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APPLICATION NOTE 44421

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

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Goal

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

Introduction

The analysis of elements in beverages is a routine practice to ensure consumer safety and product quality. A typical analysis would include both the measurement of toxic elements as well as those which provide nutritional benefit to the consumer. Historically, the analytical technique of choice was atomic absorption spectroscopy (AAS). However, due to the limitations of AAS, such as its ability to measure only one element at a time, there has been a shift towards inductively coupled plasma–optical emission spectroscopy (ICP-OES).

Within the European Union, guidance on the analysis of beverages is provided by the means of the *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). The elements typically analyzed as a result of this guidance are outlined 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

This application note will detail a procedure for the analysis of fruit juice (and other beverages) by ICP-OES. Typically, this was carried out using the standard method EN 1134:1994 *Method for determination of sodium, potassium, calcium and magnesium contents of fruit and vegetable juices by atomic absorption spectrometry*. The method has been adapted to remove the digestion stage as ICP-OES has the ability to process the sample directly, saving time and reagents. In addition, ICP-OES is a multi-element technique and therefore a greater range of elements can be analyzed without increasing the time of analysis.

Instrumentation

The Thermo Scientific iCAP PRO X Duo ICP-OES was used for the direct analysis of a range of beverages. This truly simultaneous instrument achieves powerful analyte detection and provides a highly cost-effective solution for routine analysis of liquids in laboratories with standard sample throughput requirements. A Teledyne CETAC ASX-560 Autosampler was used for the sample transport into the system. The Thermo Scientific™ Qtegra™ Intelligent Scientific Data Solution™ (ISDS) Software simplifies method development and provides easy options for post-analysis data manipulation.

Sample and standard preparation

Two different beverages were analyzed:

- Apple Juice (consumed undiluted)
- Cola (consumed undiluted)

To reduce the influence of the dissolved CO_2 gas on nebulization and transport, the samples were degassed in an ultrasonic bath. Samples were then prepared by shaking the individual containers and weighting 10 gml of each into a glass volumetric flask. A duplicate was prepared for each beverage, which was spiked with the same 1000 mg·L^{-1} elemental solutions at same concentrations as used to make the calibration standard 2. All samples were diluted to 50 mL volume with deionized water.

Calibration standards and the spiked blank solution were prepared by diluting 1000 mg·L^{-1} single element solutions from Spex CertiPrep (SPEX CertiPrep Group, Metuchen, US), the final concentrations are listed in Table 2. All solutions were made up to a volume of 50 mL in 0.5% high purity nitric acid to improve the long-term stability of the solutions.

Table 2. Calibration standard concentrations (mg·L^{-1}) and blank spike concentration (µg·L).

Element	Blank	Standard 1	Standard 2	Standard 3	Blank Spike (µg·L)
Al	0	0.5	1	2	1
As	0	0.5	1	2	5
Ca	0	2	5	10	1
Cd	0	0.5	1	2	1
Cu	0	0.5	1	2	2
Fe	0	0.5	1	2	1
K	0	2	5	10	30
Mg	0	2	5	10	5
Mn	0	0.5	1	2	1
Na	0	2	5	10	10
Pb	0	0.5	1	2	5
Sn	0	0.5	1	2	5
Zn	0	0.5	1	2	1

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.

<table>
<tr><th>Parameter</th><th colspan="2">Setting</th></tr>
<tr><td>Pump tubing</td><td colspan="2">Sample Tygon® orange/white
Drain Tygon® white/white</td></tr>
<tr><td>Pump speed</td><td colspan="2">45 rpm</td></tr>
<tr><td>Spray chamber</td><td colspan="2">Glass cyclonic</td></tr>
<tr><td>Nebulizer</td><td colspan="2">Glass concentric</td></tr>
<tr><td>Nebulizer gas flow</td><td colspan="2">0.5 L·min-1</td></tr>
<tr><td>Coolant gas flow</td><td colspan="2">12 L·min-1</td></tr>
<tr><td>Auxiliary gas flow</td><td colspan="2">0.5 L·min-1</td></tr>
<tr><td>Center tube</td><td colspan="2">2 mm</td></tr>
<tr><td>RF power</td><td colspan="2">1150 W</td></tr>
<tr><td>Repeats</td><td colspan="2">3</td></tr>
<tr><td rowspan="2">Exposure time</td><td>Axial</td><td>Radial</td></tr>
<tr><td>15 s</td><td>15 s</td></tr>
</table>

Results

The results obtained in the analysis of the different beverage samples are shown in Table 4 and are further highlighted in Figure 1. The spiked samples were analyzed in the same run as the other samples and all recoveries were within the acceptable range of ±15%.

