Author: Scott Neihardt

EdibleOil ICP – Heated Automation and Sample Introduction for Metals Analysis

Abstract

EdibleOil ICP heats the entire sample flow path to melt samples and automate elemental analysis of liquid, semi-liquid, and solid edible oil samples. EdibleOil ICP includes:

- Heated autosampler racks/vials
- Heated autosampler probe line
- Heated MP^2 peristaltic pump
- Heated glass nebulizer and spray chamber
- Heated spray chamber waste line
- Heated rinse station solution

This note describes the EdibleOil ICP analytical procedure with an Avio 200 ICP showing low detection limits, high throughput, and stable results for extended and automated analytical runs.

Introduction

Sourced from plants, nuts, and seeds, edible oils are a vital component of the global food industry.

To improve the taste and shelf life, edible oils are typically refined and have their quality regularly measured to ensure impurities have been removed and industry standards are met.

Elemental analysis of edible oils is an important aspect of overall quality control. This can be a complex procedure, especially for oils which are solid at room temperature, which are not compatible with traditional sample introduction systems used with ICP instruments.

The development of an automation system with a fully heated flow path allows for analysis of the full range of edible oil products with greater ease, speed, and accuracy than was previously possible.

Completely Heated Automation System

EdibleOil ICP is a completely automated sampling system for the analysis of edible oils – whether solid or liquid at room temperature. The system combines a DXCi autocorrecting autosampler with a heated sample deck and a fully heated sample pathway to assure a reliable sample flow. This design ensures reliable and repeatable analysis over extended operation for a wide range of sample types.

DXCi autocorrecting autosampler:

- Automatically goes to the correct sample position, even if accidentally obstructed
- Reports obstruction to the instrument software
- Enhances productivity with high speed operation

EdibleOil ICP heated system advantages:

- Heated sample deck reduces sample preparation time
- Reduced sample dilution for better detection limits
- Viscosity reduction through heat allows for single method for all sample types
- Solid samples remain liquid through analysis and out to waste
- *FAST* valve combined with the ICP for even higher throughput

