

## Determination of Trace Elements in IPA by ICP-DRC-MS

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At the ICP-MS measurement, organic solvent such as isopropyl alcohol (IPA) is known to generate polyatomic interference such as  $^{12}\text{C}_2$  on  $^{24}\text{Mg}$ ,  $^{40}\text{Ar}^{12}\text{C}$  on  $^{52}\text{Cr}$  and  $^{12}\text{C}^{14}\text{N}^1\text{H}$  on  $^{27}\text{Al}$ . The ICP-DRC (Dynamic Reaction Cell)-MS is possible to remove polyatomic ions that were generated by argon, sample matrix etc. The conventional cool plasma needs to run 2 methods to a single sample, because the hot plasma is essential for W, Zn, U and others. On the other hand, ICP-DRC-MS is possible to determine all elements under only one method and plasma condition. In this study, under the conditions such as optimum cell gas flow rate, ICP-DRC-MS performed successfully the analysis of typical organic solvent of IPA. The detection limits for Mg, Cr and Al were 6, 4 and 6 ng/L (ppt), respectively.

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Manufacturing processes of semiconductor devices require a variety of chemicals which must be ultra pure because the presence of impurities degrades the performance of the resultant devices. The Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) has been used for the determination of impurity levels in these chemicals because of its ability to perform low-level analyses.

However, it has been considered the analysis of organic solvent by ICP-MS is quite tough. One of the reasons is polyatomic ions; the carbon related polyatomic interferences are generated such as  $^{12}\text{C}_2$  on  $^{24}\text{Mg}$ ,  $^{40}\text{Ar}^{12}\text{C}$  on  $^{52}\text{Cr}$  and  $^{12}\text{C}^{14}\text{N}^1\text{H}$  on  $^{27}\text{Al}$ . As a result, the determination of these ions is difficult.

On the other hand, the ICP-DRC (Dynamic Reaction Cell<sup>TM</sup>)-MS<sup>1-5</sup> has quite unique feature to decrease the polyatomic ions. By providing a low flow of the proper reaction gas into the patented DRC and using the unique Dynamic Bandpass Tuning (DBT) feature, interferences can be chemically scrubbed out of the ion beam before it enters the analyzer quadrupole of the mass spectrometer.

In this paper, we report that the trace elements were determined in IPA (isopropyl alcohol) by optimizing the cell gas flow rate, RPa, RPq, etc. It was also possible to remove the polyatomic interference generated from carbon, argon, nitrogen, oxygen, hydrogen, etc.

### Experimental

#### Instrumentation

An ELAN DRC ICP-DRC-MS instrument (PerkinElmer SCIEX, Ontario, Canada) was used for the measurement of elements in the concerned samples.

The DRC was positioned between the ion lens and the quadrupole mass analyzer (Fig. 1).

#### Sample preparation and introduction

High purity IPA (isopropyl alcohol) was used as sample. Calibration standards were prepared from multielement PE Pure Plus (PerkinElmer, Shelton, CT).

The sample introduction system of the instrument was consisted of PFA micro flow nebulizer (sample uptake:

100-200  $\mu\text{L}/\text{min}$ ), quartz cyclonic spray chamber and quartz injector tube.

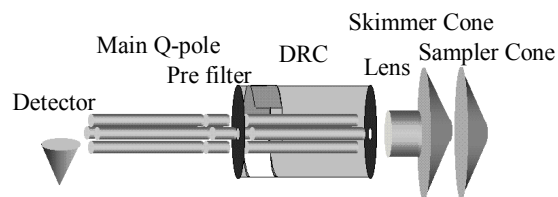


Fig. 1 Diagram of ELAN DRC.

### Results and Discussion

#### Optimization of the cell gas flow rate

When DRC was switched off (cell gas flow rate being set as 0 mL/min), background intensity of Cr ( $m/z=52$ ) was about  $1 \times 10^7$  counts/second. Similarly, the background intensity of Mg ( $m/z=24$ ) was about  $1 \times 10^6$  counts/second. However, these were not Cr and Mg signals, but the signals from polyatomic ions; the carbon related polyatomic interferences are generated such as  $^{12}\text{C}_2$  on  $^{24}\text{Mg}$ ,  $^{40}\text{Ar}^{12}\text{C}$  on  $^{52}\text{Cr}$  and  $^{12}\text{C}^{14}\text{N}^1\text{H}$  on  $^{27}\text{Al}$ . As a result, the determination of these ions was difficult. By providing a low flow of the proper reaction gas into the patented DRC and using the unique Dynamic Bandpass Tuning (DBT) feature, interferences can be chemically scrubbed out of the ion beam before it enters the analyzer quadrupole of the mass spectrometer. For example, when cell gas flow rate was 0.6 mL/min, the background intensity became less than 20. And the system could clearly differentiate between the intensity of blank and 500 ppt standard solution (Figs. 2 and 3). According to Figs. 2 and 3, the cell gas flow rate was selected 0.6 mL/min, because signal to background ratio was the maximum in the range of this cell gas flow rate.

