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The Agrifood Laboratory and official control

Generalitat de Catalunya
Departament d'Acció Climàtica, Alimentació i Agenda Rural

Rural Cat

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The technical competence of laboratories, which ensures that laboratory analyses are reliable, is achieved by having a quality system

In recent years we have seen a significant change in the safety and quality regulations in the food chain, which, broadly speaking, entails a comprehensive approach to all parts of the process and a fresh approach to the official checks in food legislation. The official control activities include the analysis of samples of agrifood products to check the legal limits of hazards such as pesticides, heavy metals, contaminants, additives, genetically modified organisms and microorganisms, among other substances, in food, feedstuffs, plant health products, fertilisers, etc.

In the laboratory, this means that analytical technical services must be subject to the appropriate training and act efficiently. The technical competence of laboratories, which ensures that laboratory analyses are reliable, is achieved by having a quality system in place, which must be regularly and systematically evaluated. Accreditation by a competent body is a further step in this direction, as it ensures that the laboratory has a quality system in place compliant with the appropriate ISO standard, and that the analyses are carried out according to characterisation criteria in the stipulated methods (accuracy, precision, repeatability, reproducibility, recovery, uncertainty, etc.).

Another important point is that the facilities and equipment are appropriate, adequate and up to date. Only analytical equipment that is suitable for the analytical purpose concerned and that is regularly and properly checked and calibrated will provide reliable results in a short time which meets the needs of clients. Having modern, cutting-edge technology at our fingertips helps considerably.

The environmental aspects inherent in the laboratory's work must not be overlooked, from both the legal point of view and from the perspective of sustainability and respect for the environment. Certification according to the ISO environmental standard must therefore be a medium-term objective for a laboratory.

Last but not least, I would like to mention the staff who work in the laboratory. Without properly trained, skilled and fully competent people it would be impossible to achieve the objective of ensuring the safety and quality of food and feedstuffs throughout the food chain.

The Agrifood Laboratory not only provides a service for the competent authorities in the food chain, but also aims to be a benchmark laboratory in the official control process. Becoming a leading national laboratory in the field of animal nutrition is a challenge for the future.

I hope that this *Dossier* on the Agrifood Laboratory will raise awareness of its tasks and responsibilities, and its role in official checks. In short, I hope you have a better understanding of this laboratory, which belongs to all of us.

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THE OFFICIAL AGRIFOOD CONTROL SYSTEM IN CATALONIA (I). Food safety

01. Public intervention in food safety

Public policies are the measures that governments implement to respond to the demands and needs of society. These actions must be useful in the sense of having an impact on the correction, mitigation or prevention of identified or potential problems.

In food safety, a high level of compliance with the standards established in the applicable regulations on facilities, equipment, self-checks, personnel training, process quality and hygiene is expected to contribute to minimising the prevalence of hazards throughout the food chain, and to a high level of compliance with food quality standards. These factors must protect the health of the public and ensure the quality of the food available on the market.

Catalan Food Safety Plan 2022-2026. Source: ACSA.

A series of well-defined interventions must be implemented to achieve this chain of results, including risk assessment, food safety regulation, awareness-raising and the promotion of good practices, implementation of monetary systems, official control, information exchanges and the management of food alerts, as well as interactive communication, co-ordination and collaboration by all the parties involved in the food chain.

In Catalonia, food safety policy is undertaken on the basis of the principles of strategic planning. Pursuant to Law 18/2009, on public health, the Government of the Generalitat de Catalunya approves the Catalan Food Safety Plan every five years. This is the indicative instrument and reference framework for all public measures by the Administration of the Generalitat and the local authorities in Catalonia in the area of food safety. This strategic plan establishes an intervention model based on the definition of objectives to be achieved, monitoring indicators and interventions aimed at creating an impact that improves the existing situation until the planned objectives have been reached and maintained. This all involves explicit commitments, common and shared objectives, and co-ordinated and complementary actions involving the ministries of the Generalitat responsible for the environment, agriculture and livestock farming, public health and consumer affairs, and local governments.

Public policy on food safety is based on the methodology of risk analysis, which includes risk assessment, risk management and risk communication. In this context, risk assessment interventions are aimed at providing the best possible scientific knowledge for the appropriate risk management and communication. Risk management interventions must enable risk prevention and rapid responses to imminent risks where necessary. Finally, communication and collaboration interventions are focused on the creation of joint working networks, and an interactive and permanent exchange of information and opinion between all the parties concerned.