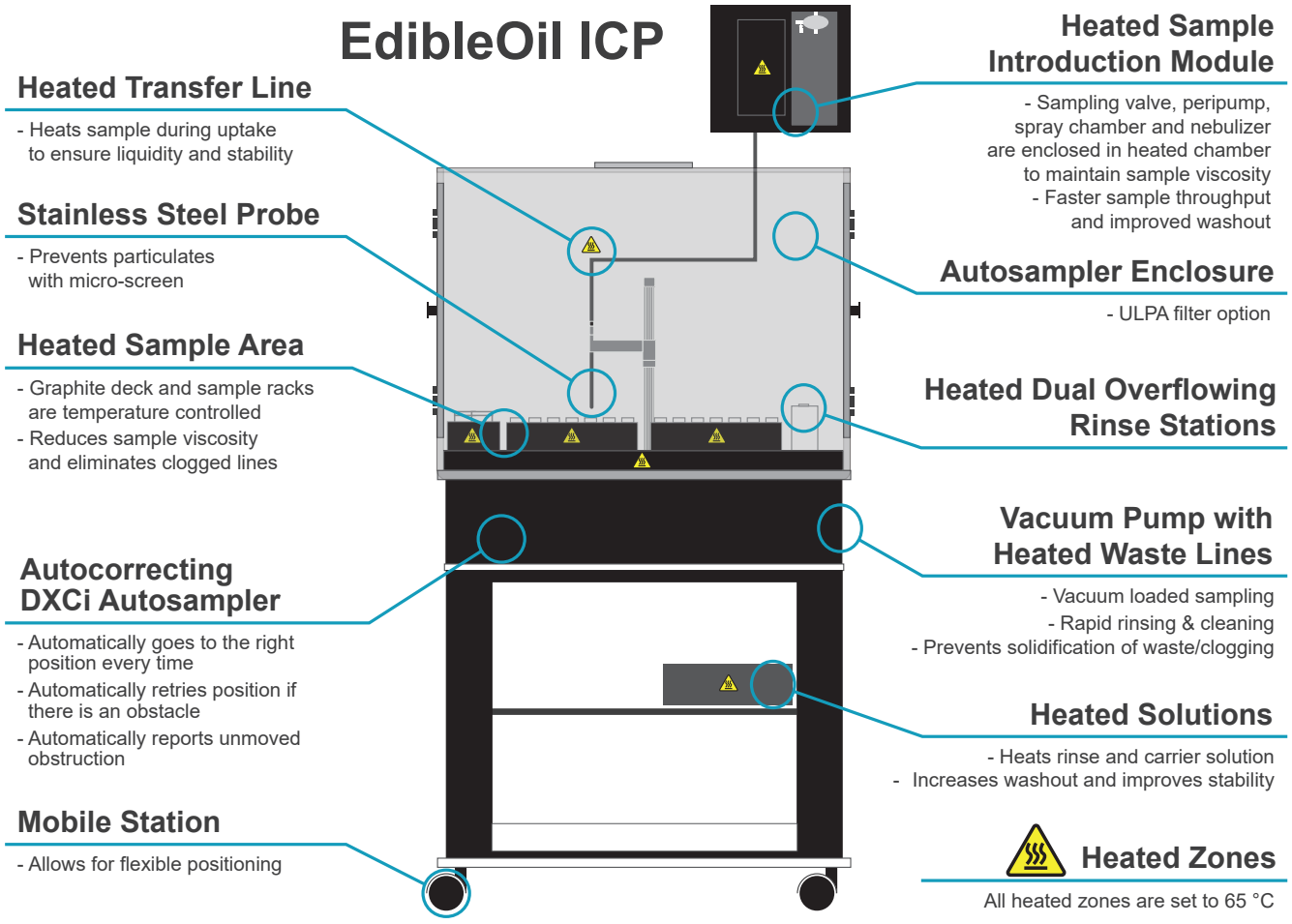


Figure 1. EdibleOil ICP features diagram.

Instrumentation

All samples were analyzed using EdibleOil ICP in combination with an Avio 200 ICP-OES system.

Features:

- Samples heated to 65°C
- Simplified sample preparation
- Automatic injection and triggering of the ICP-OES analysis
- Heated sample deck and transfer lines to reduce sample viscosity
- Heated sample introduction chamber for liquid sample injection for greater stability and accuracy

Sample Preparation

A wide range of food oil samples were diluted 1:1 (w/w) using V-Solv ICP Solvent spiked with yttrium at 40 ppm for internal standard. The autosampler rinse station was supplied with heated V-Solv.

Table 2. Food Oil Samples

Sample Type	Preparation Method	State at Room Temp	State on EdibleOil ICP
Sunflower Oil	1:1 (w/w)	Liquid	Liquid
Corn Oil	1:1 (w/w)	Liquid	Liquid
Peanut Oil	1:1 (w/w)	Liquid	Liquid
Soybean Oil	1:1 (w/w)	Liquid	Liquid
Canola Oil	1:1 (w/w)	Liquid	Liquid
Coconut Oil	1:1 (w/w)	Solid	Liquid
Crude Palm Oil	1:1 (w/w)	Solid	Liquid
Crude Shea Butter	1:1 (w/w)	Solid	Liquid
Fatty Acid Distillate	1:1 (w/w)	Solid	Liquid
Palm Kernel Oil	1:1 (w/w)	Solid	Liquid
Palm Stearin	1:1 (w/w)	Solid	Liquid

Table 1. Instrument conditions used for the Avio 200 ICP-OES

Instrument Settings	
ICP RF Power	1500 W
Nebulizer Gas Flow	0.40 mL/min
Auxiliary Gas Flow	1.2 L/min
Plasma Gas Flow	14 L/min
Nebulizer	MEINHARD® V-Groove Nebulizer
Spray Chamber	Baffled Dual Pass Glass Zip Chamber
Torch	Quartz Organics Demountable Torch
Injector	1.5 mm ID Fully Demountable Quartz Injector
Viewing Mode	Radial

