Application Note

Application of ICP-OES to the Analysis of Food and Agriculture: turkey, pork, hay and soy samples

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1 Introduction

The determination of major elements in food, cereals or nutritive solutions is essential for nutritional significance. This necessitates a reliable technique for good accuracy and precision. The ULTIMA 2 ICP-AES spectrometer was used. This paper presents a description of the technique, operating conditions and some results of certified materials.

2 Principle

2.1 Technique used

The elemental analysis of these samples was undertaken by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). The sample is nebulized then transferred to an argon plasma. It is decomposed, atomized and ionized whereby the atoms and ions are excited. We measure the intensity of the light emitted when the atoms or ions return to lower levels of energy. Each element emits light at characteristic wavelengths and these lines can be used for quantitative analysis after a calibration.

2.2 Wavelength choice

The choice of the wavelength in a given matrix can be made using the profile function, or by using Win-IMAGE, which is rapid semi-quantitative analysis mode using multiple wavelengths. The principle is the same in either case: record the scans of analytes at low concentration and of the matrix. By superimposing the spectra, we see possible interferences.

3 Sample Preparation

The preparation was done following the Norm XPV 18-116, Dec 1995. 5 g of sample are calcinated at 550 °C during 6 hours. The ashes are then diluted with 37 % HCl and demineralized water (solution completed to 250 ml). The final concentration of acid is 2.5 %.

4 Calibration and analysis mode

6 multi-element standards were prepared in 2.5 % HCl. The solutions used for calibration are listed in the Table 1 (in mg/L).

5 Instrument specification

The work was performed on a ULTIMA 2 with the specifications shown below.

Table 2: Specification of spectrometer

Parameters	Specifications
Mounting	Czerny-Turner
Focal length	1 m
Optics thermoregulation	Yes
Variable resolution	Yes
Nitrogen purge	Yes
Grating (number of grooves)	2400 gr/mm
Orders	2
1st order resolution	0.005 nm
2nd order resolution	0.010 nm

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Table 1: Calibration standards

Element	STD0	STD1	STD2	STD3	STD4	STD5	Unit
Ca	0	0.002	0.005	0.02	0.04	0.05	%
Cu	0	0.10	0.25	1.00	2.00	2.50	mg/kg
Fe	0	0.40	1.00	4.00	8.00	10.00	mg/kg
K	0	0.002	0.005	0.02	0.04	0.05	%
Mg	0	0.0004	0.001	0.004	0.008	0.01	%
Mn	0	0.20	0.50	2.00	4.00	5.00	mg/kg
Na	0	0.001	0.0025	0.01	0.02	0.025	%
P	0	0.001	0.0025	0.01	0.02	0.025	%
Zn	0	0.20	0.50	2.00	4.00	5.00	mg/kg

Table 3: Specification of RF Generator

Parameters	Specifications
Type of generator	Solid state
Observation	Radial view
Frequency	40.68 MHz
Control of gas flowrate	By computer
Control of sample uptake	By computer
Cooling	Air

The operating conditions of the spectrometer are listed in Table 4.

Table 4: Operating conditions

Parameter	Condition
Generator power	1000 W
Plasma gas flowrate	13 L/min
Auxiliary gas flowrate	0 L/min
Sheath gas flowrate	0.2 L/min
Nebulizer	0.8 L/min at 3 bars
Sample uptake	1 mL/min
Type of nebulizer	Glass concentric
Type of spray chamber cyclonic	Glass
Argon humidifier	No
Injector tube diameter	3.0 mm

6 Wavelength selection and analytical conditions

The acquisition parameters are listed in Table 5.

