Application Brief

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# Elemental Analysis of Palm Oil using ICP-OES

## Evaluation of an Agilent 5110 ICP-OES for the quality control of vegetable oils


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## Increasing demand for palm oil

Palm oil and its derivatives are used in a wide range of products. Some metals present in palm oil can affect the quality of the final products. Therefore, refiners of crude palm oil (CPO) typically carry out elemental analysis at different stages of the refining process as part of the quality control/quality assurance (QC/QA) programs. The samples used in this study were supplied by a customer, who also specified the list of elements (As, Ca, Cu, Fe, Hg, Mg, Mg, P, Pb), and sample preparation procedures.

## Analysis of palm oil samples using ICP-OES

Four palm oil sample types were prepared using two different methods: dilution in xylene and acid digestion. Bleached palm oil (BPO), refined bleached deodorized palm oil (RBDPO), and refined bleached deodorized hydrogenated palm stearin (RBDHPS) were diluted 10 times in xylene, and CPO was diluted 50 times. About 0.5 g of each sample was digested with 6 mL $HNO_3$ and 2 mL $H_2O_2$ using a microwave digestion system held at 175 °C for 10 minutes. The volume of the digests was then made up to 50 mL with de-ionized water before analysis by ICP-OES. Calibration standards for the quantification of the digested samples were prepared in 5% $HNO_3$, while standards for the diluted samples were prepared in xylene.

The Agilent 5110 vertical dual view (VDV) ICP-OES was used for the analysis. The instrument uses a vertically oriented plasma optimized for the robust, long-term analysis of samples with a high solids content or organic matrix. To ensure fast start up and reproducible performance, the 5110 ICP-OES includes a series of ease-of-use features including automatic alignment and connection of all gases and an Easy-fit torch. In this study, the 5110 ICP-OES was fitted with, a double-pass spray chamber, the SeaSpray nebulizer, and standard one piece torch. However, for the analysis of the solvent-diluted samples, a 1.4 mm i.d. semivolatile one-piece torch was used.

The method detection limits (MDLs) shown in Table 1 were based on three sigma of 10 replicate measurements of the calibration blank solutions taken during the analytical run. To calculate the MDLs in the original sample, the MDLs were multiplied by the sample dilution factor (10 times for diluted samples and 100 times for digested samples).

## Quantitative results by ICP-OES

Quantitative results for eight elements in the palm oil samples were obtained by the 5110 VDV ICP-OES. Table 2 shows the results for Ca, Fe, Mg, and P only, since As, Cu, Hg, and Pb were below the MDL.

In addition to the selected elements, non-routine elements can be quickly identified using IntelliQuant, a semiquantitative feature in the ICP Expert software. IntelliQuant can provide data for up to 70 elements, allowing QA/QC labs to monitor additional elements in their samples without changing the method (*1*).

## Spike recovery tests

Diluted samples of BPO and RBDPO were spiked with As and Hg at 0.1 ppm, and other elements at 0.2 ppm. Digested samples were spiked with As at 0.1 ppm, Hg at 0.02 ppm, and other elements at 0.2 ppm. The spike recoveries for all elements in BPO and RBDPO were within ±15%, as shown in Figure 1.

## Simple and effective ICP-OES analysis

The vertical orientation of the 5110 VDV ICP-OES torch and solid-state RF system generate a robust, stable plasma, delivering reliable performance for the direct, routine analysis of palm oil. There was good agreement between the results for the 'diluted' and 'digested' CPO samples, suggesting that they can be prepared for analysis following dilution in a suitable solvent. The 5110 ICP-OES can be used for the quantification of all but the lowest concentration elements (*2*), making it suited to the QA/QC of palm oil products.


## Acknowledgment

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## References


1. Agilent publication, 5994-1516EN
2. Agilent publication, 5994-1953EN


**Table 1.** 5110 ICP-OES MDLs for diluted and digested samples.

| | Diluted | | Digested | |
|---|---|---|---|---|
| Element, Wavelength (nm) | MDL in Solution (mg/L) | MDL in Sample (mg/kg) | MDL in Solution (mg/L) | MDL in Sample (mg/kg) |
| As 188.980 | 0.011 | 0.11 | 0.004 | 0.4 |
| Ca 393.366 | 0.001 | 0.01 | 0.0002 | 0.02 |
| Cu 324.754 | 0.006 | 0.06 | 0.0007 | 0.07 |
| Fe 238.204 | 0.011 | 0.11 | 0.001 | 0.1 |
| Hg 184.887 | 0.011 | 0.11 | 0.002 | 0.2 |
| Mg 279.553 | 0.002 | 0.02 | 0.00004 | 0.004 |
| P 177.434 | 0.025 | 0.25 | 0.006 | 0.6 |
| Pb 220.353 | 0.009 | 0.09 | 0.004 | 0.4 |

**Table 2.** ICP-OES results for elements measured at levels above the LOQ in the palm oil samples (mg/kg).

| | Ca | | Fe | |
|---|---|---|---|---|
| Sample | Dilution | Digestion | Dilution | Digestion |
| CPO | 14.9 | 13.5 | 2.94 | 2.85 |
| BPO | <0.04 | <0.05 | <0.37 | <0.30 |
| RBDPO | <0.04 | <0.05 | <0.37 | <0.30 |
| RBDHPS | <0.04 | <0.05 | <0.37 | <0.30 |

| | Mg | | P | |
|---|---|---|---|---|
| Sample | Dilution | Digestion | Dilution | Digestion |
| CPO | 3.40 | 3.58 | 20.1 | 21.0 |
| BPO | <0.08 | <0.01 | 1.61 | <2.10 |
| RBDPO | <0.08 | <0.01 | 1.08 | <2.10 |
| RBDHPS | <0.08 | <0.01 | <0.83 | <2.10 |

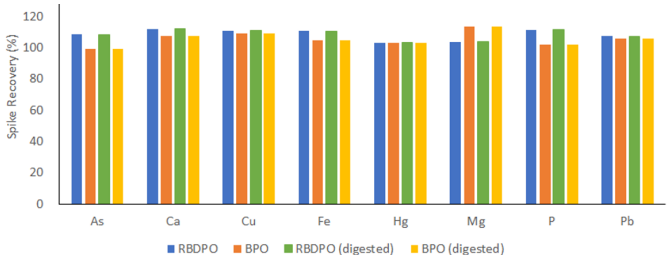


**Figure 1.** Spike recovery results for BPO and RBDPO samples prepared by dilution (blue and orange bars) and acid digestion (green and yellow bars).

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