APPLICATION NOTE

ICP - Mass Spectrometry


**AUTHORS**

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# Elemental Analysis of Wines using the NexION 2000 ICP-MS with TotalQuant

## Introduction

In recent years, food alteration has increased substantially, leading to substandard products. Therefore, to ensure the health and safety of the consumer, it is essential to characterize foods for toxic and nutritional elements accurately and rapidly.

Inductively coupled plasma mass spectrometry (ICP-MS) is the ideal technique for multi-elemental analyses due to its sensitivity and ability to measure trace and matrix elements in one quick analytical run.

In this work, a NexION® 2000 ICP-MS was used for the determination of toxic elements in wine. TotalQuant, a feature in Syngistix™ software unique to NexION ICP-MS systems, offers the capability to quantify multiple elements in a sample via the interpretation of the complete mass spectrum. Measuring the full mass range takes only a couple of minutes and the spectral interpretation itself takes a few seconds. In addition to being an ideal tool for semi-quantitative analysis during method development, TotalQuant can also be used for final material characterization, offering a rapid, convenient, and easy way to evaluate the levels of contaminants and essential elements in food and other matrices.

TotalQuant not only measures the concentration of many elements, but can also be used as a tool for fingerprinting and profiling wines based on their elemental composition. Such analysis can assign the geographical origins of unknown wines and highlights adulterated and fraudulent samples.

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## Experimental

### Instrumentation

The NexION 2000 represents a truly significant advancement in ICP-MS and in the removal of spectral interferences. It offers both the simplicity and convenience of a traditional collision cell with kinetic energy discrimination (KED) and the recognized superior detection limits of a dynamic reaction cell (DRC). With the patented Universal Cell Technology (UCT) and three gas channels, analysts can choose the most appropriate mode for analysis: Standard, Collision (KED) or Reaction (DRC). Instrumental parameters utilized in TotalQuant analysis of wines are listed in Table 1.

*Table 1: Instrumental Parameters.*

| Component/Parameter | Type/Value |
|---|---|
| Instrument | NexION 2000 ICP-MS |
| Nebulizer | PFA-ST |
| Sample uptake rate | 0.2 mL/min |
| Spray chamber | Cyclonic, quartz |
| Torch | One-piece quartz with 2 mm injector |
| RF power | 1600 W |
| Cell gas | Helium |

### Analytical Method

Analysis of wines was done using the TotalQuant (TQ) method. During the analysis, the TQ measures the whole mass spectrum (all isotopes) and then each element is assigned a response value which is updated when single-standard calibration is performed. TotalQuant analysis is usually done in Collision (KED) mode which automatically corrects for many spectral polyatomic interferences. Figure 1 shows the method used in this application.

### Samples and Sample Preparation

Fourteen red wines, mainly Merlot and a couple of Red Blends, were analyzed. Seven wines came from the US (California, Napa Valley region), and the other seven from South and Southeastern Australia (Figure 2). These wines were made from similar grapes, but due to different soil compositions, the method of processing, and environmental contamination, the elemental composition is expected to be different.

All samples were diluted 10 times with 1% $HNO_3$. A blank and standard were prepared in 1% $HNO_3$ with the addition of 1.5% ethanol to match the alcohol content in the wines.

| | Begin Mass (amu) | End Mass (amu) | Scan Mode (*) | MCA Channels | Dwell Time per AMU (ms) | Integration Time (ms) | Profile (*) | Ammonia Flow | Helium Flow | Oxygen Flow | RP a | RP q |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 6 | 40 | Peak Hopping | 1 | 50 | 17500 | Helium KED | 0 | 4.5 | 0 | 0 | 0.25 |
| 2 | 42 | 210 | Peak Hopping | 1 | 50 | 84500 | Helium KED | 0 | 4.5 | 0 | 0 | 0.25 |
| 3 | 230 | 240 | Peak Hopping | 1 | 50 | 5500 | Helium KED | 0 | 4.5 | 0 | 0 | 0.25 |