Table 3. Analytes and Wavelengths Measured

Analyte	Wavelenth (nm)	Analyte	Wavelenth (nm)
Y (IS)	361.104	Na	589.590
Al	308.215	Ni	227.025
Cd	228.802	P	213.620
Cu	324.752	Pb	220.357
Fe	259.939	Si	251.611
Mg	285.213		

Figure 2. EdibleOil ICP on an Avio 200 ICP-OES

Calibration of Analytes

Calibrations were automatically performed at 0, 2.5, 5 and 10 ppm in mineral oil

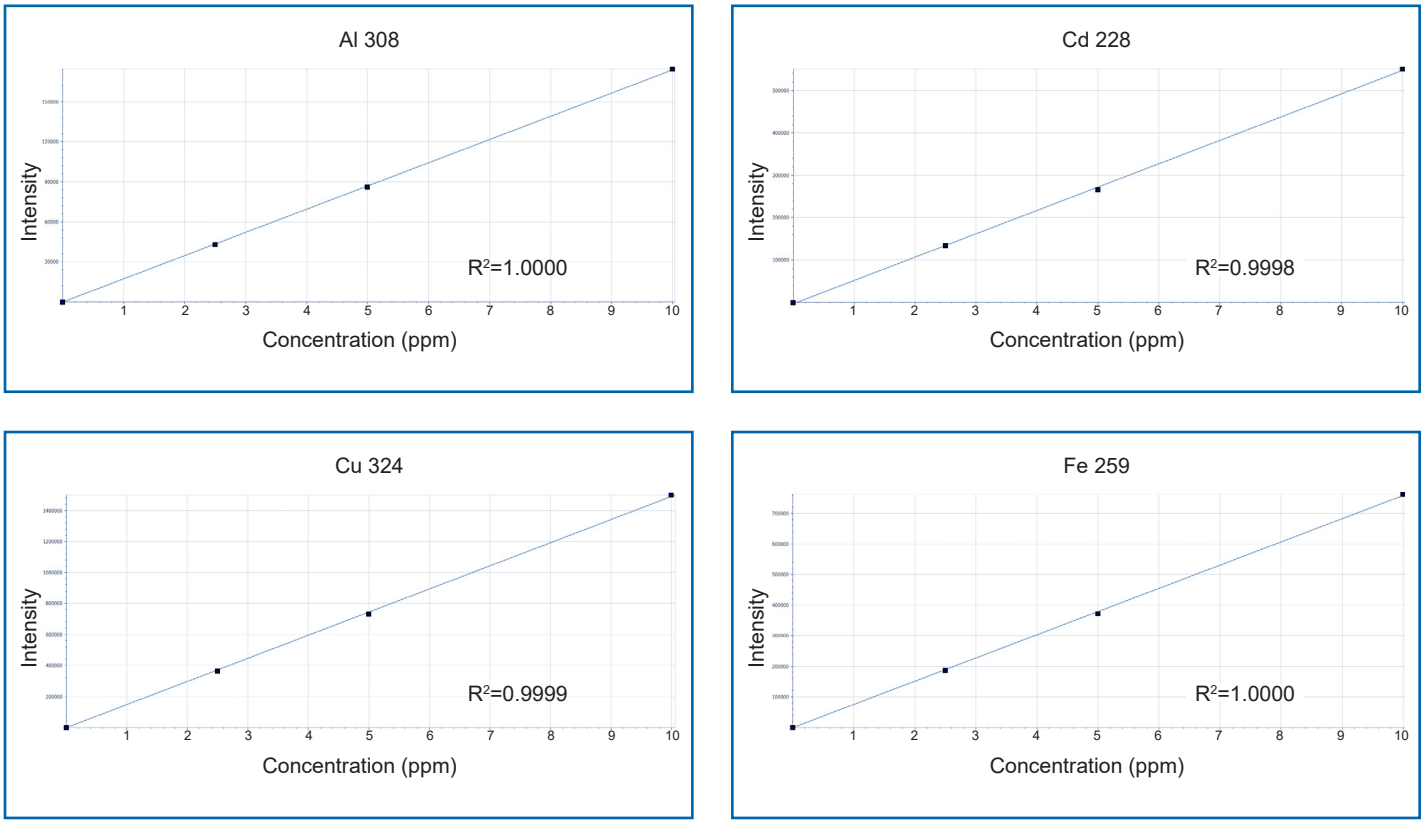
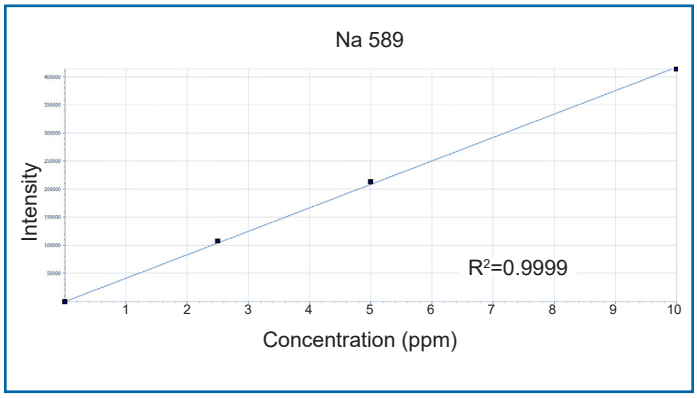
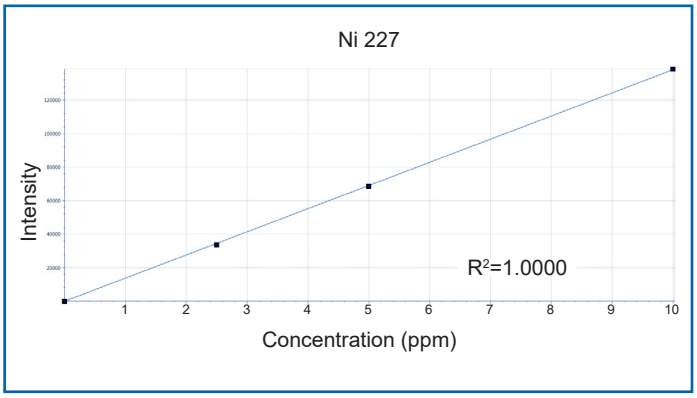


