MEASUREMENT OF HIGH PURITY METALS USING THE NU ASTRUM GD-MS: THE EFFECT OF SAMPLE-CELL COOLING

INTRODUCTION

There are few analytical techniques capable of quantifying trace impurities in ultra-pure metals. One technique that has gained acceptance and is widely used in the industry is glow discharge mass spectrometry (GD-MS). Instruments of this kind require extremely low background levels. However, this can be difficult to achieve and therefore the design of the instrument is critical, particularly with regard to the glow discharge cell region. One method that can be used to reduce the presence of molecular species is to cool the glow discharge cell region using either a Peltier cooling system or else liquid nitrogen cooling. In this note, we compare the residual background measured on a Nu Astrum GD-MS for a number of elements using different glow discharge cell temperatures.

Instrumentation

The Nu Astrum is the latest generation of GD-MS instruments developed to be the benchmark in GD-MS. The instrument was designed in conjunction with the users of the most widely-used GD-MS, the VG9000. The best design concepts of the VG9000 were combined with advances in sample cell design, control electronics and pumping technology to produce a high performance new instrument designed for ultra-trace analysis of solids.

Experiment

In order to study the effect that glow discharge cell cooling has on instrumental background, we acquired mass scans at different m/z values using three different cooling regimes: (i) uncooled, (ii) liquid nitrogen cooled to approximately -35°C (to emulate Peltier cooling) and (iii) liquid nitrogen cooled to <-150°C. The sample was 7N+ copper, prepared as a pin sample. The Cu ion beam current was ~1 x 10^{-9} A and the glow discharge current was ~2.5mA. For low beam currents the electron multiplier was used. However, larger beam currents would saturate the electron multiplier and in these cases the Faraday detector was used. The electron multiplier efficiency was ~80%, determined by detector calibration performed before and after the measurements. All measurements were made at approximately 4,000 resolution.

Discussion

The three plots in figure 1 show the effect that cell cooling has at m/z 117 (tin). There is a large peak present when the cell is un-cooled. However, due to the purity of the sample it is not likely to be ^{117}Sn. We believe this interference to be ^{65}Cu + ^{40}Ar + ^{12}C. When the sample cell is cooled to typical Peltier-cooling temperatures, this molecular species is reduced by an order of magnitude but is still sufficiently high to cause isobaric interference with the ^{117}Sn peak. However, cooling to < -150°C almost totally eliminates this molecular species, allowing an unambiguous measurement of ^{117}Sn.

Figures 2 to 5 show measurements for other m/z values. A general observation is that without any cell cooling, there are isobaric molecular species that can prevent accurate measurement of trace elements. Cooling to ~ -35°C reduces those molecular species but they may still be present at levels high enough to interfere with the trace elements. However, cooling the glow discharge cell to < -150°C has a dramatic effect and reduces many of the molecular species to a level whereby they do not cause interference with the trace elements.

Figure 2 details the effect that cooling has on ^{12}C, which is present due to atmospheric gases. Note that the triply-charged ^{36}Ar peak is unaffected by the cooling, although it cannot be clearly observed in the first plot as this plot was measured using the Faraday detector. In figure 3, the effect that cell cooling has on ^{16}O is shown. There is a reduction in ^{16}O ion signal of about 10^4 when comparing the un-cooled glow discharge cell to the cell cooled to < -150°C

