Determination of trace elements in isopropyl alcohol using an Agilent 4200 MP-AES with External Gas Control Module

Application note

Semiconductor and petrochemical

Authors

Kim Young-Jin
Agilent Technologies
South Korea

Introduction

Isopropyl alcohol (IPA) is one of the most commonly used organic solvents in the semiconductor industry. Silicon wafers are dried after surface cleaning in the manufacturing process and IPA is frequently used during the drying step. The purity of IPA used is critical since these materials come in direct contact with the silicon wafers and any contaminants present at this stage could be detrimental to the overall performance of the final product.

Plasma-based instrumentation is widely used in the semiconductor industry due to its multielement capability and sensitivity compared to techniques such as Flame Atomic Absorption Spectroscopy (FAAS). However a robust plasma is required for the analysis of organic matrices. Microwave Plasma Atomic Emission Spectroscopy (MP-AES) uses magnetically-coupled microwave energy to generate a stable plasma that is highly suited to the analysis of organic solvents. The Agilent 4200 MP-AES features an advanced microwave cavity and a torch designed to handle a wide range of samples, with better detection limits and an increased working range, compared to FAAS. Major elements (Ca, K, Na and Mg) in IPA can be easily determined using MP-AES with little sample preparation.

The nitrogen used to generate the 4200's plasma can be generated from air using a nitrogen generator. This eliminates the need for the hazardous or expensive gases required by other techniques, reduces running costs and greatly improves laboratory safety. The instrument is relatively simple to use, even for inexperienced users. It has a plug-and-play torch and intuitive MP Expert software that facilitates instrument setup and method development with minimal training.

This application describes the direct determination of trace elements in undiluted, high purity IPA using the Agilent 4200 MP-AES fitted with an External Gas Control Module (EGCM).

Experimental

Instrumentation

Figure 1. External Gas Control Module (EGCM) accessory for the Agilent 4200 MP-AES

All measurements were performed using an Agilent 4200 MP-AES. In order to analyze Na, K, Mg, and Ca in IPA directly, without dilution, the instrument was fitted with an Agilent Organics Kit comprising the EGCM, inert OneNeb nebulizer, solvent resistant tubing, and a double pass spray chamber.

The EGCM (shown in Figure 1) injects air into the plasma. This prevents carbon deposition in the torch, overcomes any plasma instability that may arise from the analysis of organic samples, and reduces carbon based background emissions. The EGCM setting is selected automatically for each wavelength by the MP Expert software, but can be fine-tuned if required.

The OneNeb inert nebulizer is ideally suited to the analysis of organic solvents such as IPA. It offers increased nebulization efficiency and a narrow distribution of small droplets compared to other nebulizers.

The 4200 MP-AES operates in fast sequential mode and has a peltier cooled charge coupled device (CCD) detector which allows background and spectral interferences to be simultaneously corrected easily and accurately using the MP Expert software. Auto background correction mode was used to correct for any emission background arising from the organic matrix.

Instrument operating parameters and method conditions are listed in Tables 1 and 2.

Table 1. Agilent 4200 MP-AES operating conditions

Parameter	Value
Sample tubing	Orange/green solvent resistant
Waste tubing	Blue/blue solvent resistant
Read time (s)	3
Number of replicates	3
Sample uptake delay (s)	10
Stabilization delay (s)	10
Rinse time (s)	10 (fast pump: On)
Pump speed (rpm)	7

Table 2. MP-AES method conditions: list of analytes with wavelength, background correction, nebulizer flow and automatically-set EGCM settings.

Element	Wavelength (nm)	Background Correction	Nebulizer Flow (L/min)	EGCM Setting
Na	588.995	Auto	0.6	High
K	766.491	Auto	0.6	High
Mg	285.213	Auto	0.6	Low
Ca	393.366	Auto	0.6	Medium

Samples and sample preparation

Electronic grade IPA (99.9% purity) purchased from Dongwoo Fine-Chem Co., Ltd., South Korea was analyzed directly, without any sample preparation. IPA was spiked with a multi-element standard at 106.5 ppb to test the stability of the method.

Results and discussion

Linear Dynamic Range

The linear dynamic range (LDR) for Na, K, Mg, and Ca was determined by calibrating against a set of standards at 10 ppb, 50 ppb, 100 ppb and 200 ppb. All elements showed excellent linearity over the calibrated range with a correlation coefficient of >0.999 in all cases. The calibration curves for Na, K, Mg, and Ca are shown in Figure 2

Methods Detection Limits

Method Detection Limits (MDLs) were determined by analyzing 7 samples at a concentration of 106.5 ppb. For seven samples (with six degrees of freedom), the t value for a 99% confidence interval is 3.14. The MDLs were found to be under 5 ppb for all elements. Results are shown in Table 3.