Risk management is one of the key components within the risk analysis process, and is characterised by its complexity and the large volume of activities involved. One of the most important of these risk management activities is official control.

02. Official controls

Among all the public interventions in food safety, official controls are particularly notable due to their capacity to have an impact on the issues to be addressed and because of their scope, as they are the primary instrument for public intervention in this field.

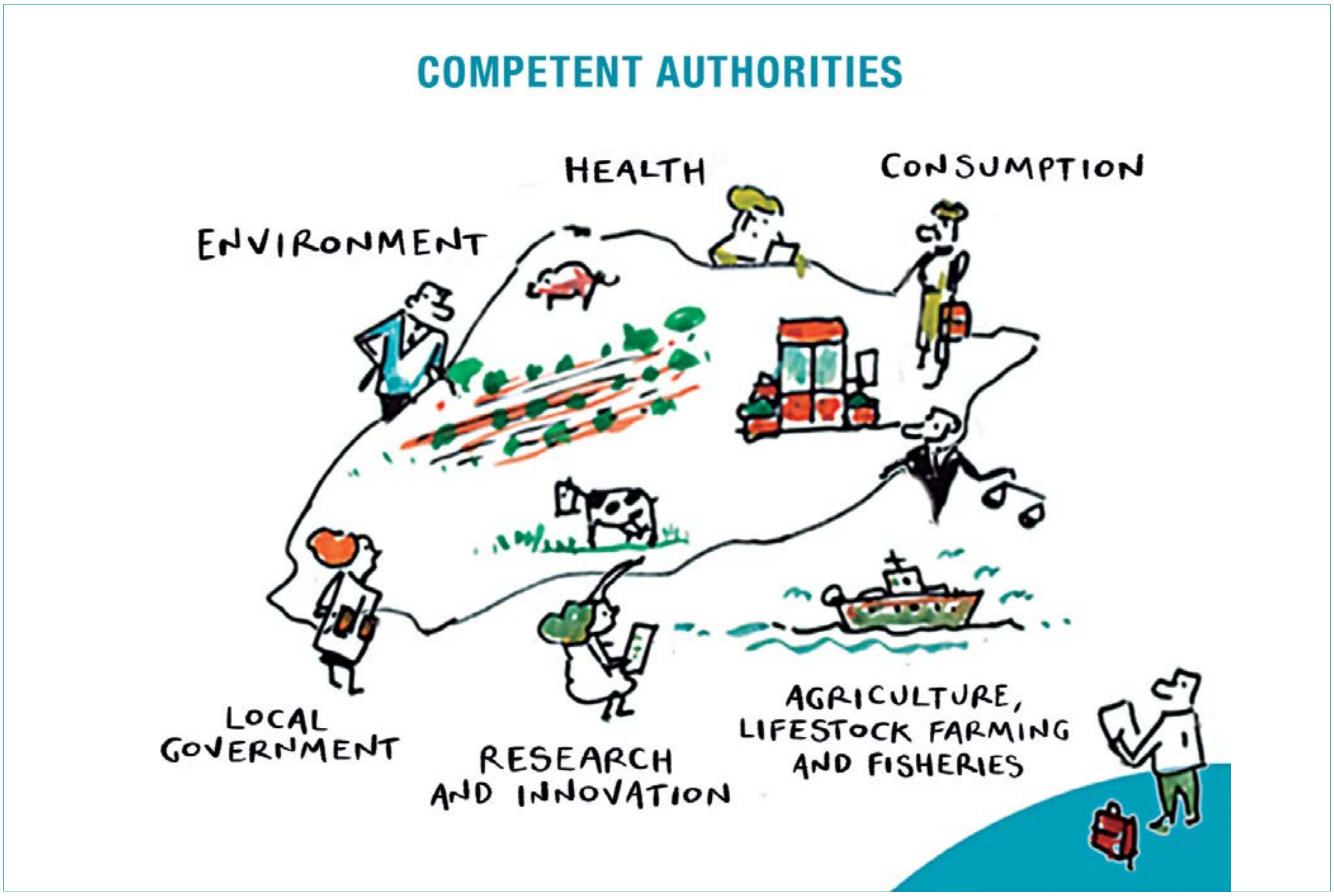
An official control can be defined as any activity carried out or ordered by a competent authority to verify compliance with regulations in the areas of its competence, including the implementation of the measures necessary for correcting of cases of non-compliance found.

