

## ICP-Optical Emission Spectroscopy

## Authors:

Yvette Vingerhoets

Henk Lamers

NutriControl BV  
Veghel, Netherlands

Wim van Bussel

PerkinElmer Inc.  
Groningen, Netherlands

Ken Neubauer

PerkinElmer Inc.  
Shelton, CT USA

## Determination of Nutrients in Animal Feed with the Avio 550 Max ICP-OES in Accordance with EN 15621

### Introduction

With the growing importance of domesticated animals as both a food source (i.e. livestock) as well as pets and service animals, their health is important. Healthy, happy pets provide more pleasure to their owners, while service animals play a critical

role helping people with physical and/or emotional limitations. Healthy livestock provides more food per animal, as well as diminishing the possibility of negative health effects being passed along to humans. As a result, it is important to monitor the nutrient content in animal feed.

Nutrients exist in a variety of forms, including biological, organics, minerals, metals, and vitamins, which must be accurately measured to ensure that the feed meets the nutritional needs of the animals. Due to the importance of the analysis, a variety of standardized methods have been developed to ensure accurate nutrient analysis.

Most mineral and metallic nutrients are present at high concentrations and can be measured with atomic absorption (AA), inductively coupled plasma optical emission spectroscopy (ICP-OES), or ICP mass spectrometry (ICP-MS). Considering the benefits and limitations of these three techniques, ICP-OES is perhaps the preferred instrument for performing these analyses as it strikes the best balance between simplicity, ruggedness, matrix tolerance, accuracy, sample throughput, and cost.

PerkinElmer's Avio® 550 Max fully simultaneous ICP-OES was chosen for this work due to its low cost of operation, minimal maintenance requirements and its speed of analysis. The low argon flow of the Avio 550 Max results in significantly less argon consumption due to its proprietary Flat Plate™ plasma technology, leading to a much lower cost per analysis. The Avio 550 Max's ability to handle high levels of dissolved solids running over long periods of time is thanks to PlasmaShear™ technology, generating a thin stream of air which cuts off the top of the plasma, eliminating deposition on the interface window, resulting in exceptional stability in difficult matrices with no maintenance required. And finally, the instrument's advanced optical system provides true simultaneous analysis with excellent stability and accuracy for faster sample-to-sample time.

This work discusses the determination of inorganic nutrients in several types of animal feeds using the Avio 550 Max ICP-OES in conjunction with the European Standard EN 15621 method for the analysis of nutrients in animal feed.<sup>1</sup>

## Experimental

### Samples and Sample Preparation

Five different feed samples were supplied as part of an interlaboratory testing program sponsored by Bipea (Paris, France). Table 1 shows the different samples, the required analytes, and their concentrations. The samples were prepared for analysis following a proprietary preparation technique which meets the requirements of EN 15621.

Calibration standards were prepared in 5% HNO<sub>3</sub> (v/v) at the levels shown in Table 2. Ytterbium (Yb) was added to all standards and samples as an internal standard. All measurements were made against external calibration curves.

### Instrumental Parameters

All analyses were performed on an Avio 550 Max ICP-OES using the conditions and parameters in Table 3 and the wavelengths in Table 4. To minimize matrix effects from the samples and reduce analysis time, all measurements were made using radial view. The analytical time was 30 seconds per sample.

Table 2. Calibration Standards.

Analyte	Standards (mg/L)
Cu	0.05, 0.2, 10
Fe, Mn, Zn	0.05, 0.2, 10, 40
Ca, K, Mg, Na, P	0.05, 0.2, 10, 40, 200

Table 3. Avio 550 Max ICP-OES Instrumental Parameters.

Parameter	Value
Sample Uptake Rate	1.5 mL/min
Nebulizer	SeaSpray™
Spray Chamber	Baffled glass cyclonic
RF Power	1500 W
Injector	2.0 mm alumina
Plasma Gas Flow	10 L/min
Aux Gas Flow	0.2 L/min
Nebulizer Gas Flow	0.65 L/min
Torch Position	-4
Plasma View	Radial
Replicates	3
Read Time Range	1-5 sec
Integration	Auto

Table 4. Wavelengths.

Analyte	Wavelength (nm)
Ca	317.933
Cu	324.756
Fe	238.204
K	766.491
Mg	279.071
Mn	257.610
Na	589.592
P	177.434
Zn	206.197

Table 1. Samples, Analytes, and Assigned Concentrations.

