

MODERNIZATION OF QUALITY CONTROL LABORATORY IN A CATTLE FEED PLANT

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Coverage of presentation

- Introduction
- Commonly analyzed nutrients
- Sample handling
- Conventional and latest test methods
- Advantages and limitations of old and new methods /techniques
- Factors affecting the quality of results
- Safety measures
- Summary

Introduction

- Increased production and growing demands for livestock products offers tremendous growth potential for feed plants to manufacture various types of good quality animal feed.
- Feed quality influences animal production, product quality, safety and environment. In developed countries, it is common practice to undertake most of the analysis in accredited laboratories.



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- However, in India the availability of this kind of service is constrained due to fewer laboratories and lack of infrastructure.
- Manufacturers and buyers do not always have confidence in the data.
- NDDDB has set up a state of the art laboratory to cater to the requirement of cattle feed plants and feed industry.



Commonly analyzed nutrients

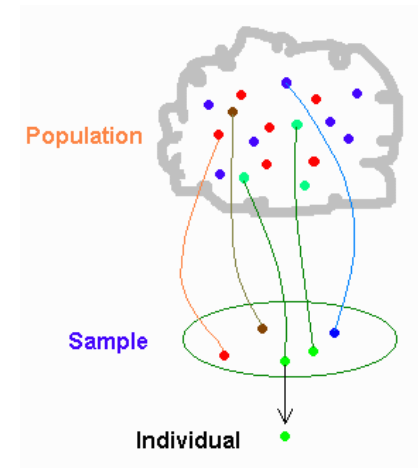
Proximate parameters	Mineral elements	Vitamins	Contaminants/ adulterants
Moisture	Calcium, Phosphorus, Sulphur, Magnesium	Vitamin A	Arsenic
Crude Protein	Copper, Zinc, Cobalt, Manganese, iron, Iodine	Vitamin D3	Lead
Ether extract		Vitamin E	Fluorine
Crude fiber			Urea, Aflatoxins

Equipment used

Hot air oven, Kjeldahl, Soxlet, Fibre Tech	Titration, Atomic absorption spectrometer, UV-Vis Spectrophotometer, ICP-OES	HPLC	ICP MS, Ion analyzer, Spectrophotometer, HPLC
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Sampling

- Sampling is the most crucial aspect.
- It should be done as per laid down procedure.
- Sample should be representative of population.
- Sample taken from different bags should be mixed properly.



Sample Processing



Grinding mill:

- The sample coming to laboratory should be about 0.5 kg.
- The samples must be prepared in such a way that the amount weighed out is homogeneous and representative of the final sample.
- Grinding, mixing and sieving should be carried out as quickly as possible.
- Particle size is important.
- Test sieve should be checked, cleaned and replaced if required.

Sample weighing

Most critical activity after grinding of samples, which may directly affect the accuracy of the results.

- Analytical balance should be accurate to 0.1 mg.
- Analytical balance should be kept on anti vibration table.
- Before weighing ensure your balance is leveled correctly.
- Verification of balance should be done regularly.
- Should be calibrated by accredited lab annually.
- Maintain and record the environmental condition.



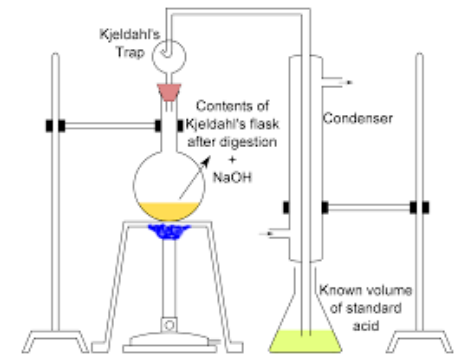
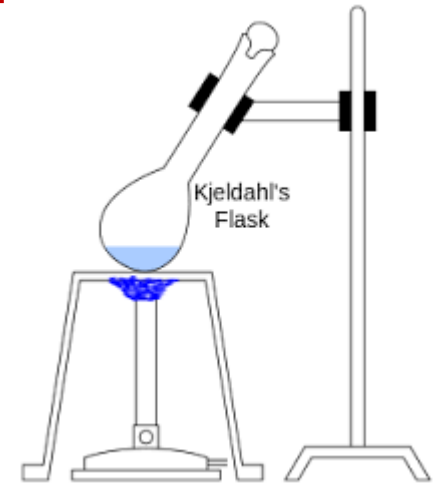
Moisture

