Chapter 4

Some Aspects of Animal Feed Sampling and Analysis

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Additional information is available at the end of the chapter

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Abstract

Animal feed plays an important part in the food chain and the composition and quality of the livestock products (milk, meat and eggs) that people consume. Animal feeds are either classified as fodder, forage, or mixed feeds. Fodders could be classified as roughages (fresh cut forage, hay or dry forage, straw, root crops, stover and silage) and concentrates such as grains, legumes and by-products of processing. Safety is perhaps one of the most important reasons for feed analysis by the manufacturers and consumers. Storage duration and conditions for feed samples, as well as of stable and unstable parameters are important in sample preparation. A number of sub-samples for preparing final sample for various categories of feed products are recommended. Some analysis conducted on feed include; dry matter, crude ash, ash insoluble in acid (sand), crude protein, crude fat, fibre analysis, starch, gross energy, minerals. More are amino acids (excluding tryptophan), amino acids (tryptophan), fatty acids, vitamins, reducing sugar, mycotoxins, and pesticides. Various types of samples depending on their purposes and uses are available from check, standard, working and referee samples to composite types. Sampling errors in procedures exists and can be minimized by standards or purposes of the analysis, appropriate sampling equipment and using the right quantity of materials.

Keywords: animal, feed, sampling, analysis, quality control

1. Introduction

Food is any substance, originating from plants, animal or any other source, consumed by any organism for the purpose of providing nutritional support. When consumed and assimilated, food is used in the body to maintain and repair body tissues, promote health and growth, sustain life, provide energy, for reproduction and other vital body processes through the release of its nutrients. Essentially, the basic nutritive components of food are carbohydrates, proteins, fats, minerals, vitamins and water, which are absorbed in the body in various usable forms.

Food given to food-producing animals, whether made up of single or multiple materials, are generally referred to as feed or feedstuff, and could be fed as raw, semi-processed or processed [1]. Feeds may be live organisms, particularly in the production of aquatic organisms. Animal feeds are either classified as fodder, forage, or mixed feeds. Fodders could be classified as roughages (fresh cut forage, hay or dry forage, straw, root crops, stover and silage) and concentrates such as grains, legumes and by-products of processing. Plant materials consumed by grazing animals either directly as pasture, crop residue, and immature cereal crops are referred to as forage. However, forage materials cut as fodder, particularly fresh, hay, and silage are sometimes loosely referred to as forage. Mixed feeds are produced from several feed ingredients combined in different proportions to achieve a particular nutritional quality. Feed ingredients, including additives, may or may not add any nutritional value to the mixed feed and comprises of components originating from plant, animal, or aquatic sources, which could be organic or inorganic in nature [1]. Several ingredients used for the production of feedstuff are limiting in one or more nutrients, and must therefore be blended in appropriate proportions to meet the nutritional requirements of the animals. Mixed feeds are usually produced in the form of mash or pellets.

Animal feeds are important, not only to the feeds manufacturers and animal producers, but also to the regulators, policy makers, processors and the final consumers of the end-products. This is because animal feed is an integral part of the food supply chain and it is critical to the efficient and profitable production of quality and safe food. Thus, feed safety is critical to food safety. Stakeholders interested in producing safe foods must be, and are rightly, concerned with the safety of animal feeds. Research evidence regarding risks associated with consumption of contaminated feeds and several epidemics which were traceable to animal feeds in different countries have made the demands for safe feed even more serious in recent times [2]. Ingredients, suppliers and processing methods used in the process of feed production may significantly impact public health [3]. As part of the measures to ensure that feed ingredients and feedstuffs meet the various quality and safety requirements, a wide range of analyses, both scientific and socio-economic, are carried out in the feed and food industry. Some of the reasons stakeholders carry out feed analysis are for regulations and enforcement, recommendations, labelling, validation of manufacturers' quality claims, feed/food safety and defence, quality control in feed production, and for research and development. Adoption of standard sampling and analytical methods assist in accurately characterizing the problems and contribute to the integrity of the results. This chapter focuses on some aspects of analysis in the feed industry to ensure the production of nutritious and safe food animals.

2. Animal feed and food supply chain

Every step from primary production to final consumption, that is, from farm to fork, makes up the food chain. Feed production, plays significant role in the production of food of animal origin and it is, therefore, a critical aspect of the food chain (**Figure 1**). Therefore, all key actors on every nodes of the food chain are responsible for the production of safe, healthy and nutritious feeds.

