Analysis of Trace Elements in Whisky Using the Thermo Scientific iCAP 7000 Plus Series ICP-OES

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ABSTRACT

Purpose: To demonstrate the ability of ICP-OES to determine trace elements and major components in whisky.

Methods: A multielement method including UV and visible wavelengths was used to determine the concentrations of a range of elements. An autosampler was coupled to an ICP-OES system to automate sample introduction.

Results: The ICP-OES system was able to simultaneously determine both major and minor element concentrations in a number of commercially available whiskys with good spike recovery showing robust matrix tolerance.

INTRODUCTION

The production of whisky is a complex multi-step process. One of the key stages is distillation, which may occur more than once. Copper is used to make distillation equipment, as it has beneficial properties including good heat conduction and catalytic properties. It also has the ability to neutralize sulfur compounds, which are considered to possess unpleasant and undesirable aromas that negatively affect the final product (Harrison, 2011).

During the distillation process, trace metals may leach into the final product from the distillation equipment including arsenic. If poor quality copper is used, arsenic concentrations could reach toxic levels so it is important to be able to accurately quantify trace elements.

Other metal or trace element contaminants can come from raw ingredients used for the production. Elements such as manganese, zinc, lead or cadmium can be introduced from the water or grain used in production (Iwegbue, 2014) and the likelihood of their presence can be increased due to use of fertilizers and pesticides.

MATERIALS AND METHODS

Instrumentation:

A Thermo Scientific™ iCAP™ 7600 ICP-OES Duo (Figure 1) was used in the experiments described in this paper. Method parameters, listed in Table 1, were applied for all analyses .The duo system configuration was chosen for its ability to detect trace elements in the axial view and for its wide dynamic range that can be extended with the radial view. A Teledyne CETAC ASX-560 autosampler was used to transfer the sample to the introduction system of the ICP-OES.

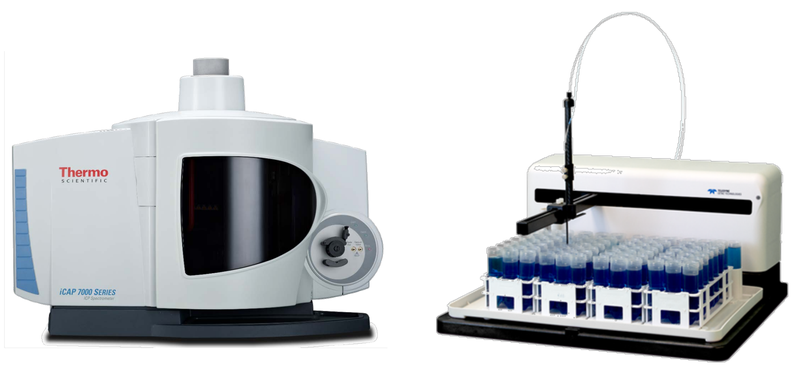

Figure 1. iCAP 7600 Duo ICP-OES, CETAC ASX-560 autosampler.

Data Analysis:

For each element, wavelengths were selected using the intuitive Wavelength selection tool of the Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software.

To ensure freedom from interferences, the subarray plots were examined and background correction points were set appropriately, as demonstrated in Figure 2.

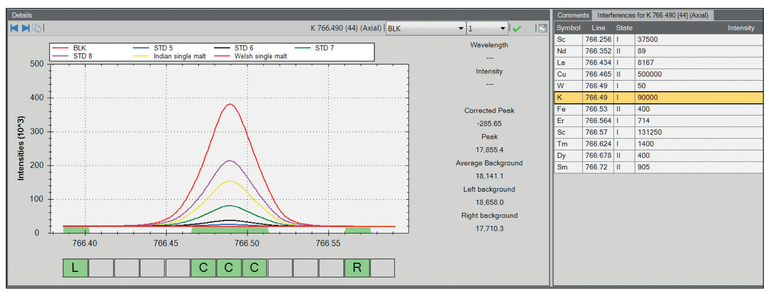

Figure 2. Subarray plot for K 766.490 nm, indicating the peak center and background points.

Sample Preparation:

Calibration standards and spike solutions were prepared from single element solutions (1000 mg·L^{-1}) using 18 MΩ ultra-pure water and trace metal grade nitric acid (67-69%) as well as analysis grade ethanol (99.8+%), to a final concentration of 0.2% nitric acid and 40% ethanol for each solution.

Five different whisky samples were analyzed. The accuracy for trace elements analyzed was tested by spiking the samples with a concentration of 100 µg·L^{-1} of the elements used for the Calibration.

To account for physical interferences due to different matrices in the whisky samples, an internal standard solution of yttrium (10 mg·L^{-1}) was added to all solutions online via a T-piece.

Table 1. Method Parameters for the iCAP-7600 ICP-OES

Parameter	Setting	
Pump tubing	Sample: Drain: Internal standard:	Solvent Flex white/white Solvent Flex blue/yellow Tygon™ orange/green
Pump speed	25 rpm	
Spray chamber	Baffled cyclonic	
Nebulizer	V-groove	
Center tube	1.5 mm	
Torch	EMT	
Nebulizer gas flow	0.35 L·min^{-1}	
Auxiliary gas flow	1.5 L·min^{-1}	
Coolant gas flow	16 L·min^{-1}	
RF Power	1300 W	
Exposure time	Axial view	Radial view
	UV 15 s, Vis 15 s	UV 15 s, Vis 15 s

Table 2. Calibration standard concentrations (µg·l^{-1}).

