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SampleSense *FAST* – Analysis of Inorganic Nutrients in Animal Feeds Using Method EN 15621

Abstract

Determination of inorganic nutrients in various forms of animal feed is well defined in the European Standard Method EN 15621. Due to the increased prevalence of domesticated pets and enhanced scrutiny on the quality of livestock-derived food, laboratories need to rapidly and effectively determine the elemental composition of animal feed. The presence of minerals and nutrients such as Ca, Na, P, Mg, and K at high concentrations makes ICP-OES the ideal technique for analyzing animal feed, but traditional sample introduction systems sometimes have difficulty analyzing these high-matrix samples. The addition of an automated sample introduction system with sample detection capability improves analytical performance by automatically accounting for variable sample viscosity, detecting missed sample events and minimizing sample consumption. This application note demonstrates the SampleSense *FAST* with the Avio 500 ICP-OES using the European Standard Method EN 15621 for the determination of nutrients in animal feed.

Figure 1. SampleSense valve

1. Elemental Scientific, Inc., Omaha, NE, USA
2. PerkinElmer at Nutricontrol Netherlands, Veghel, Netherlands

Sample Loading Sequence for the SampleSense *FAST* with the Avio 500

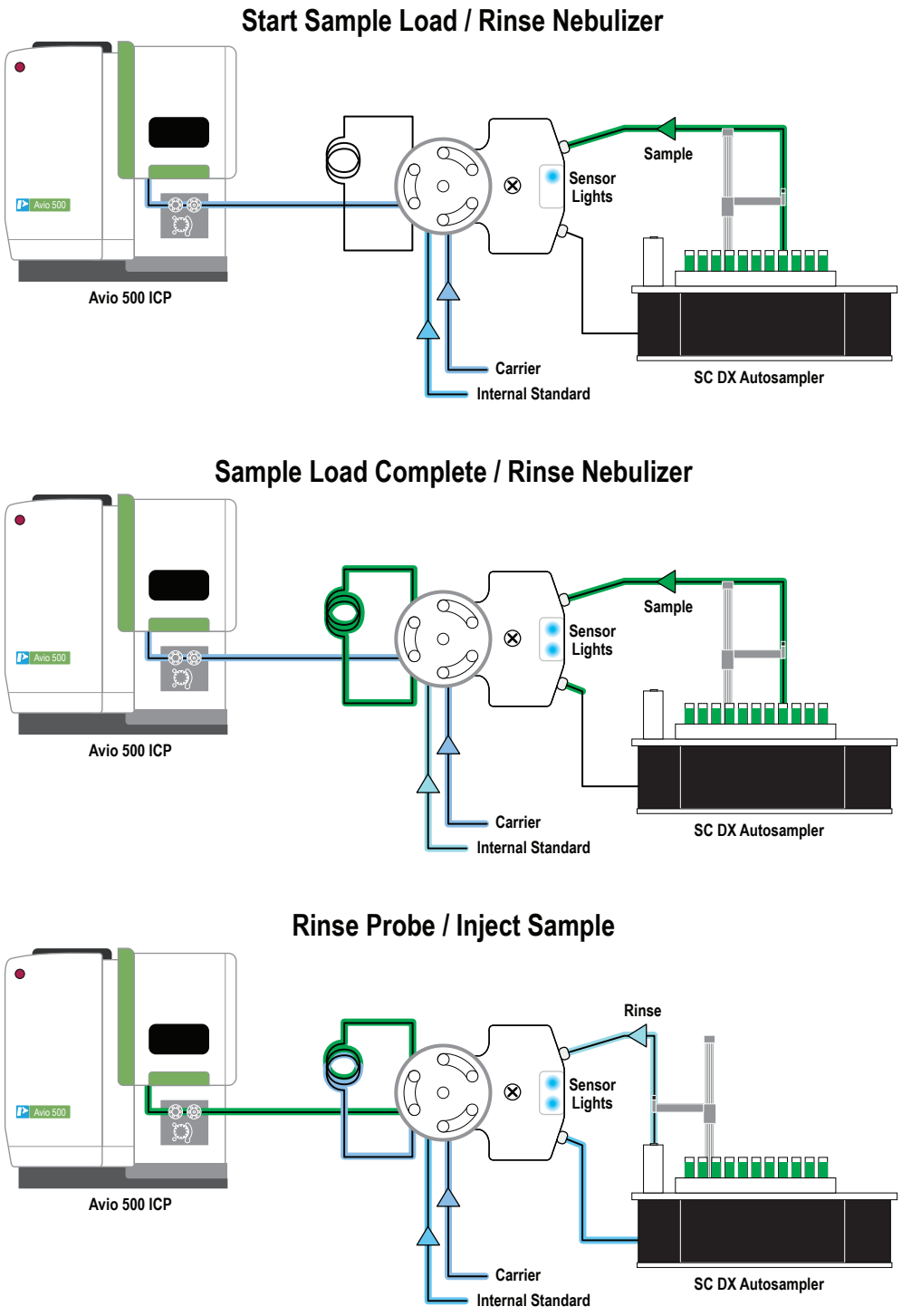


Figure 2. The SampleSense integrated optical sensors automatically detect the presence of a non-segmented liquid sample as it is quickly vacuum loaded and as the nebulizer is rinsed. The sensed sample is automatically injected from the loop and the ICP analysis is triggered in a tightly timed analytical sequence. As the sample is being injected, the autosampler probe is rinsed.

SampleSense *FAST*

SampleSense *FAST* combines an autosampler and inert sample valve with integrated optical sensors that automatically detect the presence of a non-segmented liquid sample as it is quickly vacuum loaded onto a sample loop. The sensed sample is automatically injected from the valve loop and the ICP analysis is triggered in a tightly timed analytical sequence (Figure 2).

SampleSense eliminates method customization and the need to adjust valve timing parameters to account for differences associated with variations in sample viscosity, simplifying the analysis of multiple, variable sample types and volumes that are often placed on the same autosampler deck.

With SampleSense, throughput is enhanced and the presence of the sample at the valve is positively confirmed prior to sample injection, adding a higher level of authentication to analytical results. SampleSense also detects and reports sample loading failures. Unsensed samples – for example, empty or capped tubes – are identified and logged, saving the operator the time and hassle of deciphering ICP data from non-sample events.