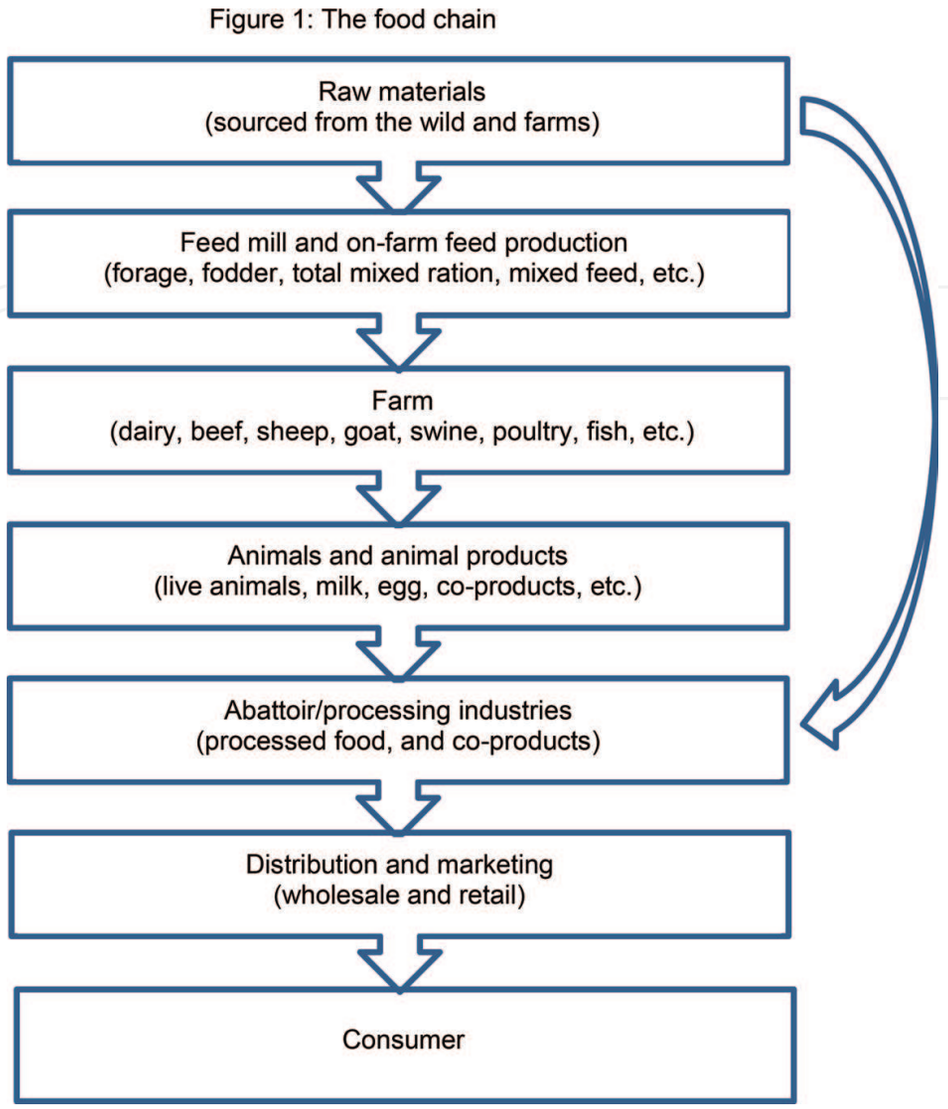

Figure 1. The food chain.

3. Feed hazards

Food safety hazards connected with feed safety has heightened the concern and level of seriousness given globally to feed analysis in recent times. Contaminants in feeds can be inherent or naturally occurring such as mycotoxins and heavy metals, or industrial substances such as polychlorinated biphenyls (PCBs) and pesticides [4]. Feed contaminants, as in the case of food contaminants, can also be biological, chemical, and physical (radionuclides) [5, 6]. Three important criteria used for selecting hazards of current importance in feeds are relevance of hazards to public health; extent of occurrence of the hazard; and impact of the hazard on international trade in food and feed [7].

Codex Alimentarius standards for contaminants in feeds and foods applies to substances with food and feed significance but no public health significance, pesticides residues, residues of

veterinary drugs and feed additives, microbial toxins, and residuals of processing aids [8]. Related with each hazard are specific sources and routes of contamination and exposure, which may be deliberately or accidentally introduced along the feed production value chain. Examples of sources of hazards that may be present in animal feeds and feed ingredients are presented in **Table 1**.

