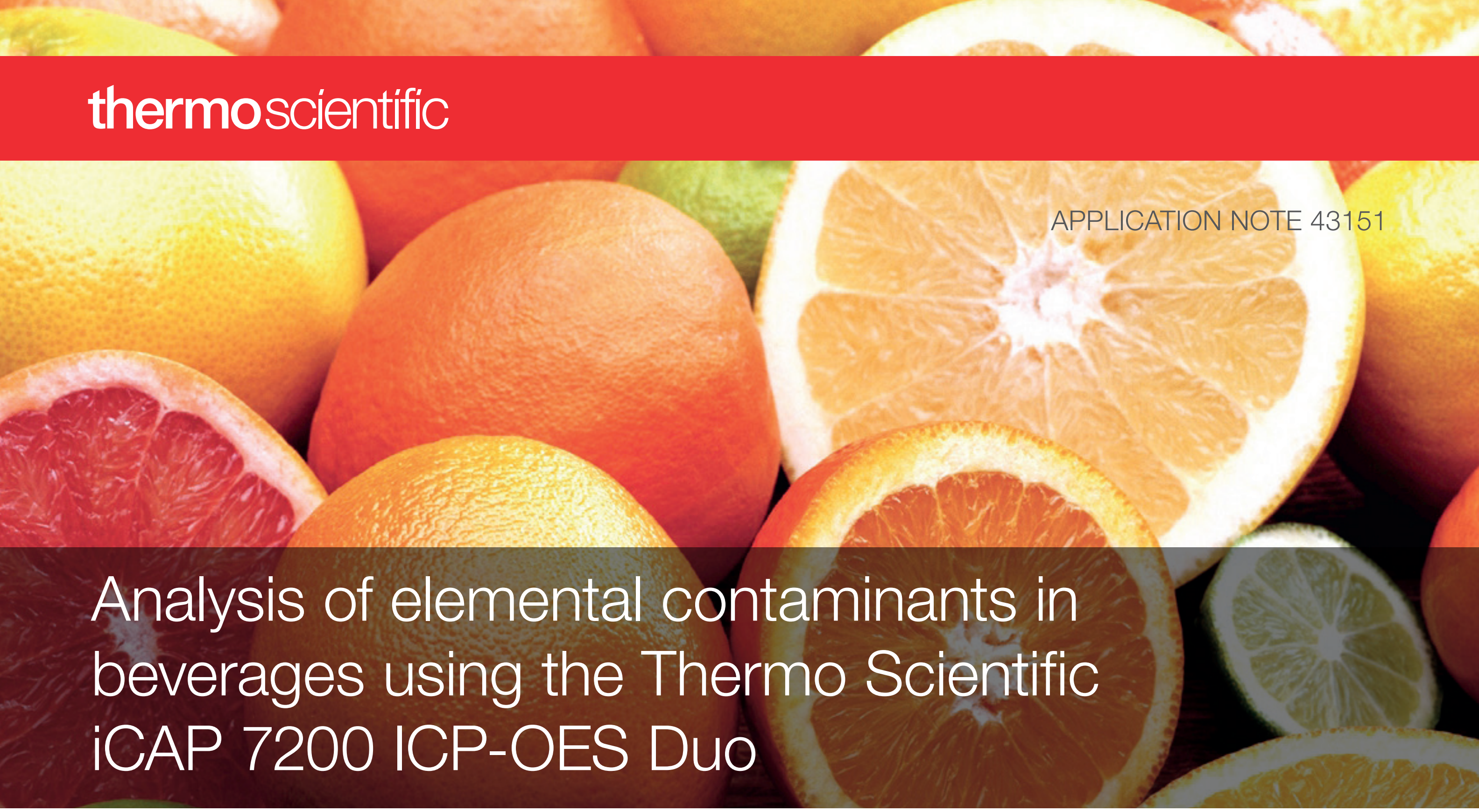

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APPLICATION NOTE 43151

Analysis of elemental contaminants in beverages using the Thermo Scientific iCAP 7200 ICP-OES Duo

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Keywords

Beverages, Contaminants,
EN 1134:1994E

Goal

This application note describes the high performance of the Thermo Scientific iCAP 7200 ICP-OES Duo when analyzing elemental contaminants in different types of beverages. The duo view offers optimal method conditions using axial plasma view for traces and radial plasma view for major elements.