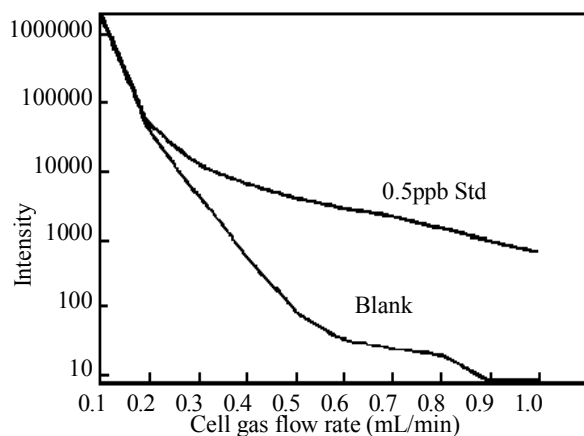


Fig.2 Effect of cell gas flow rate (Mg).

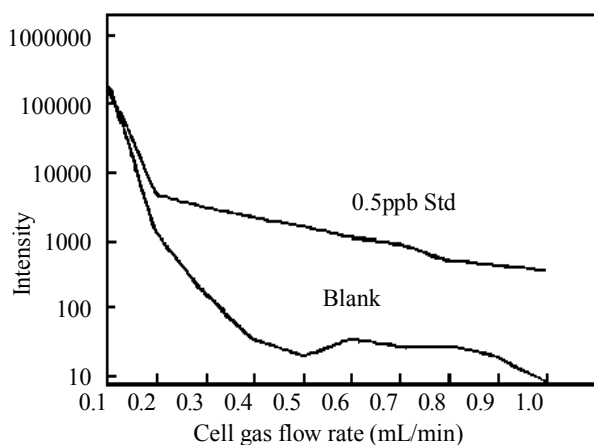


Fig. 3 Effect of cell gas flow rate (Cr).

#### Plasma conditions

Table 1 shows the plasma conditions. Especially, the hot plasma was used, it was RF power of 1500 W. Hot plasma conditions were better than cool plasma with respect to influence by matrix interference. ICP-DRC-MS was possible to determine all of the elements under only one method and plasma condition.

Oxygen was introduced for preventing to adhesion the carbon.

Table 1 Instruments parameters

Plasma parameter	
RF power (W)	1500
Plasma gas flow rate (L/min)	15
Auxiliary gas flow rate (L/min)	1.2
Nebulizer gas flow rate (L/min)	0.90
Sample uptake (mL/min)	0.1
Oxygen gas flow rate (mL/min)	40
DRC parameter	
RPa	0
RPq	0.5
NH <sub>3</sub> gas flow rate (mL/min)	0.6

RPa: DC voltage at reaction cell quadrupole.

RPq: RF voltage at reaction cell quadrupole.

#### Calibration curves

For example, the calibration curves for IPA are shown in the Figs. 4 and 5. These calibration curves are not corrected background intensity. Thus, the very low BEC (background equivalence concentration) was obtained.

#### Detection limits and background intensity for IPA

Table 2 summarizes the detection limits of IPA by 3 sigma for 10 replicate measurements. These results show very low detection limit and BEC. It should be noted that these results were obtained by only one method for all of the elements measurement. The conventional cool plasma must use 2 methods, because W, Zn and U need hot plasma.

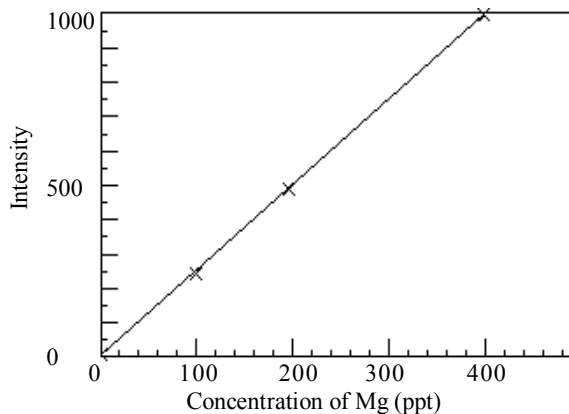


Fig.4 Calibration curve of Mg in IPA.