It is worthy of note that among a wide range of sources, feedstuffs especially those of plant origin are the most common potential means of exposing animals to toxic levels of minerals [23]. However, feedstuff of animal origin can contain potentially toxic levels of some minerals (**Table 2**). Toxic levels of minerals in feedstuff, which may lead to death of the animals, may occur as a result of utilizing feed ingredients sourced from areas with high concentrations of heavy metals, processing methods, feed formulation and manufacturing errors, and contamination during storage or transportation.

Type of hazard	Causative substance/agents	Sources in animal feeds	Analytical method
Physical hazards	Glass, metals, plastic and wood	Handling at various stages of production and processing	Physical inspection
Chemical Hazards	Dioxins, dibenzofurans, dioxin-like PCBs	Contaminated mineral sources, food by-products, fish by-products	Gas chromatography-high resolution mass spectrometry (GC/HR-MS); gas chromatography with other lowre solution mass spectrometry instruments; Calux-assay methods
	Mycotoxins – Aflatoxin B1, ochratoxin A, zearalenone, fumonisin B1, deoxinivalerol, T-2, HT-2	Cereals (especially maize), cotton seed, peanut (groundnut), copra, distillers' dried grains with soluble (DDGS)	Semi-quantitative ISO method based on thin-layer chromatography [11] and a methods applying HPLC with fluorimetric detection after immuno-affinity clean-up; dipstick-like immunochemical screening methods are also applied; Official methods of [12].
	Veterinary drugs	Terrestrial and aquatic-based feed ingredients, medicated feeds, DDGS	HPLC methods; Enzyme-Linked ImmunoSorbent Assay (ELISA); Microbiological inhibition assays; LC-MS/MS or liquid chromatography with diode array detector (LC-DAD) methods; official AOAC method [13].
	Organopesticides – DDT, hexachlorobenzene and aldrin.	Contaminated feed ingredients and feeds	GC-MS; GC with electron-capture detection (ECD) methods
Microbial contaminants	Brucella, salmonella, endoparasites (Echinococcus, *Toxoplasma gondii*, Cisticercus and Trichinella)	Contaminated pasture, forages, and animal and vegetable protein meals.	Methods of [11, 14–22]

Source: Adapted from Refs. [9, 10].

Table 1. Sources and analytical methods for detection of some chemical and microbiological hazards in feeds.

Mineral	Major sources	Animal health concerns	Analytical method	Difficulties with analysis
Undesirable heavy metals				
Arsenic	Sea plants, fish products; and supplemental minerals.	Medium	Hydride Atomic absorption spectrophotometry (AAS); Plasma mass spectrometry; Graphite Furnace AAS; and Silver diethyldithiocarbamate colorimetric methods.	Incomplete extraction; Retention time irreproducibility; Co-elusion of species; Presence of unidentified species; Lack of standards; and Detection interference.
Cadmium	Mineral supplements such as phosphate, zinc sources; Forage/grains (depending on geographical area); Manure, sewage sludge, or phosphate fertilizer enriched soil or biosolids.	High	AAS; or inductively coupled atomic emission spectroscopy methods. Neutron activation analysis or X-ray fluorescence in living animals.	Susceptibility to contamination and Detection interference
Lead	Contaminated soil, lead paints, water from plumbing systems that contain lead, batteries. Mineral supplements (copper sulphate, zinc sulphate, zinc oxide).	High	Flame atomic absorption spectrometry (AAS), graphite furnace atomic absorption spectrometry (GFAAS), anode stripping voltametry (ASV), inductively coupled plasma-atomic emission spectroscopy (ICP/AES), inductively coupled plasma mass spectrometry, and X-ray fluorescence spectroscopy methods.	Lead contamination during sample collects
Mercury/ methyl mercury	Anthropogenic contamination, fish meal.	High	Cold vapour AAS, atomic fluorescence spectrometry (AFS), electrothermal atomic absorption (ETAAS), neutron activation analysis (NAA), mass spectrometry (MS), and anodic stripping voltammetry (ASV) methods. Capillary gas-liquid chromatography with electron-capture method is used to determine methyl mercury levels in biological samples	Quantification of each species is important; Relative volatility and loss during sample storage, preparation and analysis; oxidizing properties of lab ware can lead to loss of methyl mercury; contamination with mercury; repeated freezing and thawing of wet biological samples can lead to loss of methyl mercury
Other heavy metals				
Copper	Gras and leguminous forages; cereal grains, leguminous oil seed meals; Poultry and swine wastes; Mineral supplements (cupric sulphate, tribasic cupric chloride, copper oxide (primarily cupric oxide), cupric carbonate, and various organic copper sources).	High	Atomic absorption spectrophotometric (AAS) methods (flame or graphite furnace). Inductively coupled plasma-atomic emission spectroscopy (ICP-AES).	Contamination during sample collection