*Figure 1: TotalQuant method used for wine analysis.*

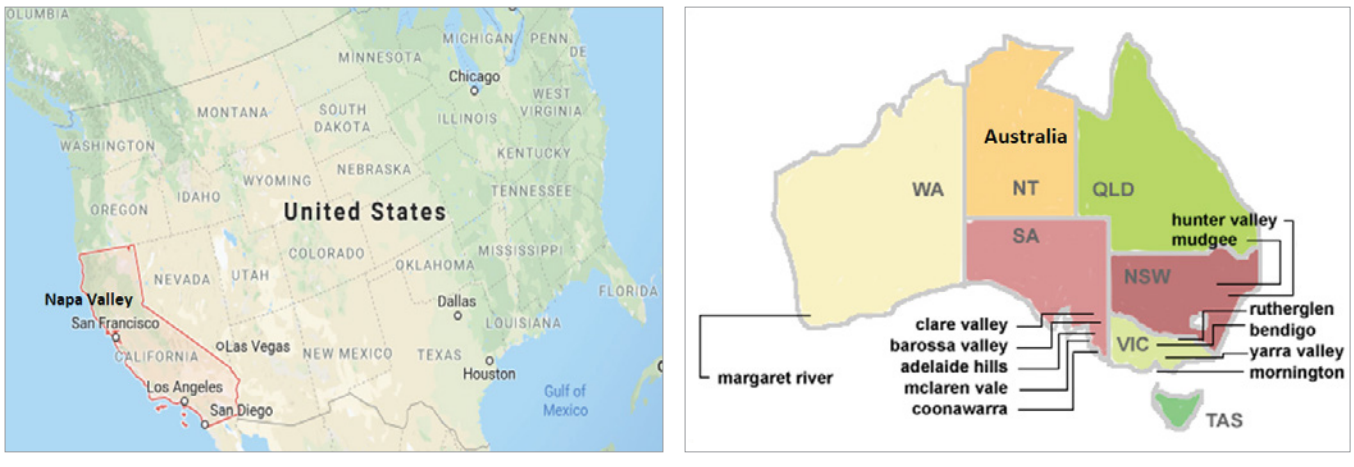


*Figure 2: Areas in the US and Australia from where analyzed wines originated.*

## Results and Discussion

TotalQuant analysis provided results for 81 elements, but elements with concentrations on average below 0.1 µg/L were omitted, so only 51 elements were represented in the plots. Elemental concentrations for seven wines from the US Napa Valley and Southeastern Australia are shown in Figures 3 and 4 respectively.

The fingerprint of wines based on 51 elements from Napa Valley and Australia looks similar and it is difficult to pinpoint any differences. Therefore, results for each element in seven wines for each region were averaged and results were plotted as % difference in concentration between US Napa Valley (positive %) and Australian (negative %) reds. Using this approach, a variance for some elements, such as Na, Cu, Br, Sr, and rare earth elements (REEs) in Australian wines is quite visible (Figure 5).