Element and Wavelength (nm)	View	Blank	STD 1	STD 2	STD 3	STD 4	STD 5	STD 6	STD 7	STD 8
Na 589.592	Axial	0	0	0	0	0	250	750	2500	7500
K 766.490	Axial	0	0	0	0	0	250	750	2500	7500
Ca 393.366	Axial	0	2.5	7.5	25	75	250	750	0	0
Cr 283.563	Axial	0	2.5	7.5	25	75	250	750	0	0
Mn 257.610	Axial	0	2.5	7.5	25	75	250	750	0	0
Fe 238.204	Axial	0	2.5	7.5	25	75	250	750	0	0
Co 238.892	Axial	0	2.5	7.5	25	75	250	750	0	0
Ni 231.604	Axial	0	2.5	7.5	25	75	250	750	0	0
Cu 324.754	Axial	0	2.5	7.5	25	75	250	750	0	0
Zn 213.856	Radial	0	2.5	7.5	25	75	250	750	0	0
As 189.042	Radial	0	2.5	7.5	25	75	250	750	0	0
Cd 214.438	Radial	0	2.5	7.5	25	75	250	750	0	0
Ba 455.403	Axial	0	2.5	7.5	25	75	250	750	0	0
Pb 220.353	Radial	0	2.5	7.5	25	75	250	750	0	0
Mg 279.553	Axial	0	2.5	7.5	25	75	250	750	0	0

RESULTS

The results obtained for the analysis of the different whisky samples are shown in Table 3 and spike recoveries are highlighted in Figure 3.

Table 3. Concentrations of major and trace elements in whisky samples, with method detection limits (MDL) in µg·L^{-1}

Element	Indian Single malt	Welsh Single malt	Irish 1	Irish 2	Irish 3	MDL (µg/L)
Na	769	2160	7182	4890	7901	1.17
K	5271	13976	4795	5246	2101	1.74
Ca	250	501	307	217	428	0.06
Cr	<DL	<DL	8.69	<DL	<DL	1.3
Mn	19.8	23.9	7.45	6.11	4.82	0.3
Fe	41.7	21.8	28.1	24.8	21.7	2.19
Co	<DL	<DL	<DL	<DL	<DL	3.09
Ni	4.78	6.48	0.03	<DL	<DL	3.67
Cu	1636	131	21.5	46.1	37.2	1.34
Zn	33.4	21.7	3.06	2.33	4.5	1.47
As	41.8	39.8	<DL	<DL	<DL	30.1
Cd	2.03	<DL	<DL	<DL	<DL	1.94
Ba	0.764	4.36	1.33	0.794	0.493	0.12
Pb	<DL	<DL	<DL	<DL	<DL	33.4
Mg	318	473	99.7	69.1	73.8	0.06

<DL: below detection limit.

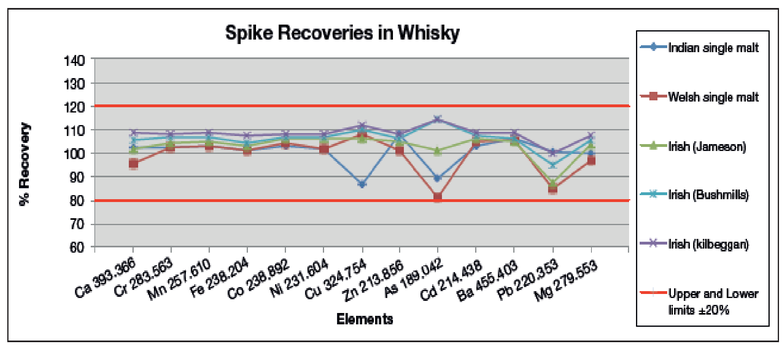

Figure 3. Spike recoveries obtained for the analysis of the different Whisky samples.

The spiked samples were analyzed in the same run as the samples and all recoveries for the trace elements were within the acceptable range of ±20%.

The detection limit study, which is also shown in Table 3, reports values in the single digit µg·L^{-1} range or lower. Exceptions are arsenic and lead, with 30 µg·L^{-1} and 33 µg·L^{-1} MDLs respectively.

CONCLUSIONS

This study demonstrates that the iCAP 7600 ICP-OES Duo also delivers robust performance when analyzing complex organic sample matrices such as whisky.

Careful selection of interference-free wavelengths allows determination of trace elements with very low detection limits (single µg·L^{-1}) and excellent accuracy, proving that the iCAP 7600 ICP-OES Duo is an ideal choice for analysis of trace elements in alcoholic beverages.

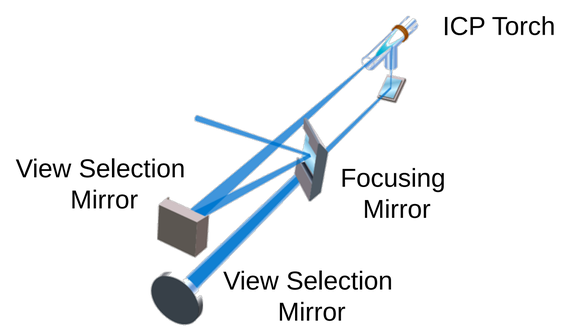

Figure 4. Optical layout of the iCAP 7600 Duo ICP-OES

REFERENCES

1. Harrison, B., Fagnen, O., Jack, F., Brosnan, J. (2011). *J. Inst. Brew.* **117:** 106–112
2. Iwegbue, C. M. A, Overah, L. C., Bassey, F. I., Martincigh, B. S. (2014). *J. Inst. Brew.* **120:** 521–528

TRADEMARKS/LICENSING