Mineral	Major sources	Animal health concerns	Analytical method	Difficulties with analysis
Iron	Alfalfa; Cereal grains. Leguminous and oil seeds; meat meals; fish meals; Mineral supplements (Ground limestone, oyster shell, and many forms of calcium Phosphate); iron sulphate, iron chloride, iron proteinates, and blood meal.	Medium	Flame AAS, graphite furnace AAS, ICP-AES, ICP-MS, and X-ray fluorescence spectroscopy methods.	Iron contamination during sample collection and processing; Contamination from the atmosphere during analysis if sample is not covered and analysed in a hood.
Zinc	Pasture herbage; cereal grains; leguminous meals; fish meal; whale meal; meat meal; mineral supplements	Medium	Atomic absorption spectrophotometry and inductively coupled plasma emission spectrophotometry	Volatilization loss during ashing at >500°C; contamination of samples
Chromium	Feed grade monocalcium phosphate and defluorinated phosphate Sources	Low	Graphite furnace atomic absorption spectrometry, neutron activation analysis, or mass spectrometry methods are used for low concentrations. Flame atomic absorption spectrometry and inductively coupled plasma-atomic emission spectrometry are used for potentially toxic levels in feedstuff	Background and environmental contamination of biological samples with chromium during collection, storage, and preparation of samples for analysis can present a major source of error. Losses through volatilization during heating or acid digestion of samples.
Molybdenum	Marine origin soil; alkaline soils; pasture; Sodium molybdate	High	Colorimetric; atomic absorption spectroscopy (Graphite furnace AAS); inductively coupled plasma-atomic emission spectrometry methods	Interferences from ferric iron and tungsten' artificially elevated level due to molybdenum carbides accumulation on the wall of graphite furnace
Selenium	Plants from selenium-rich soil	High	Fluorometry; Atomic absorption spectrometry; improved atomic absorption method based on Zeeman effect background; Neutron activation analysis (NAA); HPLC-ICP-MS (inductively coupled plasma mass spectrometry) or HPLC-ESI (electronspray ionization)-MS Methods	Volatility and instability of certain forms of selenium and the non-homogeneity of sample materials; contamination with selenium during sample collection, preparation and storage; explosion hazard; extended digestion time may be required for some samples like urine, some plants and kidney tissue; equipment scarcity for NAA method;

Source: Adapted from NRC [23].

Table 2. Heavy metals, sources and analytical methods.

In view of the potentially harmful effects of toxic levels of elemental minerals in animal feeds, it is essential for stakeholders to have a good grasp of the maximum tolerable levels (MTL) so that it will not be exceeded. Maximum tolerable limits of a mineral is the dietary level that will not impair the performance or health of the animal when fed over a particular period of time [23]. In 2005, National Research Council's Committee on Minerals and Toxic Substances in Diets and Water for Animals recommended MTL of some minerals in feeds of food-producing animals and may serve as a guide depending on the condition of the animals.

4. Methods of analysis in the feed industry

Types of analyses conducted by laboratory are proximate analyses, macro-minerals, micro-minerals at trace level, chromatographic analyses (such as amino acids, fatty acids, etc.) and chromatographic analyses at trace level (contaminants such as aflatoxins, pesticides and pesticide residues, antibiotics, etc.) [24]. Several standard and laboratory methods have been developed over the years for the detection of both nutrients and contaminants in feed ingredients and feedstuffs. Garfield [25] classified the methods into official methods (required by law and used by regulatory and complying organization), reference methods (developed by collaborating organizations for validation purposes), screening or rapid methods (usually for large samples to determine whether further analysis are required with more accurate methods), routine methods (can be official, standard or modified methods used for routine testing), automated methods (may be official or screening methods that adopts automated equipment), and modified methods (usually official or standard methods, which have been modified to make it simple and applicable to wide range of samples).