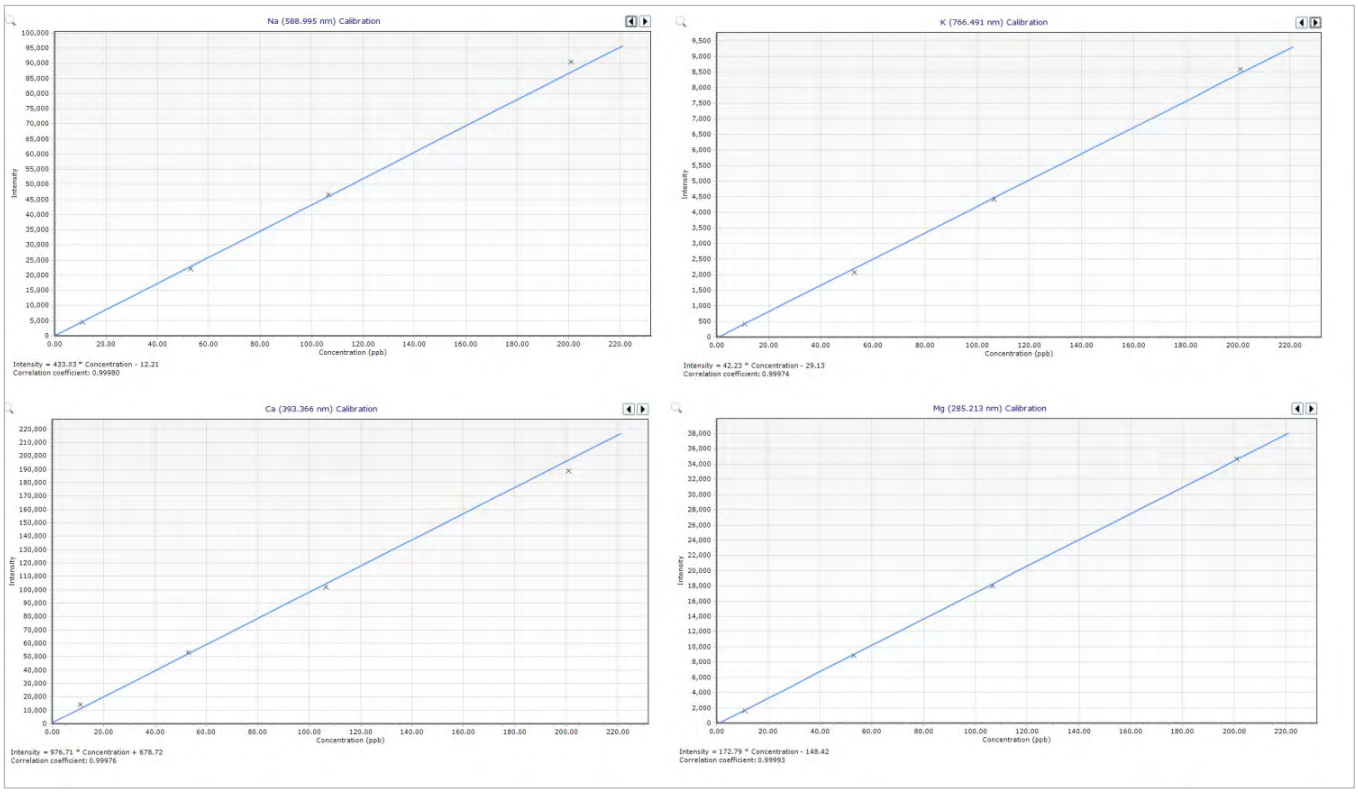

Figure 2. Calibration curves for Na 588.995, K 766.491, Ca 393.366 andMg 285.213

Spike recoveries

To test the method, a spike of 106.5 ppb was analyzed repeatedly over more than 3 hours. The average spike recovery for each element over the duration of the experiment was found to be within 97% to 102%. Spike recoveries are shown in Table 3.

Table 3. MDL, recovery of 106.5 ppb spike, and long term stability for Na, K, Mg, and Ca in IPA.

Element (ppb)	Wavelength (nm)	MDL (ppb)	Spike Recovery (%)	Long Term Stability (% RSD)
Na	588.995	2.5	100	1.4
K	766.491	3.0	97	1.4
Mg	285.213	2.8	102	1.6
Ca	393.366	3.8	97	0.9

Long term stability

The long term stability was measured for Na, K, Mg, and Ca spiked at 106.5 ppb in IPA. The stability plot is shown in Figure 3 and the %RSD for each element is given in Table 3. Excellent stability was achieved with %RSD of less than 2% over more than 3 hours.

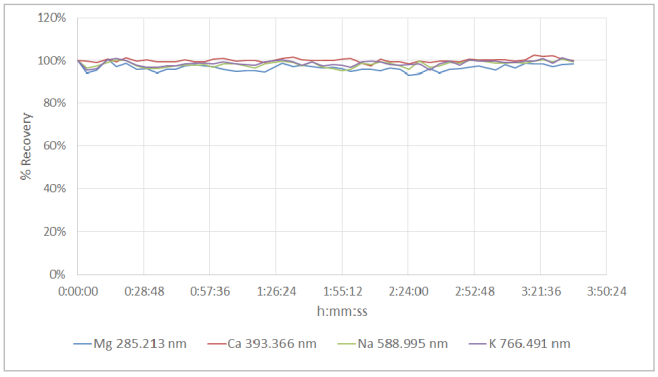

Figure 3. Long term stability plot for Na, K, Mg, and Ca in IPA.

Conclusions

The Agilent 4200 MP-AES fitted with the EGCM accessory was successfully used to determine ppb levels of Na, K, Mg, and Ca in undiluted isopropyl alcohol. With method detection limits of less than 5 ppb for all elements, excellent spike recoveries within the range 97% to 102%, and long term stability of less than 2% RSD, the method is highly suited to the routine analysis of these 4 key elements in IPA.

The 4200 MP-AES offers:

- Improved performance and sample throughput compared to FAAS
- Ease-of-use through the MP Expert software and plug-and-play hardware that simplify method development and torch alignment, with minimal operator training
- Improved safety by eliminating hazardous or expensive gases, such as acetylene or argon
- Productivity gains with safe, unattended operation.

www.agilent.com

Published July 17, 2015
Publication number: 5991-6021EN

Agilent Technologies