Method detection limits (MDLs) were established for the trace elements by analyzing the spiked blank solution (see Table 2) with ten replicates. The standard deviation of the result of the spike blank was added to that of the instrument detection blank and was multiplied by 3 to give a detection limit in mg·L^{-1} that equates to a confidence interval of approximately 99.7%.

Table 4. Results of the analysis of the diluted beverages.

Analyte wavelength (nm)	Plasma view	Spike value (mg·L^{-1})	Apple juice measured (mg·L^{-1})	Spiked apple juice measured (mg·L^{-1})	Apple juice spike recovery (%)	Cola measured (mg·L^{-1})	Spiked cola measured (mg·L^{-1})	Cola spike recovery (%)	MDL (µg·L^{-1})
Al 167.079	Axial	1	0.12	1.05	93	0.002	1.03	103	0.12
As 189.042	Axial	1	0.01	1.04	103	0.002	1.00	100	4.17
Ca 393.366	Radial	5	11.41	16.01	92	4.12	9.25	103	0.94
Cd 214.438	Axial	1	<DL	0.94	94	4.9	0.99	99	0.17
Cu 224.700	Axial	1	0.02	0.94	93	<DL	1.00	100	0.74
Fe 259.940	Axial	1	0.13	1.10	97	0.02	1.00	100	0.98
K 766.490	Radial	5	166.51	170.95	89	12.52	17.71	104	260
Mg 279.553	Radial	5	8.77	13.32	91	0.997	6.16	103	15
Mn 257.610	Axial	1	0.08	1.05	97	<DL	1.01	101	0.26
Na 589.592	Radial	5	3.74	8.44	94	14.01	19.34	107	14
Pb 220.353	Axial	1	<DL	0.90	90	<DL	0.98	98	2.4
Sn 189.989	Axial	1	0.01	0.89	88	0.001	0.96	96	1.0
Zn 206.200	Axial	1	0.02	0.97	95	<DL	0.99	99	0.022

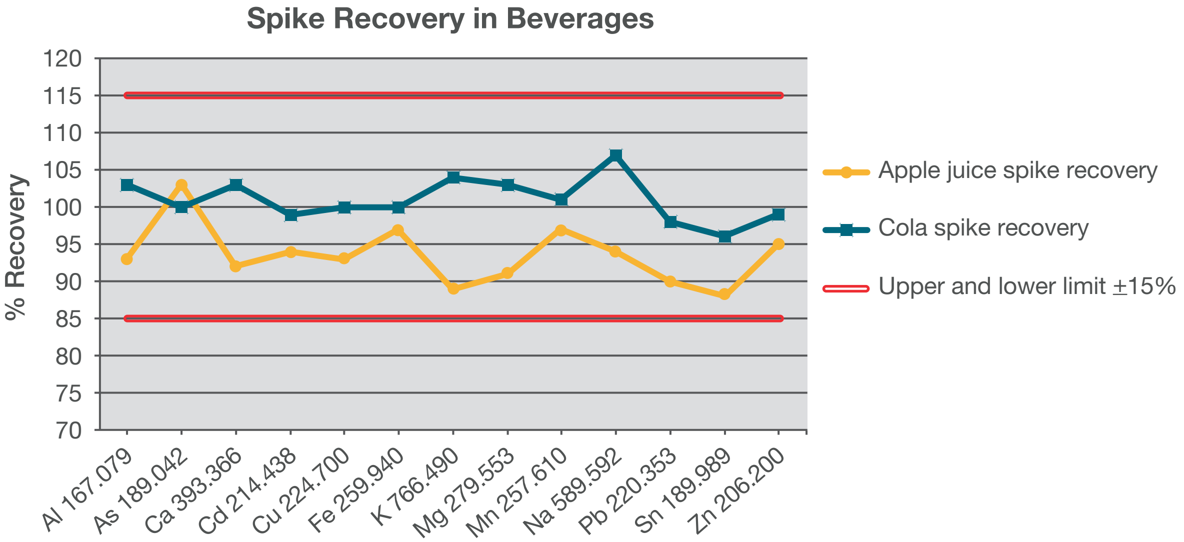

Figure 1. Percentage recovery of spikes in analyzed beverages.

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 ($mg \cdot L^{-1}$)
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 standard sample introduction kit easily handle the range of sample densities and viscosities. An internal standard may be added to account for matrix effects, 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 correction 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 PRO X Duo ICP-OES 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 saved on method development, removal of the digestion stage and the use of internal standards produce 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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