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Analytical Techniques used by the laboratories of the KZN Department of Agriculture and Rural Development

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The agriculture industry relies upon both qualitative and quantitative chemical analyses of soils, feeds and crops to ensure that the required resources meet certain specifications and to ensure optimum quality and quantity of the final product. The Analytical Services laboratories of the KwaZulu-Natal Department of Agriculture and Rural Development (KZNDARD) employs various analytical techniques, including optical, electrochemical and chromatography. Increasingly, chemical analysis is required to adhere to quality assurance (QA) principles in order to support credibility in the development and application of analytical techniques. KZNDARD Analytical Services laboratories employ recognised analytical techniques to yield accurate and valid results and recommendations to their clients.

Quality assurance

Quality control and quality assurance are incorporated in the analytical scheme to validate results. Quality assurance measures applied in the KZNDARD Analytical Services laboratories include control procedures, monitoring of blank levels of solvents, monitoring equipment and other materials, analysis of procedural blanks, recovery of spiked standards and monitoring of detector response and linearity. During extraction, blanks and duplicates are included in the analysis and re-calibration standards are frequently run to check the integrity of the calibration curve.

Analytical Techniques

Ultraviolet-Visible Spectrophotometer (UV-Vis)

Spectroscopy offers a sophisticated way to manage crop nutrition and assess food and feed quality. For example, in the quantitative analysis of total non-structural carbohydrate (TNC) in plant material: TNC is the main source of energy for plant growth and to a large extent also for animals and humans consuming plant material. TNC content is therefore not only an indicator of the physiological status of the plant, but also plays an important role in animal and human nutrition (Steegmans *et al*., 2004). Carbohydrates are hydrolysed into simple sugars by dilute acids and enzymes and the quantity of simple sugars is then measured colorimetrically using the UV-Vis spectrophotometer (Figure 1) (Silva *et al*., 2018).

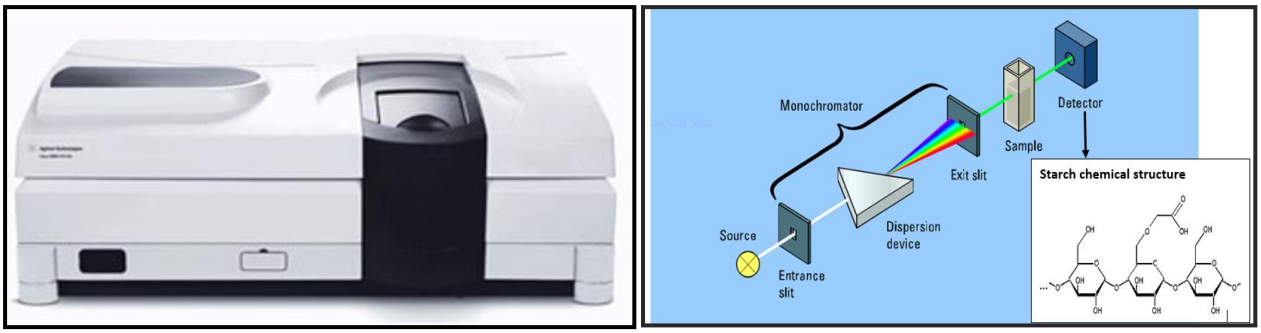

FIGURE 1. Schematic representation of UV-Vis spectrophotometer used by Analytical Services for quantification of carbohydrate concentration in feed and food samples. The light beam (source) with a wavelength varying between 80 and 1100 nm is passed through a sample in a cuvette and is absorbed by the sample at a certain wavelength. The amount of light absorbed by the sample depends on the strength of the constituents being analyse

Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

The Analytical Services laboratory uses the ICP-OES for the determination of plant elements phosphorus, potassium, calcium, magnesium, sodium, copper, zinc, iron, manganese and aluminium (Figure 2). This technique depends upon the spontaneous emission of photons from atoms and ions that have been excited in a high temperature (>5000 °C) plasma of argon gas. Samples are ashed and then extracted or acid digested; analytes of interest are present in a solution form. When the sample is introduced to the plasma energy, the component elements (atoms) get excited. When the excited atoms return to a lower energy level, emission rays (spectrum rays) are released and those that correspond to the photon wavelength are measured. Quantification of mineral concentrations in agricultural soils, growing media, hydroponics water and irrigation water is important as the mineral availability for plant uptake directly affects crop yield. In agricultural crops, mineral concentration quantification is important for evaluating the nutritional quality of food and hence estimating the intake of nutrients by humans and livestock, as well as to manage any potential health risks from toxic elements (Martínez-Ballesta *et al*., 2010).

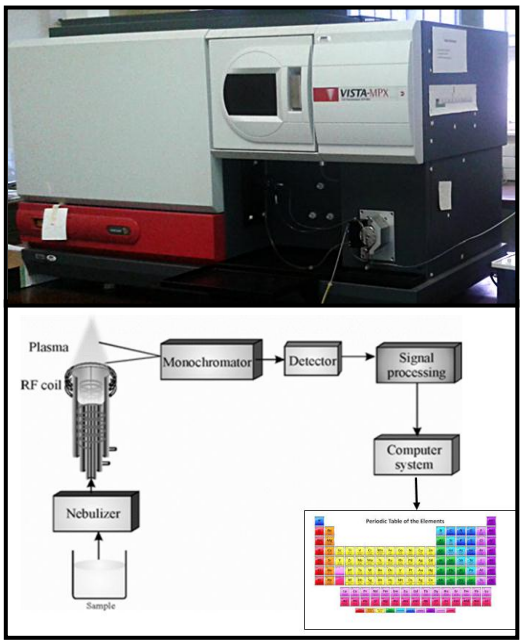

FIGURE 2. Schematic representation of ICP-OES for quantification of macro- and micro-minerals in agricultural samples.