Instrumentation

All samples were analyzed using SampleSense *FAST* in combination with an Avio 500 ICP-OES.

Features:

- Automatic sensing, injection, triggering of ICP analysis
- Detection and reporting missing or empty sample tubes as "unsensed" samples
- Integrated valve assembly
- Automatic compensation for viscous samples and those containing particulates

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 using the techniques outlined in EN 15621.

Calibration standards were prepared in 5% HNO_3 (v/v) at the levels shown in Table 4. Ytterbium (Yb) was added to all standards and samples as an internal standard. All measurements were made against external calibration curves.

Table 1. Samples, Analytes, and Assigned Concentrations (mg/kg)

Analyte	Dog Premix	Fish Meal	Growing Finishing Pig Meal	Turkey Feed	Soya Meal
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

Experimental Conditions

Instrumental Parameters

All analyses were performed on an Avio 500 ICP-OES using the conditions and parameters in Table 2 and the wavelengths in Table 3. To minimize matrix effects from the samples and reduce analysis time, all measurements were made with radial view.

Figure 3. SampleSense *FAST* on the Avio 500

Table 2. Avio 500 instrumental parameters

Parameter	Value
Sample Uptake Rate	1.5 mL/min
Nebulizer	PFA-ICN
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 s
Integration	Auto

Table 3. Wavelengths

Element	Wavelength (nm)
Ca	317.933
Cu	324.756
Fe	239.562
K	766.491
Mg	285.213
Mn	259.373
Na	589.592
P	177.434
Zn	206.197

Confirmation of Sample Loading with SampleSense

The integrated optical sensors in the SampleSense valve provide positive confirmation to the laboratory that each sample was properly dispensed into its vial and subsequently loaded into the valve for analysis. If a sample container is either underfilled or empty, the SampleSense valve reports this unsensed sample event to the software and does not inject the contents of the sample loop. To also provide confirmation of a missed sample in the raw data of the ICP, the addition of a marker element in the carrier solution can be added to provide a direct marker in the data. SampleSense automatically responds to any unsuccessful sample loading event by triggering the ICP analysis without injecting the sample loop contents, resulting in the analysis of the marked carrier solution. The presence of this marker (Sc for example) in the ICP data provides additional confirmation to the analyst that a sample was not introduced successfully.