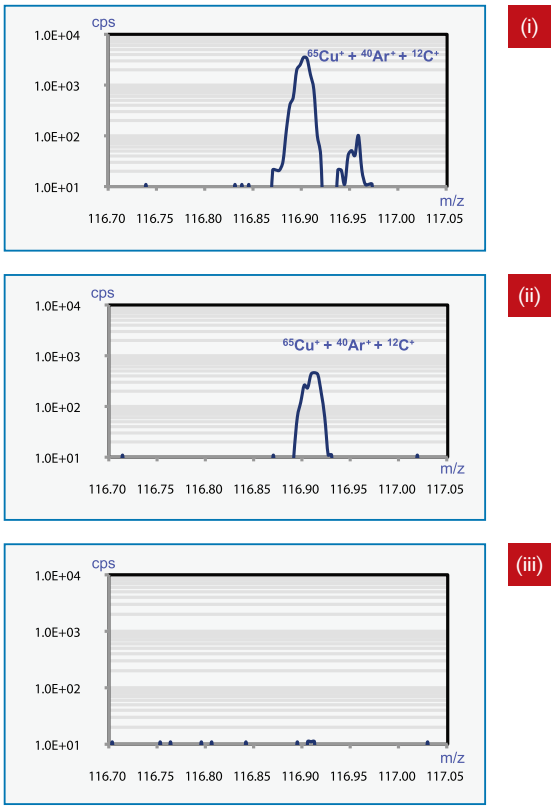

Figure 1: *mass scans for tin with the sample cell (i) uncooled, (ii) cooled to ~ -35°C, and (iii) cooled to ~ -150°C. The peak corresponds to $^{65}Cu^+ + {}^{40}Ar^+ + {}^{12}C^+$*

Conclusions

The Nu Astrum is a new high performance GD-MS that has been tailored to the exacting requirements of the high purity materials industry. We have shown that without sample-cell cooling there are numerous molecular species, which can be somewhat reduced by cooling of the glow discharge cell to ~ -35°C (to emulate Peltier cooling). However, liquid nitrogen cooling to <-150°C has a far more significant effect at reducing the background of these molecular species and therefore offers a tangible performance advantage.

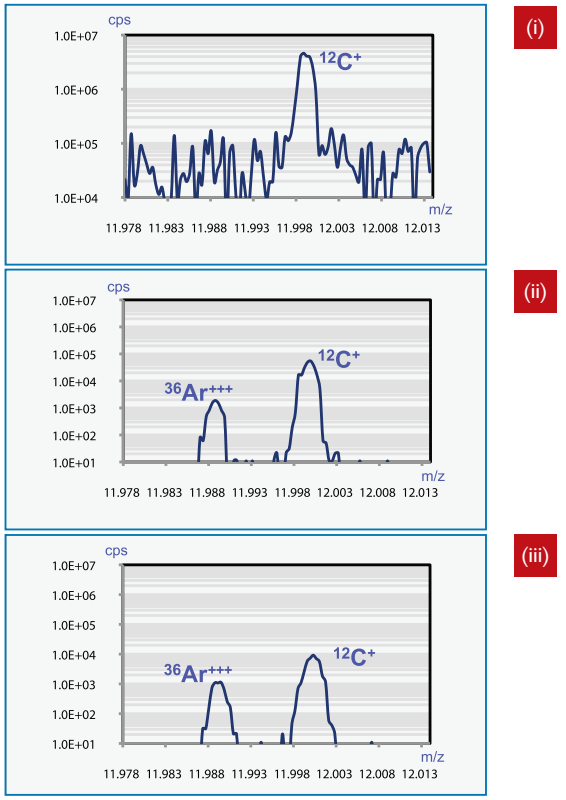

Figure 2: *mass scans for carbon with the sample cell (i) uncooled, (ii) cooled to ~ -35°C, and (iii) cooled to ~ -150°C*