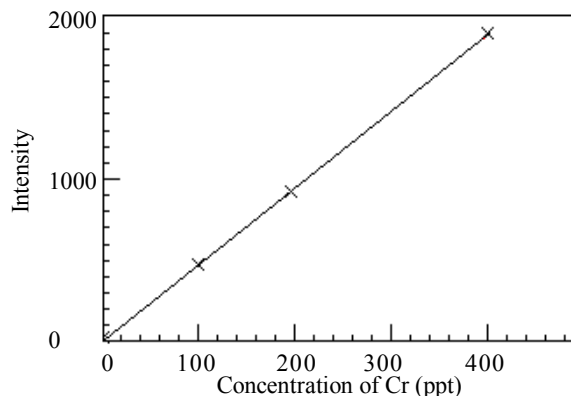


Fig. 5 Calibration curve of Cr in IPA.

Table 2 Detection limits and background intensity for IPA

Element	D.L. (ng/L)	NH <sub>3</sub> (mL/min)	Interference	BKG	
				ON	OFF
<sup>24</sup> Mg	5.6	0.6	<sup>12</sup> C <sup>12</sup> C	20	1x10 <sup>6</sup>
<sup>27</sup> Al	5.9	0.6	<sup>12</sup> C <sup>14</sup> N <sup>1</sup> H	10	1x10 <sup>5</sup>
<sup>52</sup> Cr	3.6	0.6	<sup>40</sup> Ar <sup>12</sup> C	20	1x10 <sup>7</sup>
<sup>56</sup> Fe	1.8	0.6	<sup>40</sup> Ar <sup>16</sup> O	10	1x10 <sup>7</sup>
<sup>184</sup> W	0.8	0	-	-	5
<sup>208</sup> Pb	0.3	0	-	-	10
<sup>238</sup> U	0.1	0	-	-	5

D.L.: Detection limits (3 sigma)

NH<sub>3</sub>: Cell gas flow rate

ON: DRC mode

OFF: Standard mode (cell gas flow rate: 0 mL/min)

BKG: Background intensity

#### Long terms stability

The long terms stability of intensity were evaluated by the recommended condition from 0 to 2 hours (Fig. 6). It achieved a good stability for all of the elements (RSD <2%).

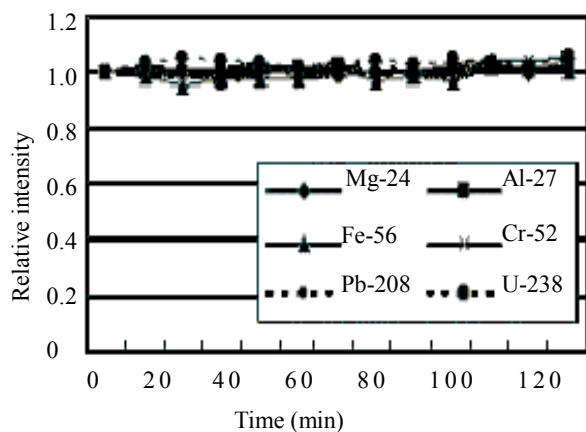


Fig. 6 Long terms stability.

### Conclusion

This presented paper, determination conditions and analytical sensitivity of ICP-DRC-MS were investigated, the results show ICP-DRC-MS method was suitable to determine trace elements in IPA.

1. ICP-DRC-MS was possible to determine all elements under only one method and plasma parameter.
2. ICP-DRC-MS was possible to remove polyatomic interference generated by argon, carbon, etc.
3. The detection limits for Mg, Cr and Al were 6, 4 and 6 ng/L (ppt), respectively.
4. The long terms stability was less than RSD 2% in 2 hours.

### References

1. V. I. Baranov, and S. D. Tanner, *J. Anal. At. Spectrum.*, **1999**, *14*, 1133.
2. S. D. Tanner, and V. I. Baranov, *American Society for Spectrometry*, **1999**, 1083.
3. S. D. Tanner, V. I. Baranov, and U. Vollkopf, *J. Anal. At. Spectrum*, **2000**, *15*, 1261.
4. J. J. Sloth, and E. H. Larsen, *J. Anal. At. Spectrum*, **2000**, *15*, 669.
5. D. R. Bandura, V. I. Baranov, and S. D. Tanner, *J. Anal. At. Spectrum*, **2000**, *15*, 921.