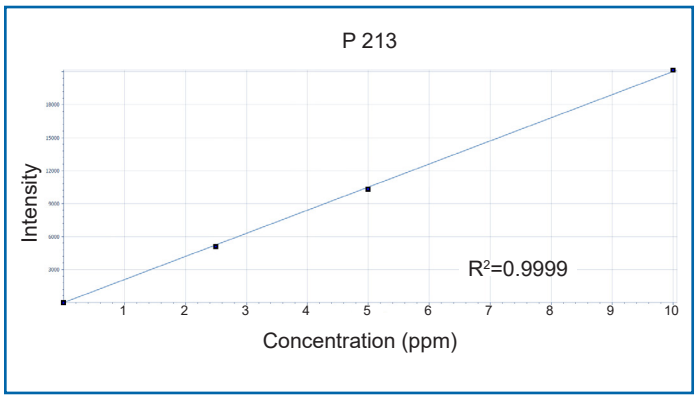
Figure 3a. 5 of 10 calibration curves generated in 12 minutes using three standards prepped from a single V-23 oil 50 ppm stock solution. A 75 cSt base oil blank was also added to maintain the same level of oil matrix present throughout the calibration.

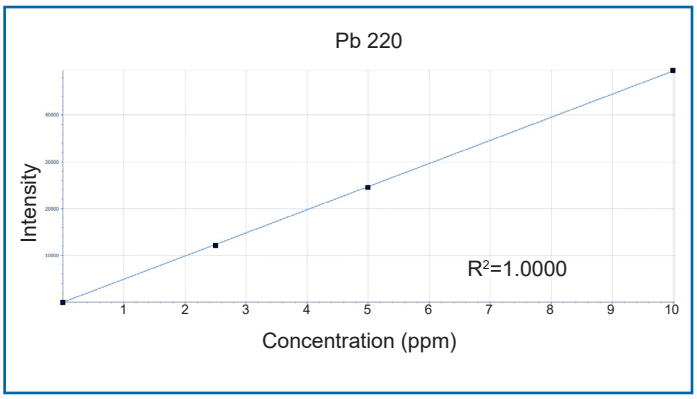
Calibration of Analytes

Calibrations were automatically performed at 0, 2.5, 5 and 10 ppm in mineral oil









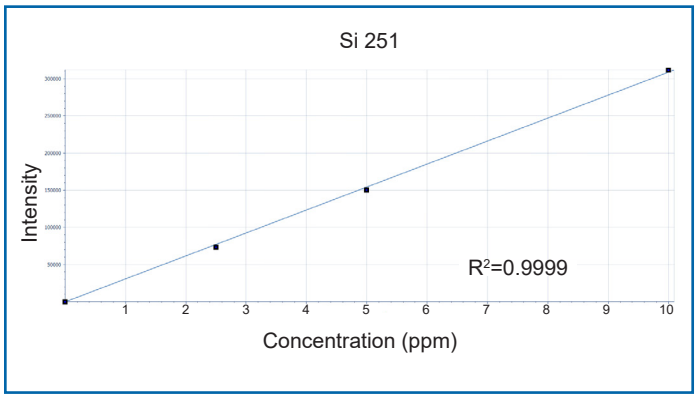


Figure 3b. 5 of 10 calibration curves generated in 12 minutes using three standards prepped from a single V-23 oil 50 ppm stock solution. A 75 cSt base oil blank was also added to maintain the same level of oil matrix present throughout the calibration.

Results

The system was calibrated at 0, 2.5, 5, and 10 ppm using a single V-23 oil 50 ppm stock solution. A 75 cSt base oil blank was also added to maintain the same level of oil matrix present throughout the calibration. EdibleOil ICP generated linear calibration curves for all elements in 12 minutes. The system then analyzed 145 solid palm stearin samples diluted 1:1 (w/w) in V-Solv (heated to liquid state) in just over 9 hours. The stability was excellent, with RSDs for all the main elements under 3%. The washout in the blank, immediately after analyzing three high standards, was at or below blank level, showing great performance for food oil analysis.