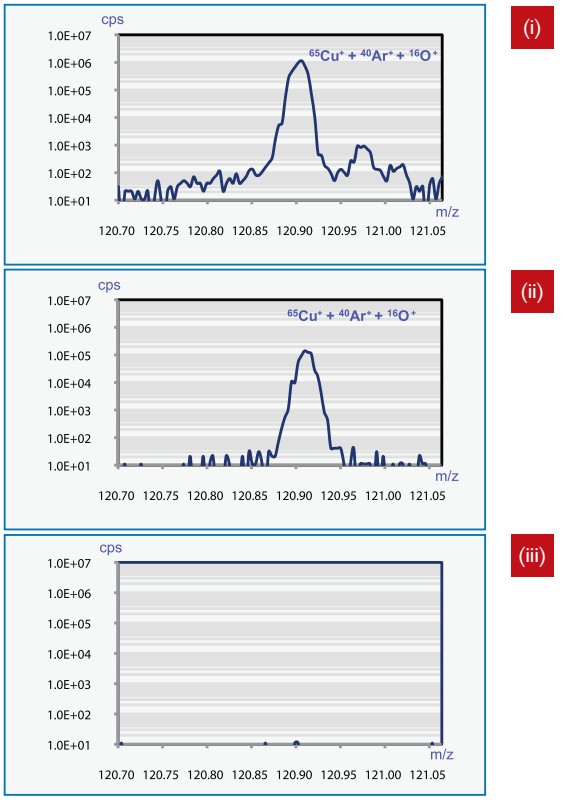

Figure 4: *mass scans for antimony with the sample cell (i) uncooled, (ii) cooled to ~ -35°C, and (iii) cooled to ~ -150°C. The peak corresponds to $^{65}Cu^+ + {}^{40}Ar^+ + {}^{16}O^+$*

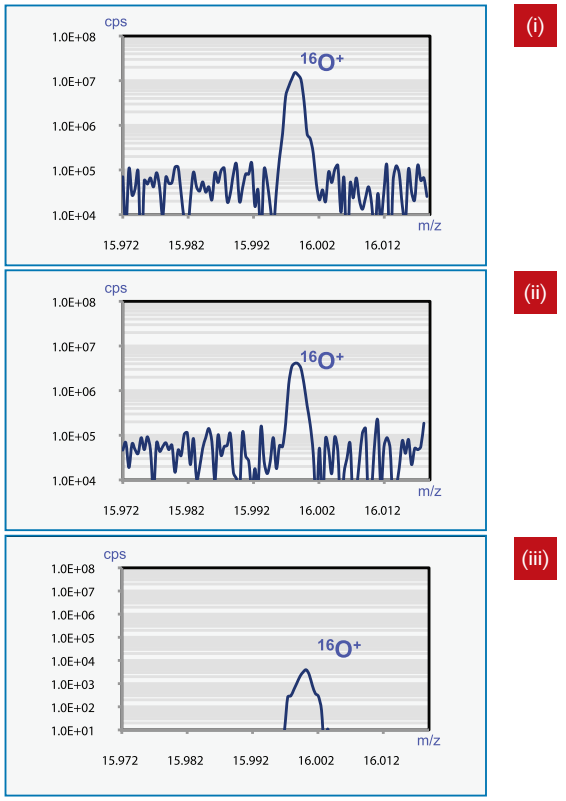

Figure 3: *mass scans for oxygen with the sample cell (i) uncooled, (ii) cooled to ~ -35°C, and (iii) cooled to ~ -150°C*

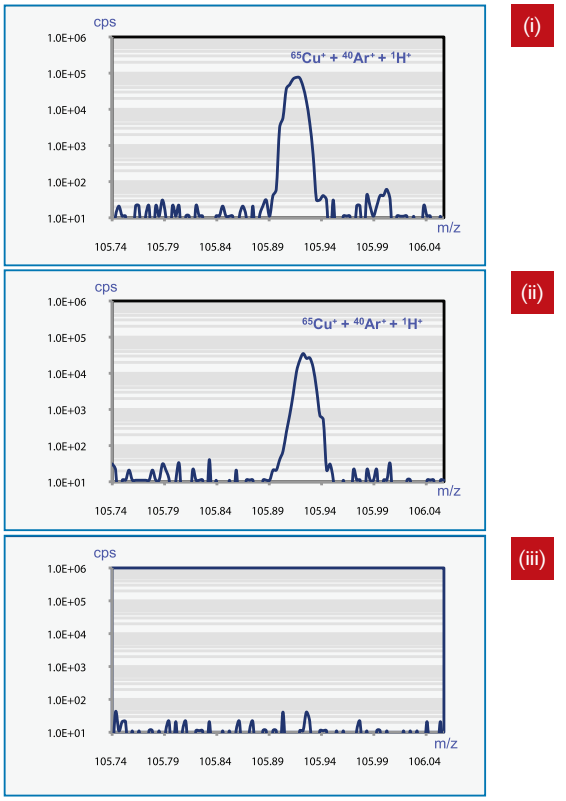

Figure 5: *mass scans for palladium with the sample cell (i) uncooled, (ii) cooled to ~ -35°C, and (iii) cooled to ~ -150°C. The peak corresponds to $^{65}Cu^+ + {}^{40}Ar^+ + {}^{1}H^+$*

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