In the absence of standardized analytical methods, laboratory methods that meet certain criteria, validated and accredited in line with international guidelines and quality assurance protocols, may serve as alternatives. 'Accuracy, applicability (matrix and concentration range), limit of detection, limit of determination, precision, repeatability and reproducibility' are some of the criteria that laboratory methods must meet be serve as an alternative to standard methods [10]. Analytical methods for detection of chemicals, including micro-minerals at trace levels and contaminants in feed ingredients and feedstuff have been highlighted in **Tables 1** and **2**.

4.1. Proximate analyses

Characterization of feeds and feed ingredients for general nutritional parameters are done using proximate analyses. The ability to conduct proximate analyses is the minimum requirement for laboratories [24]. Proximate analyses can be conducted in any basic nutrition laboratory while other analyses can be done in more complex laboratories. Analytical methods for proximate composition and some other feed components are presented in **Table 3**.

4.2. Risk analysis

Demands for higher standards in all aspects of feed production have been on the increase globally. This may be in part due to the increasing awareness of the role of feeds in potential

Parameters	Description
Dry matter	Part of the sample that remains after dying at 103°C
Crude ash	Part of the sample that remains after incineration at 550°C
Ash insoluble in acid (sand)	Ash that remains after boiling in strong acid
Crude protein	Total nitrogen content and to calculate the protein content by multiplying the nitrogen content by an appropriate conversion factor (usually ×6.25). Kjeldahl method (Nitrogen is converted into ammonia which is absorbed in boric acid and titrated against a standard acid); Dumas method (With complete combustion of sample at 950°C in the presence of oxygen, nitrogen is converted to a gaseous state and reduced to N2, followed by measurement in a thermal conductivity cell)
Crude fat	Non-polar extractable fraction of the sample. The extraction can be performed with or without prior acid hydrolysis, both being complementary methods. The laboratory should offer both options
Fibre analysis	Digestion of feed directly in the detergent solution and filtration *using crucibles* (official standard method). Digestion of sample whilst in a *nylon bag* and then washing the bag containing the digested sample to make it detergent free.
Starch	Starch can be measured by the classical Ewers method or with an enzymatic method. The enzymatic method can be used for all sample types and is therefore preferable
Gross energy	Gross energy represents the total energy value of the sample and is measured by bomb calorimeter.
Minerals	Minerals are generally measured by spectrometric methods following incineration and hydrolysis.
Amino acids (excluding tryptophan)	The standard method for the determination of amino acids is based on the hydrolysis of protein to amino acids using a strong acid with or without previous oxidation, followed by chromatographic separation and detection after derivatization
Amino acids (tryptophan)	Determination of tryptophan is based on an alkaline hydrolysis followed by chromatographic Separation
Fatty acids	The standard method for fatty acids is based on isolation and derivatization, followed by gas chromatographic separation
Vitamins	Determination of individual vitamins is based on extraction, followed by clean-up, concentration if needed, and chromatographic measurement.
Reducing sugar	Reducing sugars contain the most important sugars, including glucose, fructose and sucrose. Determination is based on the Luff-Schoorl principle.
Mycotoxins	Mycotoxins are undesirable substances produced by fungi (moulds). These present a potential danger to animal and human health. The maximum levels are nationally and internationally regulated. The different methods are based on extraction, purification, chromatographic separation and detection.
Pesticides	Pesticides are undesirable substances whose maximum levels are defined in national and international regulations. These regulations demand a low detection limit and positive identification of the pesticides, which is achieved by using mass spectrometric detection. The methods are based on extraction, purification, derivatization, chromatographic separation and identification.

Source: de Jonge and Jackson [24].

Table 3. Description of typical tests in feed analyses.

hazards associated with food of animal origin. Accordingly, appropriate codes have been developed by relevant international bodies to assist national authorities to take measures that would mitigate most of these risks, particularly those of public health importance and which may constitute barriers to international trades. Risk analysis is an objective and defensible mechanisms for risks reduction that are associated with health and other factors. For example, Article 2.1 of the Aquatic Animal Health Code, which addresses animal health issues in international trades, provided basic guide and steps for import risk analysis in relation to aquatic animals and aquatic animal products [26]. However, the principles and methods of risk analysis are the same for both aquatic and terrestrial animals and products, including feedstuff. The four components involved with risk analysis are highlighted below:

a. Hazard identification: This is a categorisation step in the risk analysis and the risk assessment should be concluded at this stage in the absence of any identified potential risk.