- High moisture in raw material and cattle feed may lead to deterioration of nutrients, development of aflatoxins and fire due to fermentation.
- Therefore determination of moisture should be done accurately as results of other parameters are expressed on moisture free basis.
- Temperature accuracy of the oven should be ± 1 °C.
- Verification of oven should be done regularly using calibrated thermocouple.
- Oven should be calibrated by accredited laboratory annually.



Conventional method for protein (Kjeldahl method)

- **Digestion:** Open vessel digestion on heater plate.
- **Distillation:** Ammonium sulphate is converted to ammonia by adding alkali.
- **Titration:** It is done with standard acid manually.
- Generation of hazardous fumes of H_2SO_4 .
- High quantity of chemicals is used.
- Lengthy and time consuming procedure.
- Tricky to fix the assembly.
- Leakage of vapours/steam.
- End point of titration etc are the issues affecting the accuracy and precision of the results.



Soxhlet method for crude fat

- Fat is extracted from the sample using petroleum ether. The solvent is distilled and the residue is dried and weighed.
- Samples are kept for 8-16 hours for extraction.
- Only 4-6 samples can be analyzed at a time.
- Continuous circulation of solvent is critical.
- Lots of solvent goes waste.
- Explosion risk is also there.



Manual Method for crude fibre

- The sample after defatting :
- Sequentially treated with boiling dilute acid and alkali.
- Washing with ethyl alcohol.
- Dry the residue in hot air oven.
- Ash the residue in muffle furnace.
- Crude fibre content is obtained by deducting the weight of ash from the weight of dried residue.
- Very cumbersome procedure.
- Only a few samples can be analysed in a day.
- Chances of manual errors are very high.
- Loss of fibre particle leads to underestimation of crude fibre.



Modern Equipment/techniques

Automatic protein analyzer

- Can digest samples efficiently in an environmental friendly approach.
- Can analyze more than 40 samples/day.
- Less chemicals are required.
- Total analysis time is reduced.
- Meets accuracy and precision criteria set by International methods .



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Automatic crude fat analyzer



- Six to twelve samples can be analyzed within 2-3 hours.
- Recovery of solvent is very high.
- Accuracy and precision of the results is very good.
- Equipment can be left unattended.

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Automatic crude fibre analyzer

- Can analyze several samples in a day.
- Very less time is required in comparison to manual method.
- Meets accuracy and precision criteria set by International methods



Latest equipment for mineral elements analysis

- **ICP-OES:** Multi-element technique can detect over 60 elements simultaneously in the range of ppm to %. (Calcium, Phosphorus, Sulphur, Magnesium, etc.)
- **ICP-MS:** Multi-element technique can detect over 80 elements simultaneously in the range of ppb to ppm.
(Arsenic, cadmium, lead, mercury etc)



Chromatographic techniques for Vitamins and aflatoxin analysis

High Performance Liquid Chromatography (HPLC)

- Used for vitamin A , D3, E analysis in feed and food samples.
- Aflatoxins such as B1 and M1 can be analysed accurately as per international methods.



Near Infrared Spectroscopy (NIRS)

NIR can be used for moisture, fat, protein, starch and crude fibre in animal feed.

Advantages:

- No sample preparation is required except grinding.
- No chemical/glassware is required.
- Several samples for multiple parameters can be analyzed in a day.