Introduction

In recent years, several investigations around the world have captured global media attention by highlighting excessive contamination of foodstuffs and beverages. This has prompted local authorities in many countries to increase the legislation and level of testing required for foodstuffs and beverages. Most method directives involving the analysis of beverages, including those in Europe, are based upon acid digestion and analysis by atomic absorption spectroscopy (AAS), these have yet to be updated to include Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES).

The analysis of contaminants in beverages in Europe is based on the recommendation of the European Union “Reports on tasks for Scientific Cooperation” (SCOOP, Task 3.2.11) and the Committee on Toxicity (COT; Chemicals in Food, Consumer Products and the Environment, 2004) which researched and established the average human intake and absorption of contaminants and nutrients from liquids. The elements ordinarily analyzed, both contaminants and major constituent elements are displayed in Table 1.

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Table 1. Elements commonly analyzed in beverages.

Major nutrients	Contaminants
Ca, Mg, K, Na	Al, As, Cd, Cu, Fe, Mn, Pb, Sn, Zn

The method most commonly used for fruit juices is EN 1134:1994 "Method for determination of sodium, potassium, calcium and magnesium contents of fruit and vegetable juices by atomic absorption spectrometry", which was developed for atomic absorption spectrometry analysis and includes microwave digestion of samples. ICP-OES has the advantage of processing these samples directly, so the EN 1134 method has been adapted to allow for removal of the digestion step, thus saving time and materials and reducing method development.

Instrumentation

The Thermo Scientific™ iCAP™ 7200 ICP-OES Duo was used for the direct analysis of a range of beverages. This instrument achieves powerful analyte detection and provides a highly cost effective solution for routine analysis of liquids in laboratories with standard sample throughput requirements. The Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software simplifies method development and provides an option of immediate analysis.

Sample and standard preparation

Three different beverages below were analyzed:

- Blackcurrant squash concentrate – normally consumed diluted with water (in a 1:4 proportion)
- Beer – a British ale was used, one drop of a silicone anti-foaming agent was added to reduce the influence of the dissolved CO_2 gas on nebulization and transport
- Pure cranberry juice (smooth) – consumed undiluted

Samples were prepared by shaking the individual containers and weighting 20 g of each into a glass volumetric flask. An additional 2 g aliquot mass was also weighed into a volumetric flask to determine higher concentration elements. A duplicate was prepared for each beverage, which was spiked with a small concentration of aqueous standard for recovery testing. All samples were diluted to 100 mL volume with deionized water.

Calibration standards were prepared by diluting traceable 1000 mg·L^{-1} single element solutions (Fisher Chemicals, Loughborough, UK), the final concentrations are listed in Table 2. All solutions were made to volume in deionized water, although 0.5% high purity nitric acid may be used to improve the long-term stability of the solutions, as required.

Table 2. Calibration standard concentrations (mg·L^{-1}).

Element	Blank	Standard 1	Standard 2	Standard 3
Al	0	0.5	1	2
As	0	0.5	1	2
Ca	0	2	5	10
Cd	0	0.5	1	2
Cu	0	0.5	1	2
Fe	0	0.5	1	2
K	0	2	5	10
Mg	0	2	5	10
Mn	0	0.5	1	2
Na	0	2	5	10
Pb	0	0.5	1	2
Sn	0	0.5	1	2
Zn	0	0.5	1	2

Method development

A method was created in the Qtegra ISDS Software. Wavelengths were selected as they were free from interferences and offered the sensitivity required for the analysis. The standard sample introduction kit was used for the analysis as per the recommendations in the method notes. The instrument was calibrated and the samples analyzed in a single run. Table 3 shows the parameters used for the method.

Table 3. Method parameters.

Parameter	Setting	
Pump Tubing (Standard Pump)	Sample Tygon® orange/white Drain Tygon® white/white	
Pump Speed	45 rpm	
Nebulizer	Glass concentric	
Nebulizer Gas Flow	0.15 MPa	
Spray Chamber	Glass cyclonic	
Center Tube	2 mm	
RF Power	1150 W	
Coolant Gas Flow	12 L·min^{-1}	
Auxiliary Gas Flow	1.0 L·min^{-1}	
Exposure Time	**Axial**	**Radial**
	UV 15 s, Vis 5 s	UV 15 s, Vis 5 s

Results

The results obtained in the analysis of the different beverage samples are shown in Table 4 and are further highlighted in Figure 1. Each sample was spiked with the same 1000 mg·L^{-1} solutions used to make the calibration standards. These spiked samples were analyzed in the same run as the samples and all recoveries were within the acceptable range of ±20%. Method detection limits (MDLs) were established for the trace elements by analyzing the cranberry juice, with ten replicates. This approach was chosen as the majority of the trace elements were very close to the expected detection limit concentrations or slightly higher. The standard deviation of the result was multiplied by 3 to give a detection limit in mg·L^{-1} that equates to a confidence interval of approximately 98.5%.