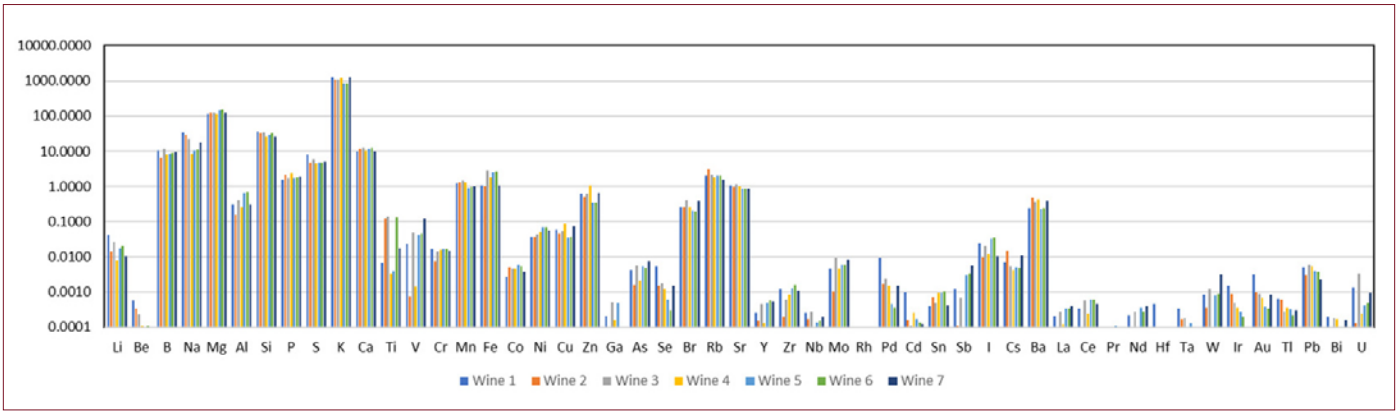


*Figure 3: TotalQuant results for 51 elements in seven US Napa Valley red wines.*

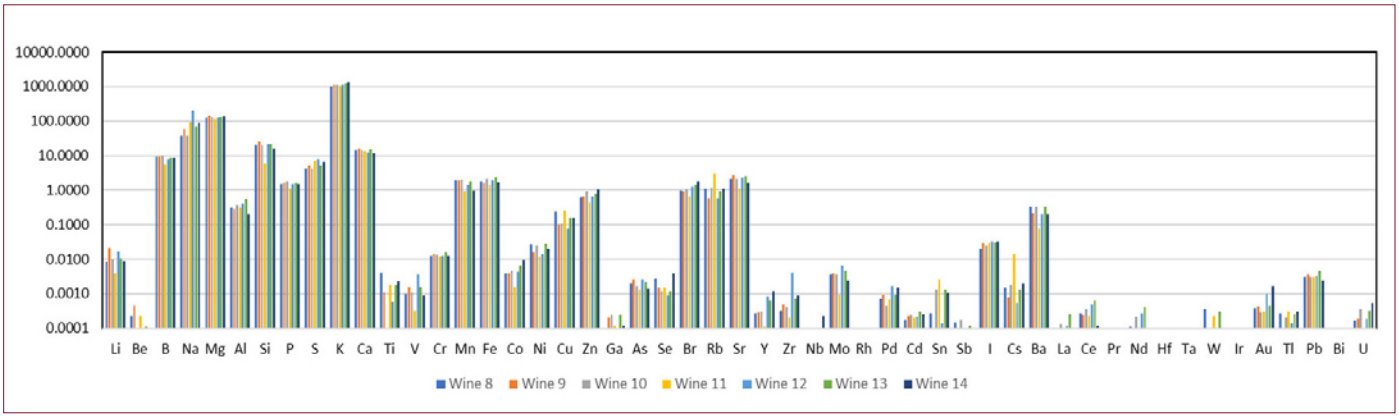


*Figure 4: TotalQuant results for 51 elements in seven Australian red wines.*

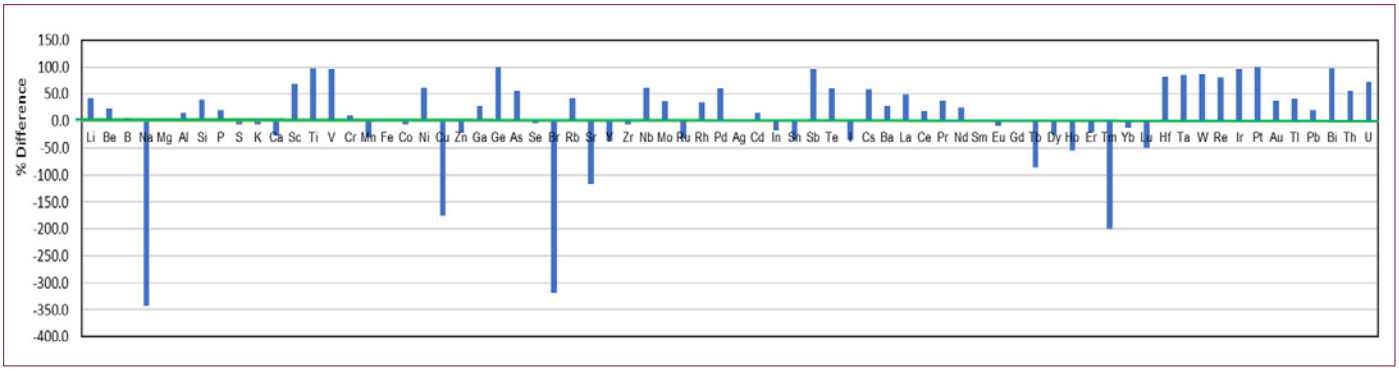


*Figure 5: Difference in averaged elemental concentrations between Napa Valley (positive %) and Australian (negative %) red wines.*