Table 4. 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

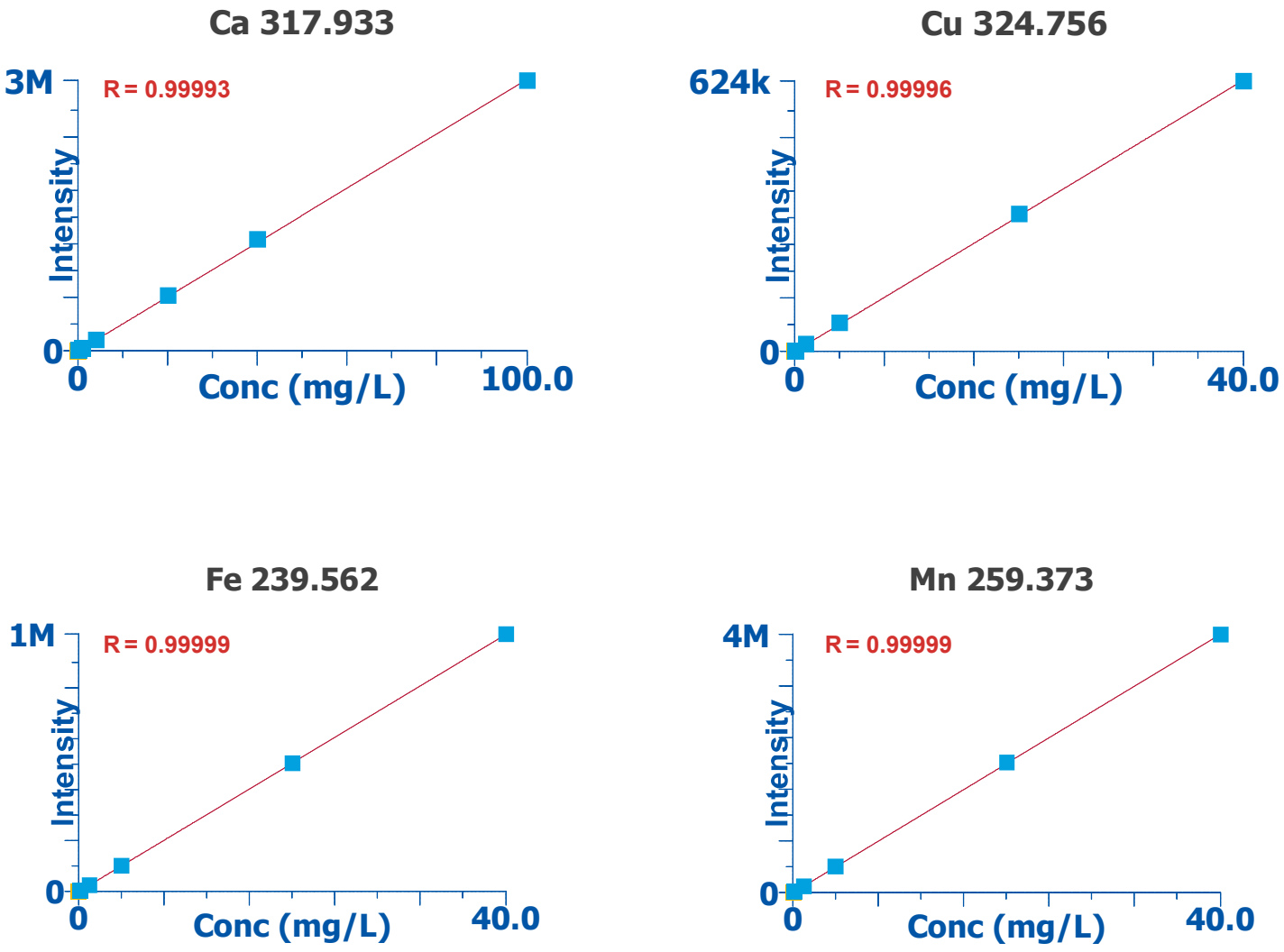
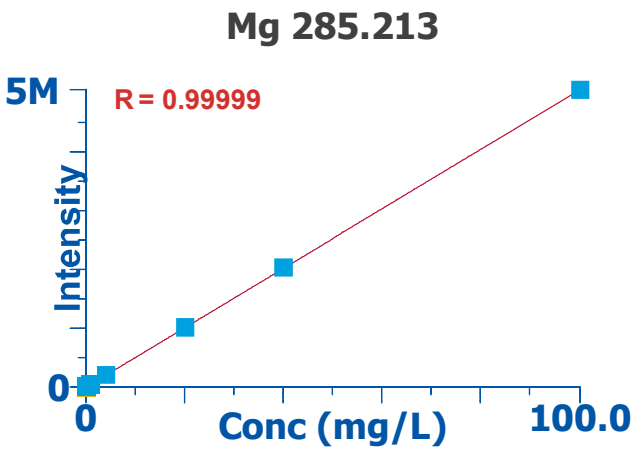
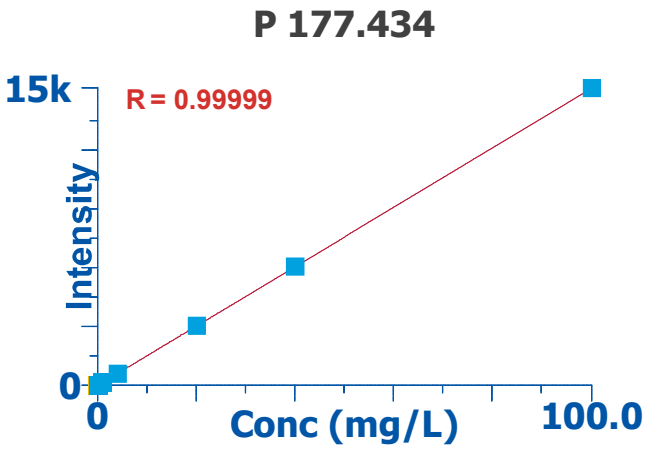
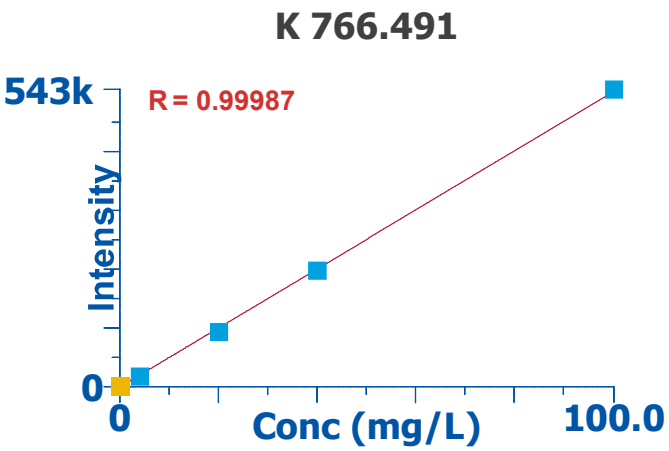


