

SEMI P13-91 (Reapproved 1104)

DETERMINATION OF SODIUM AND POTASSIUM IN POSITIVE PHOTORESISTS BY ATOMIC ABSORPTION SPECTROSCOPY

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1 Scope

1.1 This procedure is a flame atomic absorption analytical method for sodium and potassium analysis in photoresist. The applicable concentration range is 0.1 to 1 ppm when the sample is diluted one to four. The precision was found to be within 0.1 ppm in a round-robin analysis between four laboratories.

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2 Referenced Standards and Documents

2.1 None.

3 Terminology

3.1 Definitions

3.1.1 *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.

4 Atomic Absorption Spectrometer

4.1 The analytical wavelength for sodium is 589.0 nm and for potassium is 766.0 nm. The instrument conditions (i.e., slit width, burner gas flow rates) should be set according to the manufacturer's manual (the fuel flow should be set to approximately ½ the air flow to optimize for organic solvent). Optimize the nebulizer and lamp alignment to maximize the absorbance of the 1 ppm standard.

5 Sample Preparation

5.1 The sample should be diluted ¼ (1 part resist and 3 parts of solvents weight/weight) in 2-methoxyethanol or another suitable solvent for positive resist. The solvent should contain less than 0.1 ppm of the element being analyzed.

6 Standards

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

6.2 The standards are prepared by diluting a concentrated stock of organic-soluble sodium and potassium. For example, a 500 ppm standard of sodium is diluted to 50 ppm with reagent xylene. This solution is then diluted to 1 ppm with 2-methoxyethanol or another suitable solvent.

7 Procedure

7.1 Set the absorbance reading to zero with the flame ignited and no sample aspirating. Measure the absorbance of the solvent blank. It should not be more than 0.04. Measure the absorbance of the 1 ppm standard and samples. The absorbance of the standard should be in the region of 0.4. Duplicate sample and standard readings should be within 0.01 absorbance. A recovery of 88% was determined when a 1 ppm potassium internal standard was added to the resist tested. The effect of viscosity on delivery to the burner can be determined by adding 2 ppm of a potassium

internal standard to the sample and checking the absorbance versus an external 2 ppm potassium standard in the diluting solvent. The potassium level in the resist, if any, should be subtracted.

8 Calculation

$$\text{ppm (mg/kg)} = \frac{\text{Abs. Sample} \times \text{ppm Standard} \times \text{dilution factor} \times R}{\text{Abs. Standard}}$$

R = Recovery factor due to sample viscosity if applicable, for example 1.136 in above example.

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

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