Precautions:

- Instrument has to be calibrated using data of samples analyzed by international or reference method. Calibration of equipment must have sufficient number of representative samples covering variation such as:
- Combinations and composition ranges of major and minor sample components;
 - Seasonal, geographic and genetic effects on forages, feed raw material and cereals;
 - Processing techniques and conditions;
 - Storage conditions;
 - Sample and instrument temperature.

Conventional Methods/ techniques

Advantages:

- Initial cost is low.
- Low maintenance of equipment.

Limitations:

- Methods are laborious.
- Analysis is time consuming.
- Low throughput.
- High manpower cost.
- High chemical consumption.
- Safety is an issue.
- Due to manual errors, accuracy and precision is compromised.
- Difficult to meet repeatability criteria as per international methods.

Latest methods/equipments

Advantages:

- High accuracy of results.
- Wide range of analysis can be covered.
- High sample throughput.
- Several types of samples can be analyzed.
- Equipment can be left unattended.
- High safety for people and environment.
- Low manpower cost.
- Automatic data recording which can be shown as evidence.

Personnel

- Knowledge and skills of laboratory technical staff affect the quality of the results produced.
- The laboratory should have a procedure for training and authorization of their personnel for each determination.
- Documents of training should be prepared.
- Competence of analyst should also be checked by giving a blind sample.

Quality control

- **Use of certified reference material (CRM):** These samples are accompanied with certificate of analysis which can be run in each batch/day to check the accuracy of the method.
- **Use of control sample prepared in-house:** Since CRMs are expensive and not easily available, a laboratory can prepare in-house sample which can be analysed along with a batch.
- **Retesting of old sample:** Retesting of old samples can be done to check the method accuracy and competency of the analyst.
- **Spike recovery:** A laboratory can add analyte in a sample and can check how much amount is recovered.

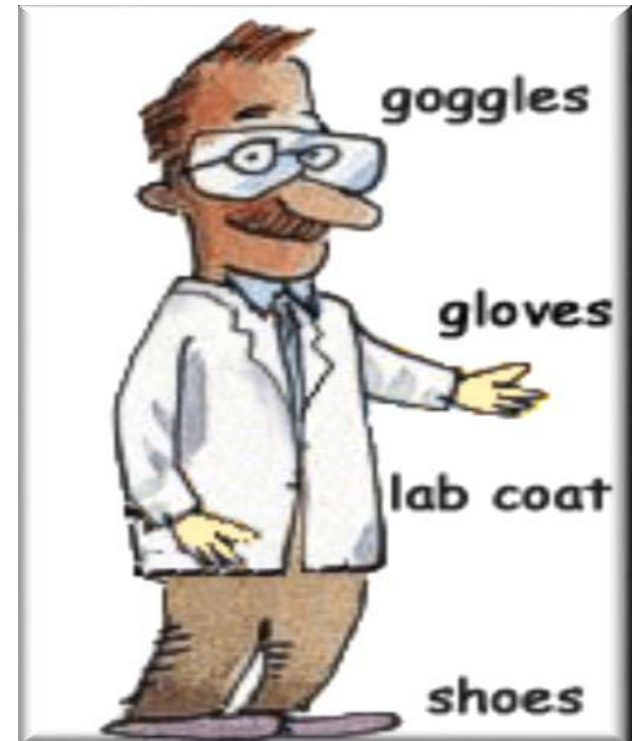
Safety Measures

All analysts must wear personal protective equipment (**PPE**).



Safety on the job is an attitude as much as it is knowledge.

Fume hood, fire extinguishers, safety showers, eye washer, first aid box etc must be available in the laboratory in working condition.



Summary

- With the use of latest equipment a laboratory can analyze adequate number of samples to ensure the accuracy and reliability of results.
- International test method may be adopted to ensure the correctness of results.
- Quality control tools must be used.
- Safety measures should be available.
- Qualified, trained and authorized analyst must perform the analysis.

Thanks