b. Risk assessment: Involves both qualitative and quantitative methods of risk assessment, each with its relevant outputs. The steps are entry assessment; exposure assessment (both entry and exposure assessment steps involve the assessment of biological, country and commodity factors); consequence assessment (direct and indirect consequences); and risks estimation which integrates results of the entry, exposure and consequence assessments to produce the overall measures of risks associated with the hazard identified at the outset. The risk assessment should be concluded at either entry assessment or exposure assessment step if no substantial risk is demonstrated. The whole risks pathway from identified hazard to unwanted outcome is taken into account by the risk estimation step.

c. Risk management: This involves deciding and implementing protective measures and at the same time minimizing the negative effects on trade. Components of risk management include risk evaluation, option evaluation, implementation, and monitoring and review

d. Risk communication: This requires having a risk communication strategy in place at the outset of each risk analysis.

4.3. Quality assurance and control in feed analysis

Variations in the results of feed analyses obtained from different laboratories have been a major source of concern in the feed industry and among relevant authorities globally [27–30]. Efforts to limit unacceptably high variations in the results of analysed samples in various laboratories, which are sometimes difficult to attribute to genotypic, environmental or inter-laboratory differences, contributed to the development of quality assurance and control for analysis [31]. Use of quality assurance schemes, inter-laboratory evaluation programmes and reference materials were recommended by [32] to reduce errors due to laboratory and methodological differences. Laboratory quality assurance scheme requires the implementation of management quality policy statement, objectives of the scheme, control of samples and records, equipment maintenance, methods evaluation, measurement principles, training, methods selection, intra- and inter-laboratory testing, reference standards, field and lab sampling, statistical considerations, audits, corrective actions, programme revisions and update [7]. These could be grouped properly under the four guiding principles of valid analytical measurement (VAM), which was developed in 1994 in the United Kingdom by the Department of Trade and Industry to contribute to validity of analytical data, namely:

i. Use of properly validated methods of measurement.

ii. Incorporate certified reference materials (CRMs) in quality assurance protocols to ensure traceability measurements.

iii. Independent assessment of laboratory's performance for particular tests through participation in national and international proficiency testing schemes (PTS).

iv. Independent approval of quality assurance arrangements of laboratories by accreditation or licensing to a recognized quality standard.

5. Some aspects of and considerations in feed sampling

The accuracy and reliability of the results of any analysis in the feed industry begins with the quality of sampling. An analysis can be said to be as good as its sampling because several challenges that can affect accuracy and reliability of the results are associated with sampling of the feeds and feed materials [4]. It is, therefore, critical to ensure sampling of feed ingredients and feeds is done in an area and in a way that makes the procedures easy, minimize the risk of contamination and cross contamination, makes proper performance of the laboratory analysis possible, and ensures all safety and health precautions for the sampler and the environment [7].

5.1. Types of samples

Pierce [33] identified various types of samples depending on their purposes and uses as follows: check sample; composite sample; discrete sample; duplicate sample; official sample; purchasing sample; referee sample; reference sample; retained sample; standard sample, and working sample.

5.2. Sampling errors

Sampling errors may be due to the heterogeneity of the inspected characteristics, the random nature of sampling, and the known and acceptable characteristics of the sampling plan [34].

Some of the measures to be taken to minimize sampling errors in the feed industry include

i. Sampling procedures should be based on the objectives, standards, or purposes of the analysis. Simple random sampling, stratified random sampling, and systematic sampling are examples of common sampling schemes used in the feed industry [35].

ii. Use appropriate sampling equipment that will not introduce contamination. For example, do not use lead containing materials to collect samples meant for lead analysis. Examples of sampling equipment include grain probes (slotted grain probes, open-handled grain probes, open-handled spiral probe); pelican grain sampler; tapered bag triers; double tube bag triers; single-tube, open-ended bag triers; bomb or zone sampler [35].

iii. Collect representative samples. If the samples collected are not representative of the whole, the results of the analysis become skewed. To collect a representative sample, the sampling scheme must be followed, adequate quantity of sample must be collected, and sampling equipment and procedure must be appropriate, required inspection of sample, among other things.

iv. Use the right quantity of materials and avoid splashing of samples during collection and analysis. Several errors can be associated with the splitting of samples, if not done carefully.

v. Use standard reference materials.

vi. Repeat analysis.

vii. Validate laboratory methodologies and use standard methods.

viii. Use well trained and knowledgeable personnel.

ix. Observe sampling precautions required for the methods of analysis.

x. Use the appropriate sampling plans.

