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