

**T**his test method quantifies the presence of natrium (Na), magnesium (Mg), aluminium (Al), sulphur (S), potassium (K), calcium (Ca), nickel (Ni) and antimony (Sb) in crude palm oil. These elements may be naturally present in vegetable edible oils which was absorbed mainly from the soil where it was grown. Furthermore, these elements may be introduced into the oil from outer sources of the production process, such as bleaching, refining and deodorisation, or by contamination from metal processing equipment (Zeiner *et al.*, 2005; Jamali *et al.*, 2008). Determination of traces and heavy metals in edible oils/fats, including palm oil, has been dominated by flame/graphite furnace atomic absorption spectroscopy (AAS) and inductively coupled plasma optical emission spectroscopy (ICP-OES). Nevertheless, both analytical techniques have poor detection limits at the level of part per billion (ppb) and part per trillion (ppt) range limits. The application of inductively coupled plasma mass spectrometry (ICP-MS) has the advantage of simultaneous determination of multi-element at a very low detection limit (ppt).

## THE TECHNOLOGY

### Principle

The basic setup of ICP-MS analysis requires the sample introduction as a liquid solution and thus, for solid matrices, an acid digestion procedure is required. Sample of crude palm oil is digested with concentrated nitric acid and hydrogen peroxide in a microwave digestion apparatus. The digest sample is diluted, fortified with internal standards, and analysed using ICP-MS depending on the elements being monitored.

## METHODOLOGY

Figure 1 shows the method procedures for multi-element determination.

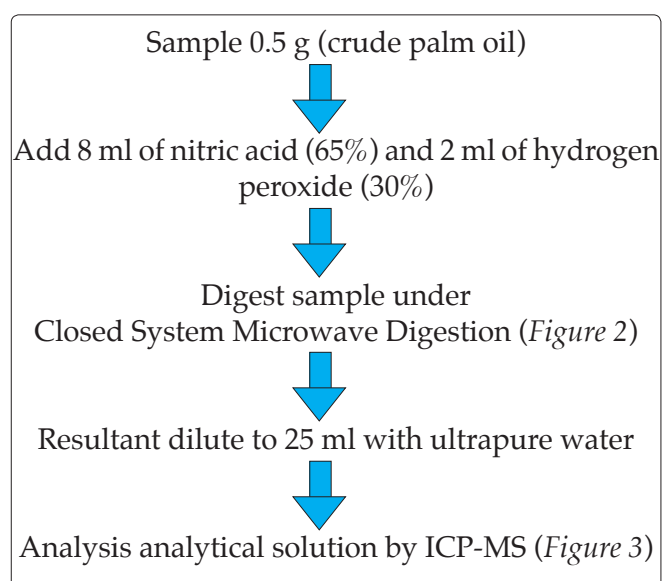


Figure 1. Procedure flow chart for multi-element analysis.

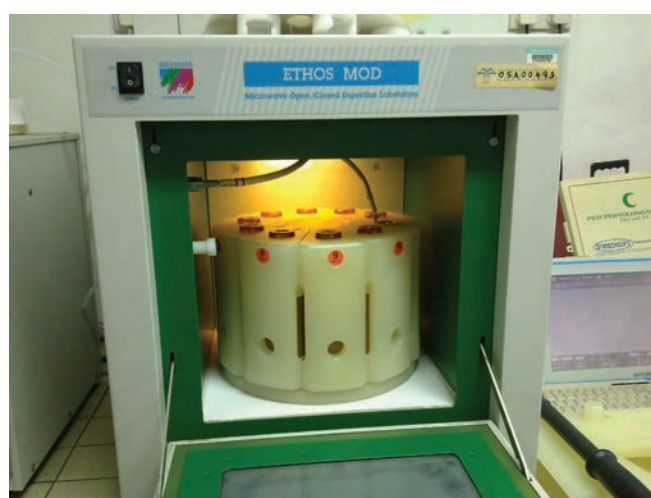


Figure 2. Closed system microwave digester.