5.3. Sampling plans selection

Sampling plan is a planned procedure that enables the choice of separate samples from a lot, for the purpose of getting the needed information, such as a decision on compliance status of a lot. It is also a scheme that defines the number of items to collect and the number of non-conforming items required in a sample to evaluate the compliance status of a lot [34]. Thus, without an appropriate sampling plan, it may be practically impossible to accurately decide the compliance status of a particular lot of a product. Codex guideline for sampling [34] recommends seven important considerations in selecting appropriate sampling plans in compliance with relevant standards in the feed industry: (i) existence (or not) of international reference document on sampling of the products under consideration; (ii) nature of control (individual or whole lot), (iii) nature of the characteristic to control (qualitative or quantitative characteristics), (iv) choice of the quality level, limiting quality or acceptance quality level, in line with principles laid down in Codex Manual of procedures and the type of risk, (v) nature of the lot, that is bulk or pre-packed products, size, homogeneity and distribution concerning the characteristics of control, (vi) composition of sample, that is those composed of single or more than one sampling unit, (vii) choice of the type of sampling plan.

5.4. Preparation of samples

Codex code [34] also sets the guidelines for sample preparation. A primary sample is prepared by direct collection of items or incremental samples. During the first stage of the sampling process, primary samples are collected from lots of items or incremental samples for pre-packed or bulk feeds, respectively. In order to facilitate laboratory analysis, sufficient quantity of the primary samples of similar size should be collected. Necessary precautions must be taken to ensure sample integrity and avoid any form of contamination throughout the entire process of sampling and analysis.

Composite sample is prepared, whenever required by the sampling plan, by carefully mixing the primary samples. This involves primary samples collected from a lot of pre-packaged products or incremental samples from a bulk (not-pre-packed) lot. In composite sample preparation, combination of primary samples may lead to loss of information on sample-to-sample variation. The composite sample should, except when too large, constitute the final sample which is sent to the laboratory for analysis (**Table 4**).

Product	Quantity in tons	Number of sub-samples	Minimum quantity of collective sample	Minimum quantity of final sample
Products in receptacles such as bags, drums, big bags, etc.				
Feed materials	Up to 50 tons	2	2 kg	300 g
	Above 50 tons	1 per 25 tons	1 kg per sub-sample	300 g
Compound feeds	All quantities	1	300 g	300 g
Premixes	All quantities	1	100 g	100 g
Feed additives	Up to 1 ton	2	250 g	100 g
	1–50 tons	2	1 kg	100 g
	Above 50 tons	1 per 25 tons	500 g per sub-sample	100 g
Products in storage tanks and silos or shed in the event of an emergency or accident				
Feed materials	Up to 50 tons	2	2 kg	600 g
	50–500 tons	1 per 25 tons	1 kg per 25 tons	600 g
	Part of the batch in excess of 500 tons	1 per 50 tons	1 kg per sub-sample	600 g
Compound feeds, premixes and feed additives	Up to 50 tons	2	2 kg	200 g
	50–500 tons	1 per 25 tons	1 kg per 25 tons	200 g
	Part of the batch in excess of 500 tons	1 per 50 tons	1 kg per sub-sample	200 g
Feed products in bulk per axle or during bagging				
Feed materials	Up to 50 tons	2	2 kg	300 g
Compound feeds	Up to 50 tons	1	300 g	300 g
Premixes	Up to 50 tons	1	100 g	100 g
Feed additives	Up to 50 tons	2	100 g	100 g
Forage products				
Forage products	Up to 50 tons	5 minimum	500 g	250 g
	Above 50 tons	10 minimum	500 g	250 g
Forage products in bulk, transport per axle				
Solid	Up to 50 tons	2 minimum	500 g	500 g
Feed products delivered by vessels or through water ways				
All products	Up to 5000 tons: for each 500 tons	5 minimum	Minimum of 1 kg for each 500 tons	300 g
	5000–10,000 tons for each 1,000 tons	5 minimum	1 kg for each 1,000 tons	300 g
	More than 10,000 tons for each 5000 tons	5 minimum	1 kg for each 5,000 tons	300 g

Source: GMP + International [36].

Table 4. Recommended number of sub-sample for preparing final sample for various category of feed products.

Tests samples are prepared from each composite sample by using appropriate grinding and crushing, sample division and mixing procedures. Some analytes or constituents may be degraded during the process of sample preparation due to a number of factors (**Table 5**).

5.5. Storage of feed samples

There are instances feed samples meant for laboratory analysis requires storage over a specified period of time. The recommended storage duration and conditions for feed samples are presented in **Table 6**.