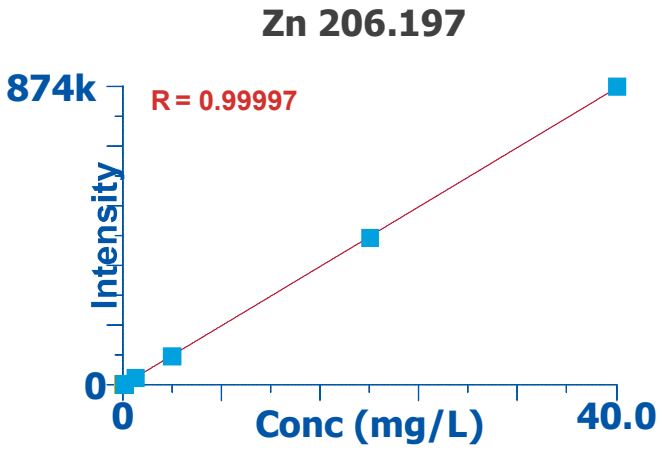
Figure 4. Automated calibration curves (continued on next page)

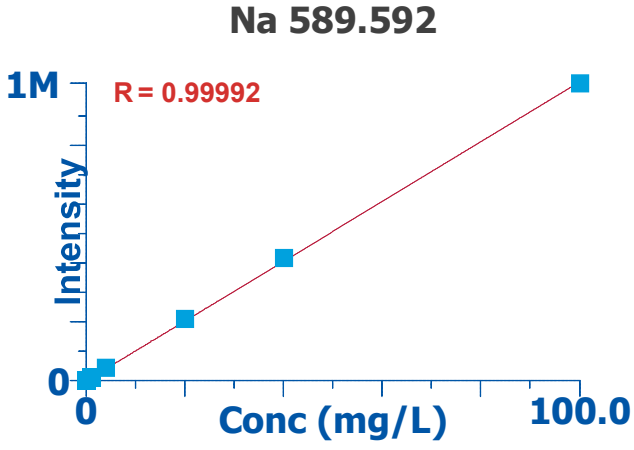
Excellent Linearity in Automatically Calibrated Curves











Results

Excellent recoveries between 90% – 110% of the reference values for all analytes in the five sample types are shown in Figure 5. Average recovery for each analyte in the five animal feeds is found in Table 5.

