



X-RAY FLUORESCENCE

Pet Food Quality & Safety: From Raw Material to Final Product

Lab Report 182

Introduction

When it comes to pet food, consumers expect top quality for their loved ones. Without access to an adequate diet, pets can experience health and welfare problems. Each cat, dog or horse breed has its own unique nutritional needs, which even change with the pet's age. It is important that the pet food we produce meets these requirements. This makes production and quality control essential tasks. State-of-the-art analytical solutions help pet food producers ensure that their products are safe, nutritious and of the highest quality. The S2 PUMA Series 2 Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer is such a solution. It is used for rapid and accurate elemental analysis along the

entire production process, from raw material identification to intermediate and final product quality control.

One analytical task for the S2 PUMA is to ensure the correct contents and ratios of macro and micro mineral nutrients. Mineral nutrients play a crucial role in various physiological processes such as maintaining skeletal structure, fluid balance, cellular function, nerve conduction, and muscle contraction. This lab report highlights the performance of the S2 PUMA for dog and equine feed quality control.

Mineral Nutrients:
The right amounts and ratios are key!

Minerals are essential for the overall health and well-being of pets. Mineral nutrients can be separated into macro- and micronutrients:

Macronutrients are Sodium (Na), Magnesium (Mg), Phosphorous (P), Sulfur (S), Chlorine (Cl), Potassium (K), and Calcium (Ca). Calcium and Phosphorous, for example, are inevitable for bones and teeth. Sodium, Potassium, and Chloride are important electrolytes and involved in maintaining the osmotic balance.

Micronutrients include Manganese (Mn), Iron (Fe), Copper (Cu), Zinc (Zn), and more. Iron is essential for oxygen transport (hemoglobin and myoglobin). Zinc is vital for e.g. the immune function, growth, and wound healing.

Not just deficiencies but also excesses of minerals can lead to various health issues. Copper excess, for instance, can damage the liver and can cause oxidative stress. High Magnesium causes diarrhea, weakness, and impaired nutrient absorption.

Why using XRF for pet food analysis?

The main benefits of using XRF over other techniques such as inductively coupled plasma optical emission spectroscopy (ICP-OES) are ease-of-use, short time-to-result, long-term stability, and low costs of operation. XRF requires minimal sample preparation and can provide results in a matter of minutes after sampling. Moreover, XRF spectrometers do not need expensive or hazardous consumables or labware and can be operated 24/7 – no daily re-calibration necessary! Thanks to Bruker's intuitive spectrometer software, SPECTRA.ELEMENTS, the operation is very easy and requires just minimal training.

Which materials can be analyzed by XRF?

Any raw material, intermediate product and final feed can be analyzed by XRF: solids, liquids, powders, pellets, granules. This includes, mineral premixes, dry ingredient blends, forages, total mixed rations, hay, and, of course, final feed pellets. Even milk, urine, and feces can be analyzed.

Optimal spectrometer configuration

The S2 PUMA Series 2, a versatile, high-performance benchtop EDXRF spectrometer, is an excellent solution for the pet food industry. The optimized beam path, the 50-Watt power X-ray tube, and the HighSense™ XP Silicon Drift Detector (SDD) ensure short time-to-results in combination with outstanding analytical performance.

The XY-Autochanger enables high sample throughput while remaining completely flexible: You can load mixed batches (liquids, powders, solids ...) and load, unload or prioritize new samples at any time. The vacuum mode results in best sensitivity for lighter elements like Na and Mg, and lowest costs of operation. And Bruker's unique SampleCare™ technology protects important system components, guaranteeing high instrument uptime even in dusty environments.



Figure 1
S2 PUMA Series 2 with
XY-Autochanger for
highest flexibility and
sample throughput

Reference Materials, Sample Preparation & Calibration

To show the performance of the S2 PUMA for pet food applications, a set of 28 secondary reference standards from AAFCO (Association of American Food Control Officials) was used for calibration. Feed materials covered by the broad range calibration:

Pet food types: Dog feed, dry cat feed, dry dog feed, horse mineral mix, equine feed, rabbit feed.

Cattle feed types: Infant pig starter, senior pig starter, chicken starter, calf starter, beef feed, sheep grower, goat feed.

The concentration ranges are summarized in Table 1.

The samples were already finely ground. The samples were pressed into pellets and measured in vacuum mode with sample rotation (see Table 2). Exemplary calibration curves are displayed in Figure 2, revealing a robust method even for a broad range of feed materials.

Table 1

Concentration range covered by the broad pet food calibration.

Type	Element	Min content	Max content
Macronutrients	Na (%)	0.13	5.32
	Mg (%)	0.06	1.90
	P (%)	0.24	8.58
	S (%)	0.17	1.71
	Cl (%)	0.04	8.71
	K (%)	0.21	2.91
	Ca (%)	0.04	13.52
Micronutrients	Al (PPM)	28	3674
	Mn (PPM)	7	1076
	Fe (%)	0.01	0.69
	Cu (PPM)	9	269
	Zn (PPM)	56	3348

Table 2

Measurement parameter.