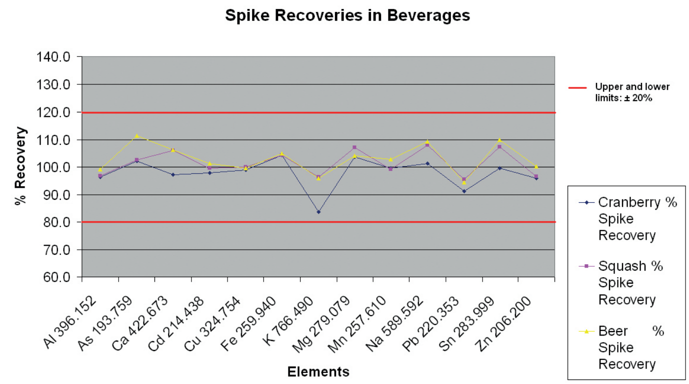

Figure 1. Percentage recovery of spikes in analyzed beverages.

Table 4. Results of the analysis of the beverages.

Element and wavelength (nm)	Spike value (mg·L^{-1})	Cranberry undiluted (mg·L^{-1})	Cranberry spike recovery (%)	Cranberry 20 g/100 mL detection limit (µg·L^{-1})	Squash undiluted (mg·L^{-1})	Squash spike recovery (%)	Beer undiluted (mg·L^{-1})	Beer spike recovery (%)
Al 396.152	0.5	0.0393	96.3	0.93	0.1305	96.8	0.1318	99.1
As 193.759	0.5	<DL	102.1	4.11	<DL	102.5	<DL	111.4
Ca 422.673	2	53.1	97.2	–	40.1	106.1	36.7	106.2
Cd 214.438	0.5	<DL	97.9	0.07	<DL	99.5	<DL	101.4
Cu 234.754	0.5	0.0425	98.8	0.85	0.0093	100.1	0.0100	99.6
Fe 259.940	0.5	0.44	104.4	6.08	0.2345	104.4	0.1338	104.9
K 766.490	2	190	83.8	–	484	96.5	532	96.0
Mg 279.079	2	21.1	103.7	–	7.72	107.1	89.4	104.0
Mn 257.610	0.5	0.3645	99.5	1.65	0.106	99.0	0.092	102.9
Na 589.592	2	40.9	101.3	–	156	107.9	28.2	109.2
Pb 220.353	0.5	<DL	91.1	1.28	<DL	95.5	<DL	94.4
Sn 283.999	0.5	<DL	99.7	5.11	<DL	107.3	0.0028	109.8
Zn 206.200	0.5	0.085	96.0	0.47	0.0340	96.7	<DL	100.2

All of the trace contaminants detrimental to human health (As, Cd and Pb) were measured and found to be below the regulation limits (see Table 5 below).

Table 5. Regulation maximum allowed concentrations in various beverages.

Element	Maximum (mg·L^{-1})
As	<0.2
Cd	<0.1
Cu	<2
Fe	<7
Pb	<0.5
Sn	<100

The results show that the robust RF generator and the optimized sample handling kit easily handled the range of sample densities and viscosities. An internal standard may be added if the recoveries degrade below regulation levels. An online, internal standard mixing kit may also be used for ease of use or to reduce labor for higher sample numbers. By connecting an additional pump tube and adding the internal standard on-line, continuous accurate dilution of the sample is assured. Qtegra ISDS Software allows the analyst the ability to turn the internal standard on or off pre- or post-analysis, saving valuable method development time as only one analysis is required to generate two sets of results.

Conclusion

The Thermo Scientific iCAP 7200 ICP-OES Duo and Qtegra ISDS Software features make the analysis of beverages rapid and analyst friendly allowing both experienced and inexperienced users alike to vastly reduce the method development time for these types of samples, resulting in cost effective analyses. In addition to the time saving on method development, removal of the digestion stage and the use of internal standards produces an easy to use, versatile method capable of analyzing a wide variety of food and beverage samples.

Find out more at **thermofisher.com/ICP-OES**

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