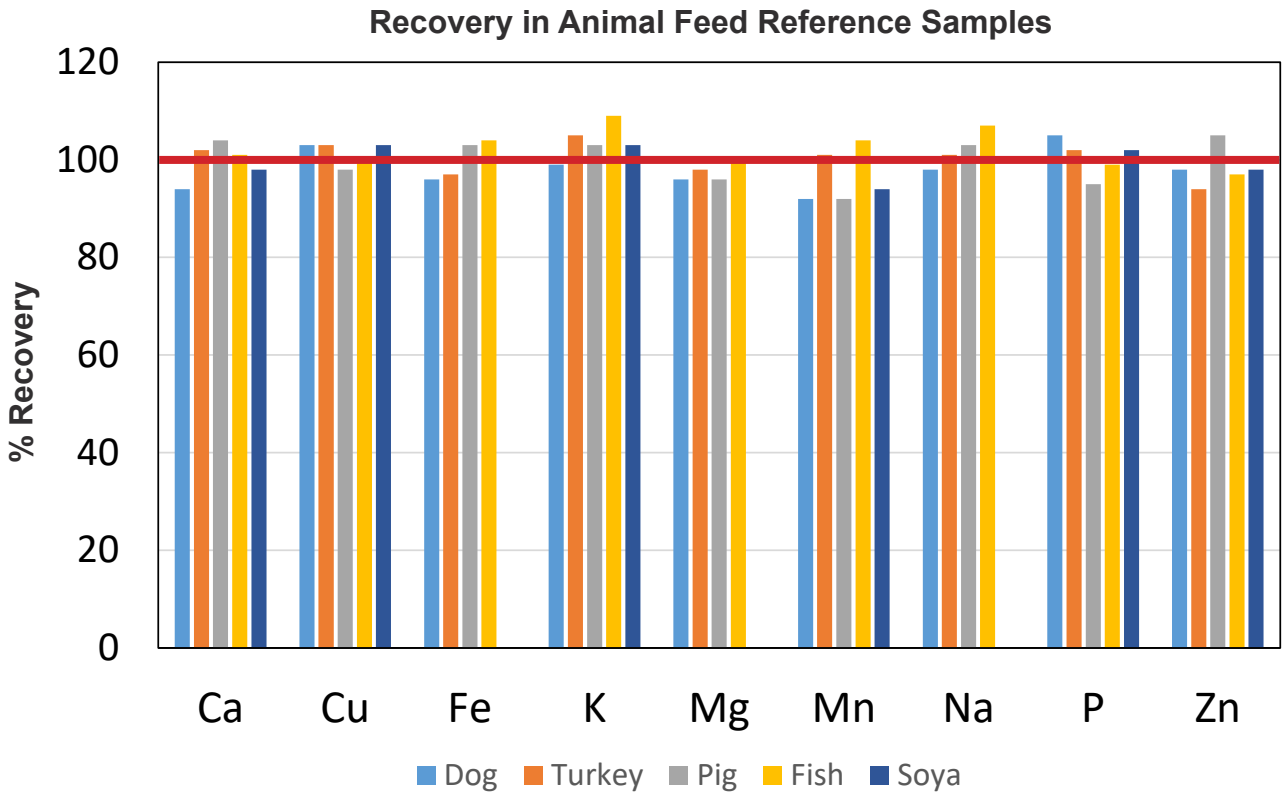


Figure 5. Percent recoveries of each micronutrient in five animal feeds

Table 5. Average percent recovery for each micronutrient in five animal feeds

Element	Ca	Cu	Fe	K	Mg	Mn	Na	P	Zn
Average Recovery (%)	100 ± 4	101 ± 2	100 ± 4	104 ± 4	98 ± 2	97 ± 6	102 ± 4	101 ± 4	98 ± 4

Conclusion

SampleSense *FAST* with the Avio 500 ICP-OES optimizes the analysis of animal feed in high-throughput laboratories using an advanced, automated valve injection sample introduction system. The integrated optical sensors maximize laboratory productivity and reduce needless sampling errors by eliminating valve timing parameters and method adjustments that account for variability of sample viscosity. They also provide positive confirmation to the laboratory that each sample is loaded into the loop for analysis. If a sample container is either underfilled or empty, the SampleSense valve logs this unsensed sample event and does not inject the contents of the sample loop. SampleSense *FAST* improves analytical efficiency in a production laboratory environment, providing a higher level of data authentication and analytical integrity.

Description	Avio 500 Part Numbers
SampleSense *FAST* 2DX	2F-SS7-37
SampleSense *FAST* 4DX	4F-SS7-37
SampleSense *FAST* 8DX	8F-SS7-37
SampleSense *FAST* 14DX	14F-SS7-37