Elements	Voltage	Current	Filter	Counting time	Mode	Rotation
Na, Mg, Al, P, S, Cl	10 kV	Automatic	none	200 s	Vacuum	On
K, Ca, Mn, Fe, Cu, Zn	40 kV	Automatic	Al (500 µm)	120 s	Vacuum	On

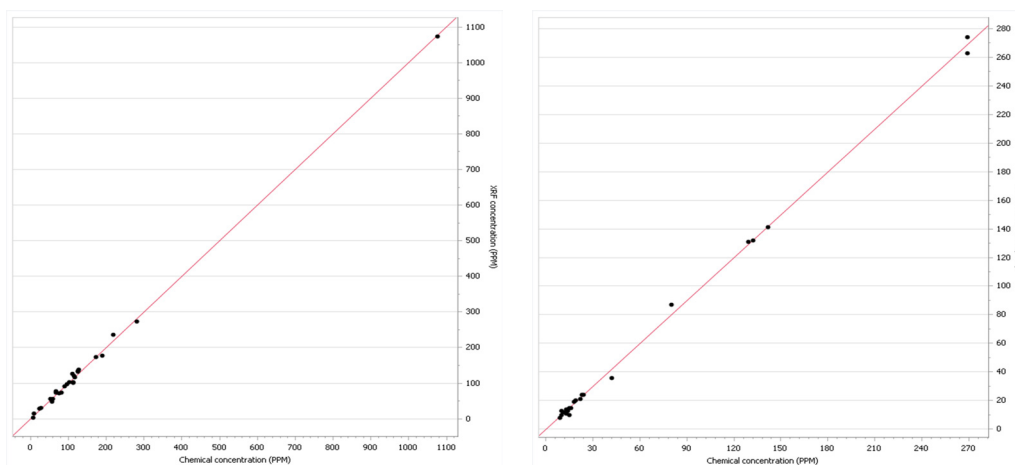


Figure 1

Calibration curves for Mn KA, $R^2 = 0.99834$ (left) and Cu KA, $R^2 = 0.99859$ (right).

Results

Repeatability tests were conducted using dog feed and equine feed. The results are listed in Table 3 and 4, showing an excellent precision for all 12 macro- and micronutrients covered by the calibration. The time to result – including sample preparation and automatic sample loading & unloading – is <10 minutes and can be reduced further if required.

Table 3

Repeatability of dry dog feed analysis.

Dog Feed	Na (%)	Mg (%)	Al (PPM)	P (%)	S (%)	Cl (%)	K (%)	Ca (%)	Mn (PPM)	Fe (PPM)	Cu (PPM)	Zn (PPM)
Rep-1	0.215	0.259	86	1.060	0.219	0.415	0.846	1.172	76	253	13	186
...
Rep- 10	0.225	0.259	68	1.040	0.217	0.410	0.814	1.168	74	251	14	186
Average	0.222	0.264	86	1.054	0.219	0.415	0.816	1.163	75	253	13	186
Std. Dev.	0.005	0.005	7	0.007	0.001	0.002	0.014	0.007	2	2	0	2
Rel. Std.	2.5%	2.0%	8.7%	0.7%	0.4%	0.5%	1.7%	0.6%	3.1%	0.6%	3.6%	0.9%

Table 4

Repeatability of equine feed analysis.

Equine Feed	Na (%)	Mg (%)	Al (PPM)	P (%)	S (%)	Cl (%)	K (%)	Ca (%)	Mn (PPM)	Fe (PPM)	Cu (PPM)	Zn (PPM)
Rep-1	0.297	0.384	348	1.150	0.337	0.696	1.324	1.725	179	431	131	386
...
Rep- 10	0.292	0.382	347	1.140	0.334	0.692	1.302	1.713	174	429	128	375
Average	0.295	0.386	345	1.149	0.336	0.695	1.322	1.722	176	432	130	383
Std. Dev.	0.009	0.004	6	0.006	0.001	0.002	0.011	0.008	4	3	1	3
Rel. Std.	3.0%	1.0%	1.9%	0.5%	0.4%	0.3%	0.9%	0.5%	2.1%	0.7%	1.0%	0.9%

Conclusion

This lab report demonstrates the excellent suitability of the S2 PUMA for rapid and easy production and quality control in the pet food industry – 24/7/365! With XRF all relevant raw materials, supplements, and feed products can be measured with or without just minimal sample preparation.

Bruker AXS

info.baxs@bruker.com

bruker.com

Worldwide offices
bruker.com/baxs-offices

Online information
bruker.com/s2puma