Leco TruMac combustion nitrogen analyzer

Nitrogen is one of the most significant elements for plant growth and quality. The measurement of nitrogen content of crop and livestock feed samples is required to determine protein quantity and hence, nutrition quality (Figure 3). This instrument uses the Duma dry combustion technique: samples are combusted in an induction furnace at 950 to 1050 °C in the presence of oxygen to form water, carbon dioxide (CO_2), sulphur dioxide (SO_2), various nitrogen oxides (NO_x), and nitrogen gas (N_2). CO_2 and SO_2 are removed, and the oxygen is removed from the NO_x. Total N_2 is then measured using a thermal conductivity detector. The nitrogen content of the sample is then multiplied by the protein conversion factor (6.25) to calculate the total crude protein content of the sample.

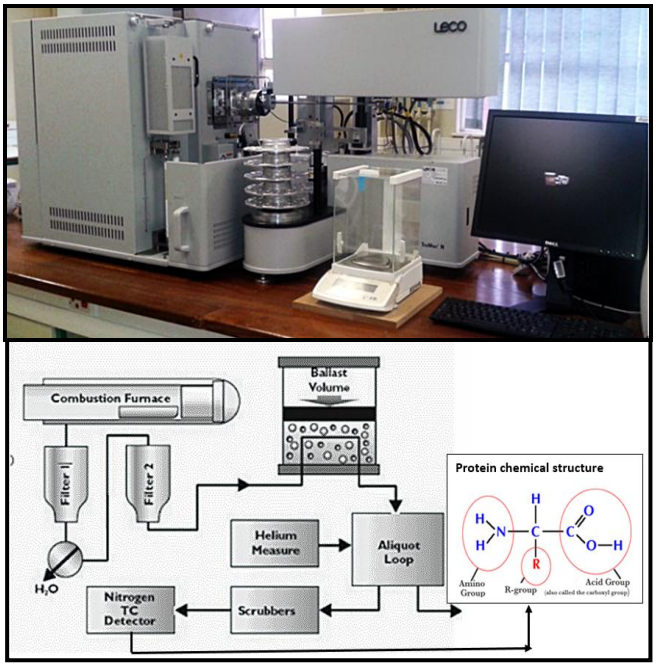

FIGURE 3. Schematic representation of Leco TruMac N combustion nitrogen analyzer for the quantification of crude protein.

Gas chromatography–mass spectrometry (GC-MS)

GC-MS is a separating technique utilized for the identification and characterization of organic compounds used in the medical, cosmetic, detergent, coatings and agrochemical industries (Figure 4). The Analytical Services laboratories use this technique for

qualitative determination of essential oils. A mixture separates into individual substances when heated. The heated gases are carried through a column with an inert gas (such as helium). As the separated substances emerge from the column opening, they flow into the mass spectrometry (MS). Electron ionization (EI), is used to ionize and fragment analyte molecules before detection.

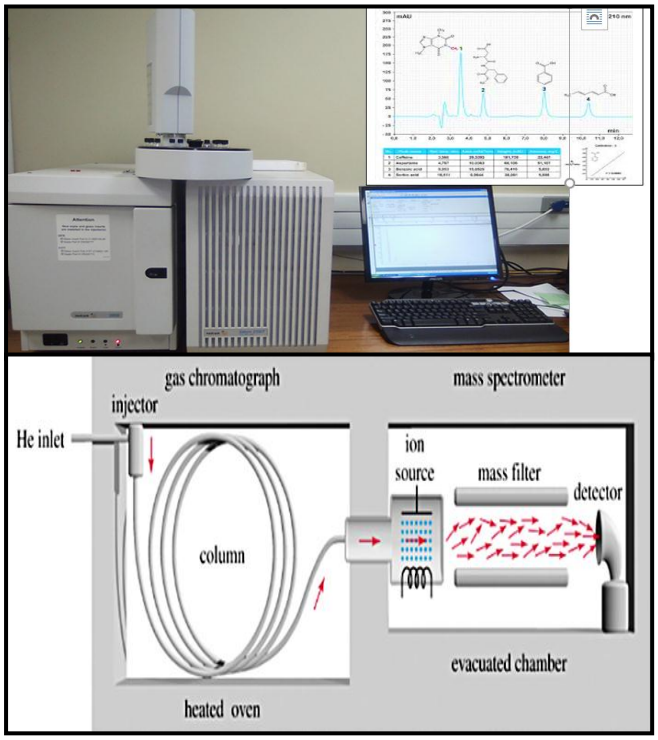

FIGURE 4. Schematic representation of GC-MS for qualitative identification of organic compounds.

Conclusion

The KZNDARD Analytical Services laboratories employ various analytical instruments and their principles to ensure quality results for their clients. The instruments were chosen because of their ability to quickly and accurately determine the concentration of elements and nutritionally-relevant compounds in agricultural samples.

Further reading

Manson, A.D., Bainbridge, S.H. and Thibaud, G.R. 2020. Methods used for analysis of soils and plant material by Analytical Services at Cedara. KZN AgriReport N/A/2020/07.

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