Table 5: acquisitions parameters

Element	Wavelength (nm)	Measuring point	Calculating point	Integration time (s)	Entrance slit (µm)	Exit slit (µm)	Calculation mode
Ca	317.933	1	1	4	20	80	Max
Cu	324.754	1	1	4	20	80	Max
Fe	259.940	1	1	4	20	80	Max
K	766.490	1	1	4	20	80	Max
Mg	279.079	1	1	4	20	80	Max
Mn	257.610	1	1	4	20	80	Max
Na	589.592	1	1	4	20	80	Max
P	214.914	7	5	2	20	15	Gauss
Zn	213.856	1	1	4	20	80	Max

7 Results

The results are given for various samples in Table 6 to 9. Each sample has been digested twice to have 2 analysis replicates. The obtained concentration is the mean of the 2 calculated concentrations using 3 replicates each. They are compared with results given by the Bureau Interprofessionel d'Etudes Anaytiques (BIPEA). This group organizes inter-laboratory tests in agriculture, food and environmental fields. It allows the laboratories to assess the quality of the analyses. They give concentration results of inter-comparison tests and associated errors at 3 sigma.

Table 6: Soy sample

Element	Obtained concentration (o.c.)	SD	BIPEA concentration (b.c.)	Recovery (%) (o.c. / b.c. x 100)
Ca	0.254 %	0.005	0.27 ± 0.05	94.1
Cu	15.358 mg/kg	0.0.6	16 ± 4	96.1
Mn	36.516 mg/kg	0.17	38 ± 6	96.1
P	0.594 %	0.0085	0.63 ± 0.05	94.3
Zn	46.687 mg/kg	0.23	50 ± 7	93.4

Table 7: Turkey sample

Element	Obtained concentration (o.c.)	SD	BIPEA concentration (b.c.)	Recovery (%) (o.c. / b.c. x 100)
Ca	1.082 %	0.023	1.10 ± 0.13	98.4
Cu	27.586 mg/kg	0.28	28 ± 5	98.5
Fe	256.25 mg/kg	2.67	268 ± 59	95.6
K	1.128 %	0.0015	1.22 ± 0.14	92.5
Mg	0.208 %	0.002	0.22 ± 0.04	95.0
Mn	97.02 mg/kg	1.76	103 ± 12	94.2
Na	0.140 %	0.0035	0.16 ± 0.04	87.5
P	0.757 %	0.012	0.78 ± 0.06	97.1
Zn	83.572 mg/kg	1.63	88 ± 11	95.0

Table 8: Hay sample
These results are concentration on dry mass (d.m.)

Element	Obtained concentration (o.c.)	SD	BIPEA concentration (b.c.)	Recovery (%) (o.c. / b.c. x 100)
Ca	2.90 g/kg d.m.	0.078	3.1 ± 0.5	93.5
Cu	4.7 mg/kg d.m.	0.027	5.1 ± 4.3	92.2
Mg	1.21 g/kg d.m.	0.014	1.3 ± 0.2	93.1
P	2.03 g/kg d.m.	0.078	2.2 ± 0.4	92.3
Zn	25.2 mg/kg d.m.	0.85	26.6 ± 3.8	94.7

Table 9: Pork sample

Element	Obtained concentration (o.c.)	SD	BIPEA concentration (b.c.)	Recovery (%) (o.c. / b.c. x 100)
Ca	0.625 %	0.021	0.65 ± 0.09	96.2
Cu	78.95 mg/kg	0.099	82 ± 10	96.3
Fe	215.79 mg/kg	4.3	221 ± 49	97.6
K	0.67 %	0.004	0.70 ± 0.09	95.7
Mg	0.14 %	0.0015	0.14 ± 0.03	100
Mn	35.91 mg/kg	0.69	39 ± 6	92.1
Na	0.12 %	0.0009	0.14 ± 0.03	85.7
P	0.42 %	0.014	0.44 ± 0.04	95.5
Zn	78.76 mg/kg	4.95	80 ± 10	98.5

8 Conclusion

The use of BIPEA samples allows the validation of the technique and the analytical method that was developed. The calculated recoveries are all acceptable. The ULTIMA 2 used with the AS 421 autosampler allows the user to run food, provender and nutritive samples with the same method. This contributes to the ease of use and reliability of the method and results.

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