## RESULTS

### Quality Control and Quality Assurance Data of ICP-MS Analysis

The performance of this analytical method was evaluated by method validation (Table 1). The method was found to show good linearity, as indicated by the coefficient correlation ( $R^2$ ) of more than 0.9990 for the calibration curve. For most elements studied, the limit of detection (LOD) and limit of quantification (LOQ) were below than  $1 \mu\text{g litre}^{-1}$  and  $2 \mu\text{g litre}^{-1}$ , respectively. The percentage of element recovery from spiked sample at three different concentration levels:  $100 \mu\text{g kg}^{-1}$ ,  $500 \mu\text{g kg}^{-1}$  and  $1000 \mu\text{g kg}^{-1}$  were above 75%. Cross-check study on the performance of analytical methods

showed that the results from MPOB laboratory were comparable to those of private laboratories (Table 2).



Figure 3. Inductively coupled plasma mass spectrometry (ICP-MS).

TABLE 1. ACCURACY AND RECOVERY OF THE METHOD

Element	Limit of detection ( $\mu\text{g litre}^{-1}$ )	Limit of quantification ( $\mu\text{g litre}^{-1}$ )	Spike concentration		
			100 $\mu\text{g kg}^{-1}$	500 $\mu\text{g kg}^{-1}$	1000 $\mu\text{g kg}^{-1}$
			Recovery (%)	Recovery (%)	Recovery (%)
Na	0.59	1.19	101.6	98.2	98.5
Mg	0.22	0.90	100.3	96.6	101.3
Al	0.49	1.09	111.2	100.5	102.1
S	1.02	2.14	94.4	97.0	96.6
K	0.55	1.11	98.4	97.9	91.8w
Ca	0.89	1.27	81.8	95.5	81.3
Ni	0.75	1.21	96.9	99.1	92.7
Zn	0.64	0.81	88.1	96.6	82.9
Sb	0.81	1.21	77.7	80.2	75.8

Note: \*  $\mu\text{g litre}^{-1}$ ;  $\mu\text{g kg}^{-1}$  = ppb.

TABLE 2. CROSS-CHECK RESULTS

Element	MPOB value ( $\mu\text{g g}^{-1}$ )	Lab A detected value ( $\mu\text{g g}^{-1}$ )	Lab B detected value ( $\mu\text{g g}^{-1}$ )
Na	25.47	23.41	26.52
Mg	6.33	5.05	5.88
Al	12.91	13.21	-
S	9.75	9.40	-
K	15.19	14.63	14.84
Ca	10.26	10.84	9.83
Ni	Not detected	Not detected	-
Zn	2.72	2.59	2.51
Sb	Not detected	Not detected	Not detected

Note: \*  $\mu\text{g g}^{-1}$  = ppm (part per million).

## BENEFITS AND ADVANTAGES

- A fast, precise and reliable method for detecting and quantifying sodium, magnesium, aluminium, sulphur, potassium, calcium, nickel and antimony in crude palm oil sample.
- The service will help the palm oil industry monitor potential toxic elements in palm oil in order to prevent a possible element poisoning and to ensure the global public safety.

## CONCLUSION

MPOB offers analytical services for the quantification of sodium, magnesium, aluminium, sulphur, potassium, calcium, nickel and antimony in crude palm oil sample. The minimum amount of crude palm oil sample required is 20 g.

## REFERENCES

Zeiner, M; Steffan, I and Cindric, I J (2005). Determination of trace elements in olive oil by ICP-AES and ETA-AAS: A pilot study on the geographical characterisation. *Microchemical J.*, 81(2): 171-176.

Jamali, M K; Kazi, T G; Arain, M B; Afridi, H I; Jalbani, N; Sarfraz, R A and Baig, J A (2008). A multivariate study: Variation in uptake of trace and toxic elements by various varieties of *Sorghum bicolor* L. J. *Hazard Mater*, 158 (2-3): 644-651.

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