

SEMI P12-0997

DETERMINATION OF IRON, ZINC, CALCIUM, MAGNESIUM, COPPER, BORON, ALUMINUM, CHROMIUM, MANGANESE, AND NICKEL IN POSITIVE PHOTORESISTS BY INDUCTIVELY COUPLED PLASMA EMISSION SPECTROSCOPY (ICP)

This Standard was technically approved by the Micropatterning Global Technical Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee.

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1 Scope

1.1 This procedure is an ICP plasma emission analysis for determination of iron, zinc, calcium, magnesium, copper, boron, aluminum, chromium, manganese, and nickel in photoresist. The applicable concentration range is 0.1 to 1 ppm when the sample is diluted 1 to 4. The precision was found to be within 0.1 ppm in a round robin analysis between four laboratories.

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2 Spectrometer

2.1 A grating instrument with resolution sufficient to separate the analytical emission lines in Table 1 is required.

Table 1 Analytical Lines of the Elements

<i>Elements</i>	<i>Analytical Line, nm</i>	<i>Elements</i>	<i>Analytical Line, nm</i>
Aluminum	309.271	Magnesium	285.213
Calcium	317.933	Nickel	231.604
Copper	324.754	Zinc	213.856
Iron	239.562	Chromium	283.563
Boron	208.960	Manganese	257.610

3 Sample Preparation

3.1 The sample is diluted $\frac{1}{4}$ (1 part of sample plus 3 parts of solvent weight/weight) in 2-methoxyethanol or another suitable solvent for positive resist. The solvent should contain less than 0.03 ppm of the above elements.

4 Standards

4.1 The standards should be weight/weight (mg/kg) and should be diluted weight/weight since results will be expressed in mg/kg.

4.2 The standards are prepared by diluting a concentrated standard of organic-soluble metals in 2-methoxyethanol or other suitable solvent. For example, a 500 ppm standard is diluted to 50 ppm with xylene. This solution is then diluted to 0.25 ppm with 2-methoxyethanol.

5 Plasma Conditions

5.1 The sample is pumped on the region of 0.7 mL/min., usually with a peristaltic pump attached to the nebulizer. Choose a tubing that is not attacked by methyl cellosolve (for example, polytetrafluoro-ethylene). The argon plasma flow rate and RF power should be optimized for the sample using settings recommended in the manufacturer's manual.

Generally, a plasma gas flow rate of 16 l/min. is necessary to ionize organic solutions and an RF power of 1.7 watts is required.

6 Quantitation

6.1 The detector gain is set by measuring the 0.25 ppm standard. The background is measured with the solvent and several sample measures. The standard and blank should be run intermittently to satisfy reasonable precision. Standard and sample readings should be repeatable within 0.03 ppm. The effect of sample viscosity on delivery of diluted resist to the plasma was not found to be a factor for the resist tested for this procedure. This effect can be checked by adding an internal standard of an element known not to be present in the resist (such as Yttrium) at ppm and checking the emission response vs. external 1 ppm Yttrium standard in the diluting solvent.

7 Calculation

7.1 ppm element (mg/kg = ppm measured $\times$ delution factor (weight/weight)

7.2 *Detection Limit* — The detection limit is a function of the dilution factor and can vary by instrument. The dilution factor should be taken into account in calculation of detection limit.

7.2.1 Detection limit (ppm) = $s \times$ dilution factor where s = standard deviation of instrument readings in ppm.

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