

Determination of Trace Elements in Spirits by ICP-OES

Method ID: N/A

Category: Food and Beverages

Technique: ICP-OES

Summary

This technical note describes an ICP method to determine trace elements in spirits. Trace elements are determined in spirits to ensure quality control and that the final product meets any regulatory requirements. Trace elements can be added to the final product from raw ingredients (e.g., grains) or from process equipment (e.g., copper from stills).

Three brands of whiskey (bourbon) were obtained to be used as samples for this tech note. All three bourbons were 80 proof (40% alcohol).

Instrumentation

A Teledyne Leeman Labs Prodigy Plus High Dispersion ICP in Dual View configuration was used to collect all the data in this technical note. The alkali elements, Li, Na and K, were determined using the radial view. All other elements were determined using the axial view.

Method Parameters

Prodigy Plus Method Parameters	
Parameter	Value
RF Power	1.2 kW
Coolant Flow	16.0 L/min
Auxiliary Flow	1.0 L/min
Nebulizer Pressure	15 psi
Nebulizer Type	Concentric Conikal™
Spray Chamber	Cyclonic with Knockout
Torch	Demountable
Injector ID	1.1 mm
Radial Integration Time	30 s
Axial Integration time	10 s
Uptake Tubing	Red/Red PVC
Drain Tubing	Black/Black PVC



Calibration

Calibration standards of 0, 0.5, 1, 5, 10 and 20 mg/L for all elements were used (K at 0, 5, 10, 50, 100 and 200 mg/L). To match the sample matrix, the standards were made in 40% ethanol to match the sample matrix and were acidified with HNO₃ to 0.5%.

Sample Preparation

The whiskey samples were acidified with HNO₃ to a concentration of 0.5%. No additional sample preparation was needed.

To determine percent recovery, each sample was spiked with 200 µg/L of the elements being determined. K was spiked at 2000 µg/L.

Results

After calibration, each of the three bourbon samples was analyzed. To measure percent recovery, each sample was spiked with 200 µg/L of the elements being determined. (K was spiked with 2000 µg/L).

Detection limits were determined by measuring the blank three times using 10 replicates. The standard deviation of these results was multiplied by 3 to obtain the detection limits.

Concentration data, spike recovery data and detection limits are shown in the table on the next page. All concentration units are in µg/L.

Percent Recovery and Detection Limits							
	Bourbon 1	% Recovery	Bourbon 2	% Recovery	Bourbon 3	% Recovery	Detection Limit
Ag 328.068	< DL	110.7	< DL	109.8	< DL	91.6	0.62
Al 167.079	< DL	105.7	< DL	91.7	< DL	103.8	1.5
As 197.262	<DL	91.1	<DL	94.4	< DL	110.5	13.4
Ca 317.933	735.9	101.8	40.2	90.6	15.5	103.6	0.5
Cd 214.441	< DL	104.5	< DL	90	< DL	103.3	0.32
Co 236.379	< DL	106.9	< DL	103.5	< DL	106.0	1.91
Cr 267.716	14.8	105.6	< DL	106.6	< DL	105.2	0.73
Cu 324.754	7.6	107.2	26.9	104.8	15.9	103.7	0.24
Fe 259.940	174.1	103.4	21.2	100.8	4.0	109.0	1.19
K 766.491 r	10976	101.1	16293	98.6	8954	106.2	0.4
Li 670.784 r	10.8	103.2	12.9	94.9	8.8	95.9	1.89
Mg 285.213	573	101.8	190.2	103.1	93.1	104.5	0.19
Mn 257.610	34.4	104.4	42.2	100.3	15.5	102.1	0.11
Mo 277.540	< DL	102.1	< DL	104.7	< DL	107.1	1.57
Na 589.592 r	2032	99	812.4	90.3	870.7	103.4	2.0
Ni 231.604	< DL	108.9	< DL	101.5	< DL	107.9	1.96
Pb 220.353	< DL	106.2	< DL	102.2	< DL	110.1	5.58
Sb 217.581	< DL	104.7	< DL	108.6	< DL	107.4	1.99
Sr 460.733	89.6	86.6	85.3	103.2	82.9	102.5	0.99
Ti 337.280	4.3	100.5	< DL	103	< DL	94.6	0.29
V 310.230	85.6	97.7	31	100.4	24.2	102.6	1.14
Zn 202.548	< DL	106.3	< DL	99.1	< DL	110.1	0.29

Conclusion

The analysis of spirit samples was successfully performed using the Teledyne Leeman Labs Prodigy Plus ICP-OES. The spike recovery results indicate that all analytes were measured within $\pm 10\%$ of the spiked concentrations. These results demonstrate the Prodigy Plus can be used to provide accurate and reliable results over a range of concentrations in whiskey samples.

The Prodigy Plus is able to run the 40% ethanol samples without the need for oxygen or any additional gases. The standard sample aqueous introduction system, using a cyclonic spray chamber and concentric nebulizer can easily handle the alcohol matrix. Due to the volatility of the alcohol sample, an organic injector with a 1.1 mm inside diameter was used.

The Prodigy Plus ICP-OES was well suited to the determination of elements in whiskey samples due to the high precision, accuracy and versatility provided by its stable, free-running 40 MHz power supply and high-sensitivity sample introduction system.