Analyte	Dog Premix (mg/kg)	Fish Meal (mg/kg)	Growing Finishing Pig Meal (mg/kg)	Turkey Feed (mg/kg)	Soya Meal (mg/kg)
Ca	1900	41200	6500	9400	2700
Cu	2670	3	17	12	10
Fe	28090	269	186	140	---
K	1600	11900	7800	9100	21900
Mg	600	2400	1700	2000	---
Mn	2157	7	68	69	25
Na	1900	12800	2000	1100	---
P	700	24100	5700	5700	6500
Zn	38333	74	81	70	50

## Results and Discussion

All feed samples were measured during a single analytical run to assess the robustness of the methodology. Figure 1 shows that the recoveries of each analyte in each sample were within 10% of its assigned value, despite the wide range of concentrations among the different feed samples. By performing all measurements in radial view, potential matrix effects of the samples were removed, allowing for accurate results between the five different feed samples without the need to matrix match the calibration standards.

With the accuracy of the methodology established, stability was assessed by measuring the turkey feed sample 105 times over 3 hours. Figure 2 shows the measured concentrations for each analyte over the analysis. Despite the wide concentration variations between the elements, all recoveries are within 10% of their assigned values for each sample. By using auto integration and the default read times, the analytical time for each sample is only 30 seconds. Sample-to-sample time could be decreased with the use of the High Throughput System (HTS) flow-injection sample introduction module, while the analytical time can be decreased by using a lower minimum read time.

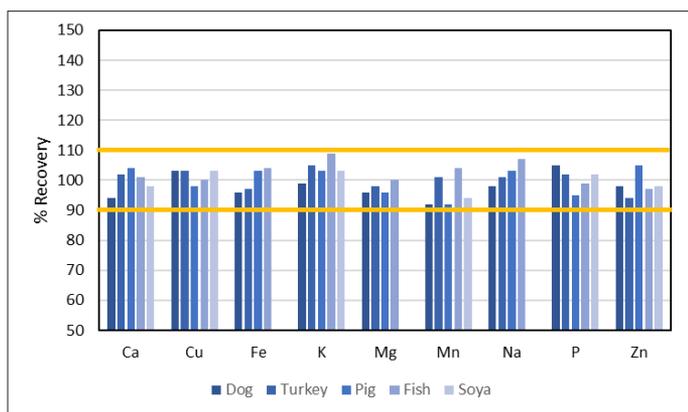


Figure 1. Analyte recoveries in five different feed samples.

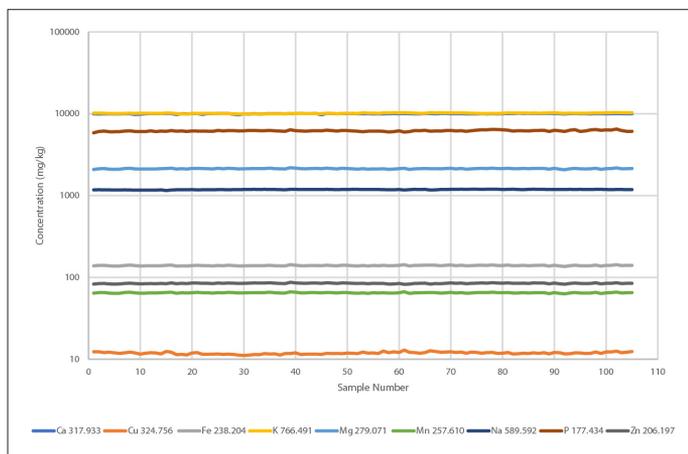


Figure 2. Three-hour stability run of turkey feed.

PerkinElmer, Inc.  
940 Winter Street  
Waltham, MA 02451 USA  
P: (800) 762-4000 or  
(+1) 203-925-4602  
[www.perkinelmer.com](http://www.perkinelmer.com)

## Conclusions

This work has demonstrated the ability of the Avio 550 Max ICP-OES to accurately and rapidly measure nutrients in animal feed samples which meets the requirements of EN 15621. By performing all measurements with a radial plasma view, matrix effects are minimized, allowing the nutrients in five types of feed samples to be measured over a wide concentration range without the need to matrix-match the calibration standards. The Avio 550 Max fully simultaneous ICP-OES delivers fast analysis time of 30 seconds to meet the demands of high-throughput labs without sacrificing analytical accuracy.

## References

1. European Standard EN 15621, EN 15621:2017 E, European Committee for Standardization (CEN), 2017.

## Consumables Used

Component	Part Number
Sample Uptake Tubing, Black/Black (0.76 mm id), PVC, Flared	N0777043
Drain Tubing, Red/Red, (1.14 mm id), PVC	09908585
SeaSpray™ Nebulizer	N0811306
Copper Standard, 1000 mg/L	N9300183 (125 mL) N9300114 (500 mL)
Iron Standard, 1000 mg/L	N9303771 (125 mL) N9300126 (500 mL)
Manganese Standard, 1000 mg/L	N9303783 (125 mL) N9300132 (500 mL)
Ytterbium Standard, 1000 mg/L	N9303811 (125 mL) N9300166 (500 mL)
Zinc Standard, 1000 mg/L	N9300178 (125 mL) N9300168 (500 mL)
Multielement Standard B: 10,000 mg/L Ca, K, Mg, Na, P – 125 mL	N9308546

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