Spiked Palm Stearin Stability Over 9 Hours

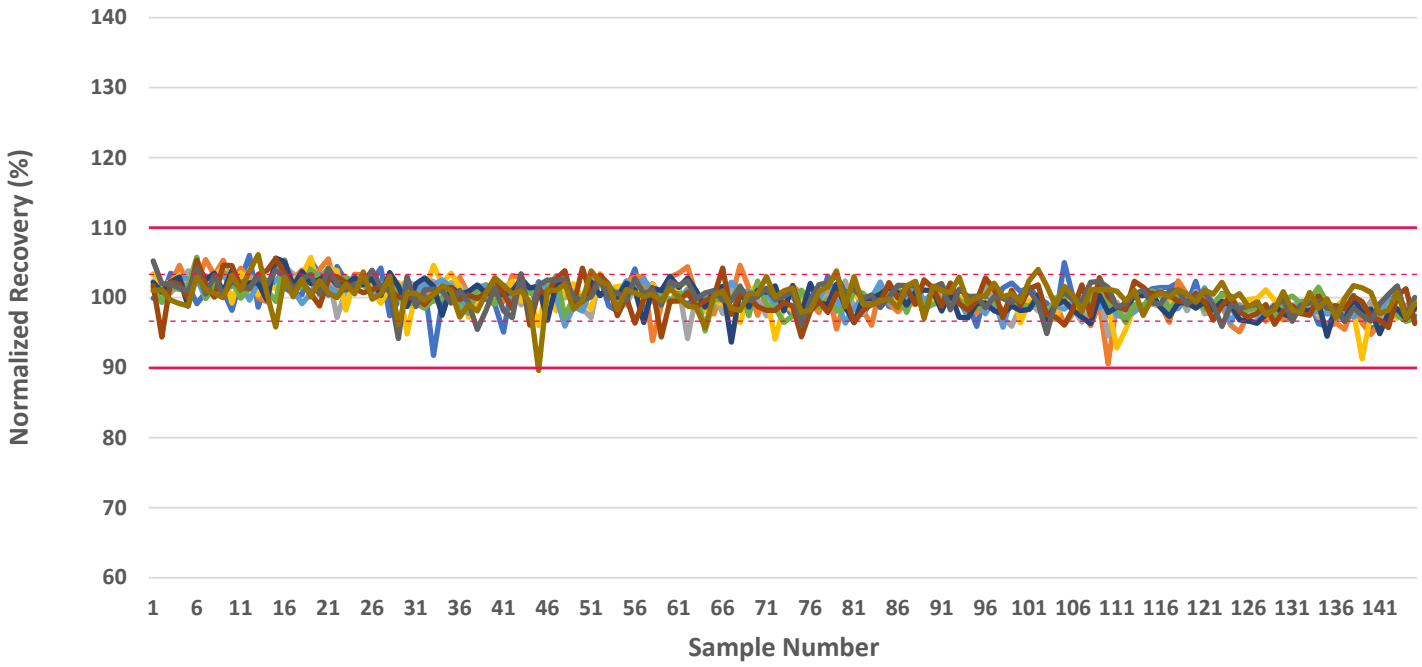


Figure 3. Normalized recoveries for solid palm stearin diluted 1:1 by weight with V-Solv and spiked at 7 ppm for all elements. 145 samples were measured for >9 hours with RSDs for all elements below 3%. Palm stearin was selected for this extended stability test as it will rapidly solidify at room temperature even when diluted 1:1.

7 PPM Spiked Palm Stearin Recovery

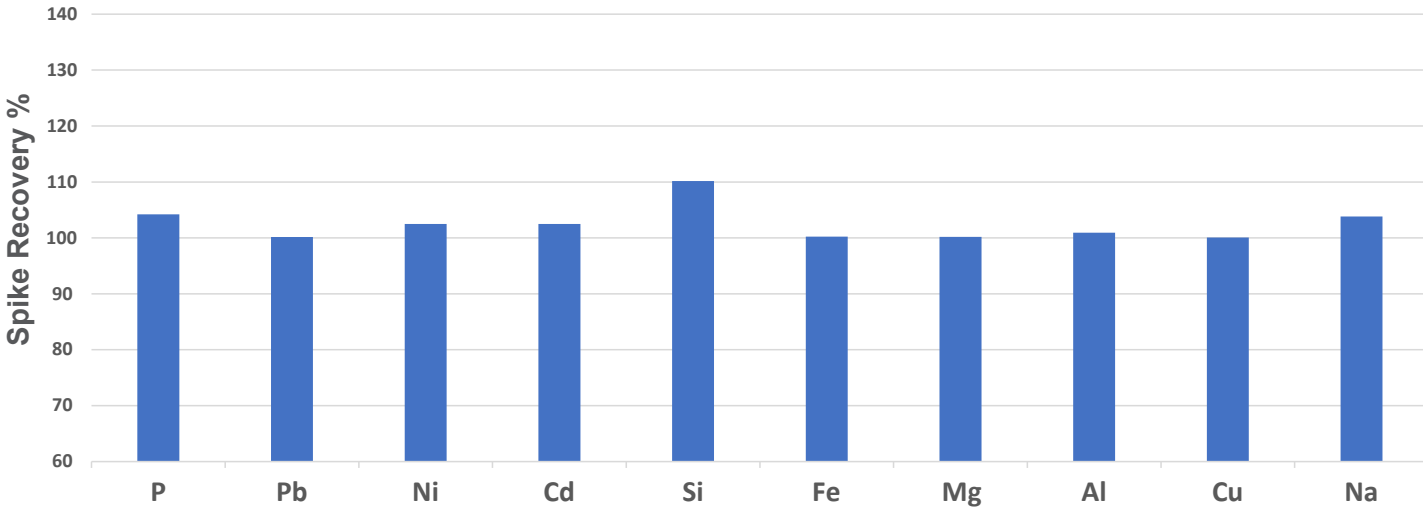


Figure 4. Recovery for 7 ppm spike in 1:1 diluted palm stearin.