Next, a statistical evaluation of the TotalQuant results was done using the principal component analysis (PCA) with TIBCO Spotfire® software. This technique was employed to differentiate wines according to their geographical origin. The PCA plot (Figure 6), based on PC1 vs. PC2, clearly shows that it is possible to distinguish between wines from Australia (red dots: wines 8-14) and US Napa Valley (blue dots: wines 1-8). Australian wines are clustered together even though they come from a much wider geographical area versus US Napa Valley wines.

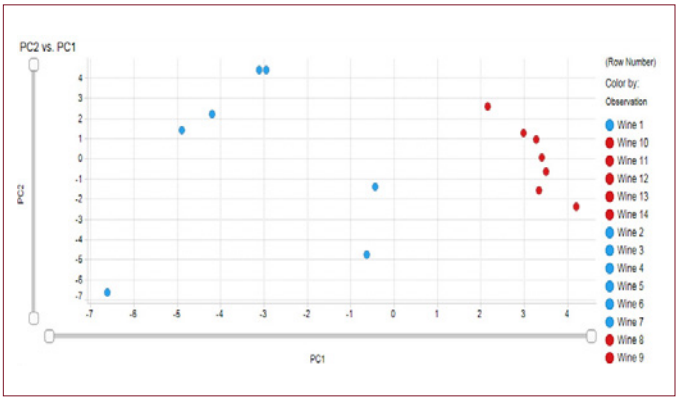


*Figure 6: PCA plot based on first and second principal component scores.*

Figure 7 shows a loading plot with the contribution of each variable (element) for the PC1. Elements such as Br, Ca Cu, Na have the highest loading in the PC1 that agrees well with the plot of % elemental differences in Figure 5.

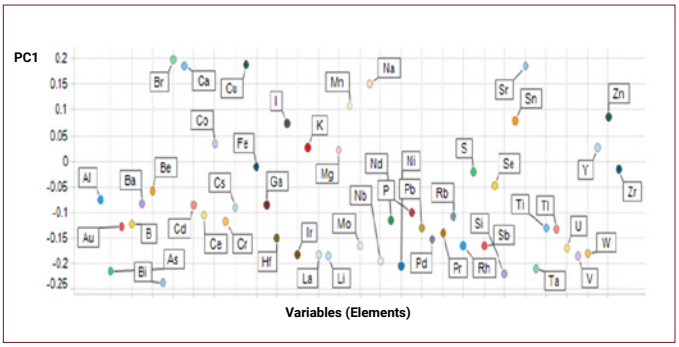


*Figure 7: Loading plot based on first principal component PC1 vs. variables (all 51 elements).*

## Conclusion

The NexION 2000 ICP-MS in combination with the TotalQuant feature in Collision mode can quickly quantify 81 elements using just one standard. Determination of major and trace elements in the same run with the same dilution factor is virtually effortless due to the instrument's wide working range. The system can handle the analysis of difficult matrices, like wines, very well despite their high levels of alcohol and sugars. TotalQuant can not only measure the concentration of many elements, but it can also be used as a tool for fingerprinting and profiling wines. Statistical analysis of results can help assign the geographical origins of unknown wines and even highlight adulteration.

## Consumables Used

| Component | Part Number |
|---|---|
| ST3 MicroFlow PFA Nebulizer | N8152378 |
| PVC Peristaltic Pump Tubing Orange/Yellow | N8145150 |
| Santoprene Peristaltic Pump Tubing Grey/Grey | N8145160 |
| Quartz Cyclonic Spray Chamber | N8152383 |
| One-piece Quartz Torch with 2 mm Injector | N8152472 |

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