Origin	Stable parameters	Unstable parameters	Reason(s) for degradation/change
Nutrients	(Crude) protein, fat, ash, fibre	Moisture	Temperature (volatile)
	Starch, sugar, lactose	Ammonia	Temperature (volatile)
	Gas production and enzyme-soluble organic substance production in *in vitro* tests	Organic acids (e.g. lactic acid, acetic acid, butyric acid, fumaric acid, formic acid)	Temperature (volatile)
	Minerals (e.g. Ca, P, Mg, Na, K, Cl)	Unsaturated fatty acids	Air oxidation (can result in production of short-chain fatty acids)
Feed additives	Trace elements (e.g. Cu, Zn, Mn, Fe, Se, Co)	Vitamins (e.g. vitamin A, C, D, E)	Temperature, ultraviolet (UV) light, air oxidation (sensitive)
	Amino acids (e.g. lysine, methionine, tryptophan)	1,2-Propanediol, ethylene glycol	Temperature (volatile)
	Enzymes (e.g. phytases, non-starch polysaccharide enzymes)	Microorganisms like probiotics (e.g. *Saccharomyces cerevisiae, Enterococcus faecium)*	Temperature (freezing), pressure (sensitive to grinding); moisture/dryness (influences growth of microorganisms)
Undesirable substances	Heavy metals (e.g. As, Pb, Cd, Hg)	Mycotoxins (e.g. aflatoxin B_1, deoxynivalenol, fumonisins, ochratoxin A, T-2 toxin, HT-2 toxin, zearalenone, ergot alkaloids)	Mould growth and change of mycotoxins possible at room temperature; UV light (sensitive – aflatoxin B_1)
	Dioxins and polychlorinated biphenyls (PCBs) with similar effects to dioxins	Drugs, antibiotics, pesticides	Temperature (sensitive)
		Hydrocyanic acid	Temperature (volatile)
Banned substances	Proteins of animal origin	Banned drugs, banned antibiotics	Temperature (sensitive)
(Other) Microorganisms		Yeasts, bacteria, moulds	Temperature (sensitive), dryness, influx of oxygen (anaerobiosis)

Source: ISO [37].

Table 5. General classification of stable and unstable parameters in relation to sample preparation.

Product	Storage duration	Storage conditions
Compound feeds (including milk replacer)	3–6 months	Cool, dry and dark
Premixes / processing aids	1 year or longer if there is still product in storage	Cool, dry and dark
feed additives	6 months	Cool, dry and dark
feed materials (dry, artificially dried, naturally dried)	6–12 months depending on the moment of delivery	Sample pot, cool, dry and dark
Fresh feed materials	Max 1 month, storage life often only a few days and will be fed as soon as possible	In air-tight sample bag in freezer
Preserved feed materials (products which are acidified or which have been subjected to natural acidification for the purpose of extending the shelf life of these products)	As long as the product is provided as feed up to a maximum of 2 years.	Preserved product (for example wrapped grass hay bale or green maize silage) is therefore "packaged", that it is available during the storage period for analysis.
Liquid and wet feed materials which are sensitive to decay due to their high moisture content	3 months or as long as it may be assumed that the product will be provided as feed.	In air-tight deepfreeze sample pot
Liquid and wet feed materials which are not sensitive to decay	3 months or as long as it may be assumed that the product will be provided as feed.	Sample pot, cool, dry and dark

Source: GMP + International [36].

Table 6. Storage duration and conditions for feed samples.

6. Conclusion

Food is deemed to be unsafe if it has an adverse effect on human health or it would make the food derived from food-producing animals unsafe for human consumption. Animal feed plays a critical role in the production of safe and nutritious food. There are several considerations that enhance quality and effective decision making in the feed and food production chain(s). Feed sampling and analyses are essential parts of the processes to ensure that feedstuffs and the resultant food animals meet all necessary standards. The reliability and quality of the analysis depends on the accuracy of sampling. Therefore adequate care must be taken to ensure that the analytes are handled in a way that will prevent degradation and errors. Where a feed which has been identified as not satisfying the feed safety requirement is part of a batch, lot or consignment of feed of the same class or description, it shall be presumed that all of the feed in that batch, lot or consignment is so affected, unless following a detailed assessment there is no evidence that the rest of the batch, lot or consignment fails to satisfy the feed safety requirement. This is an important point if you get an adverse sample result when sampling.

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