Washout After High Calibration Standard

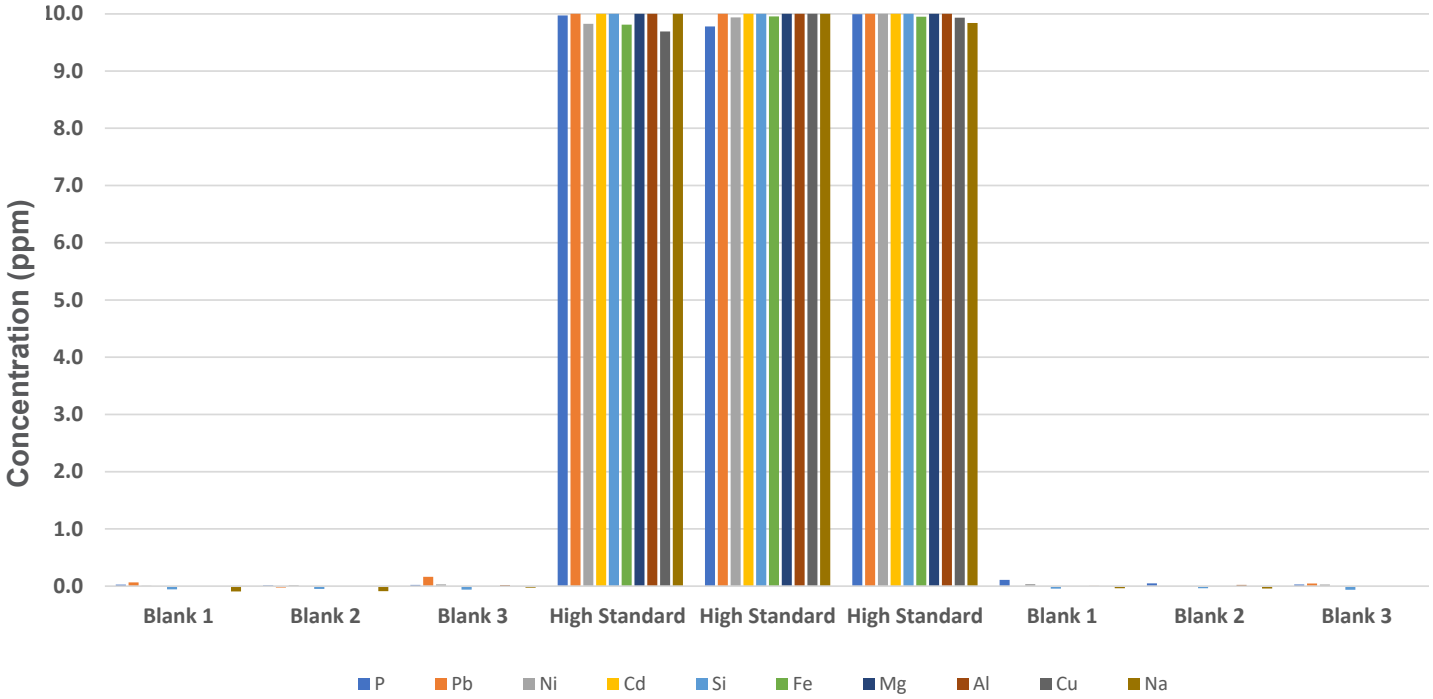


Figure 5. Washout to blank level for all elements from high standard. Three blanks analyzed, then three high standards, followed immediately by three blanks.

Conclusion

EdibleOil ICP optimizes the analysis of edible oils and fats for laboratories by using an automated valve injection sample introduction system combined with a fully heated setup. The heated sample deck, heated sample introduction chamber, and heated lines allow for easier sample preparation, reduced dilution of samples – for better detection limits – and assures sample is liquid from sample deck to ICP to waste – even for solid fats. EdibleOil ICP is a highly effective automation system that improves analytical efficiency, providing accurate determination of trace elements in both edible oils and fats.