In order to analyse what official food safety controls are and what they entail, our benchmark must be Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products.

02.01 Resources required and control techniques

Regulation (EU) 2017/625 states that in order to carry out official controls, competent authorities must be appropriately resourced and equipped, and offer guarantees of impartiality and professionalism, and ensure quality, consistency and effectiveness.

The methods and techniques for official controls include various instruments, including the following: the examination of operators' controls



Official control bodies in Catalonia. Source: ACSA.

and documentation; the inspection of equipment and installations, processes, animals and products; the assessment of the implementation of procedures based on the Hazard Analysis and Critical Control Point (HACCP) principles; interviews with operators and their staff; sampling and analysis, and any other activities required to identify cases of non-compliance.

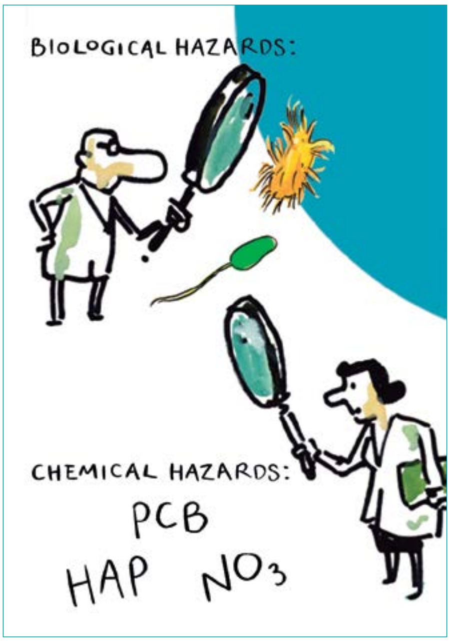
In order to carry out an official control on sales via the Internet or other remote means, the Regulation also states that competent authorities may obtain samples by anonymously placed orders and subject them to compliance checks.

The Regulation states that appropriate information systems must be implemented and that compatibility and interoperability must be ensured in order to contribute to a more efficient management of official controls.

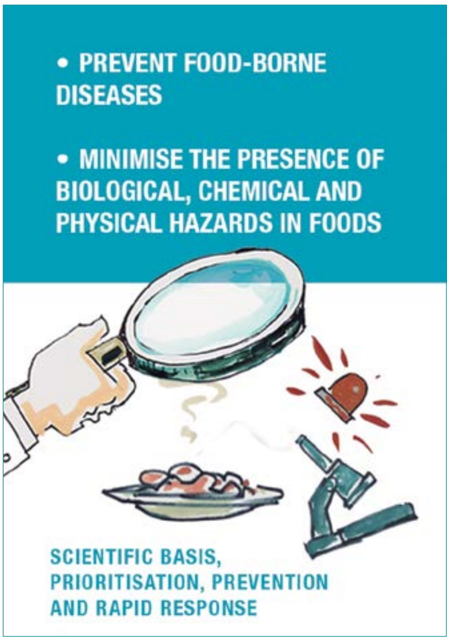
Staff performing official controls must receive regular technical and legal training appropriate to their control responsibilities to create a uniform approach. The availability of adequate financial resources to carry out official controls must also always be ensured. For this reason, the competent authorities must levy fees or duties to cover their costs.

02.02 Sampling and analysis conditions

The methods used for laboratory sampling, analysis, testing and diagnosis must meet scientific standards and provide sound and reliable results in accordance with EN ISO/IEC 17025 on General requirements for the competence of testing and calibration laboratories. Accreditation must be issued by a national accreditation body operating in accordance with EU accreditation regulations. Operators subject to sampling, analysis, testing or diagnosis in the context of official controls shall have the right to a second expert opinion.



Food hazards. Source: ACSA.



Aims of the Catalan Food Safety Plan (I). Source: ACSA.

The Regulation states that appropriate information systems must be implemented and that compatibility and interoperability must be ensured in order to contribute to a more efficient management of official controls.

02.03 Planning, standardisation and auditing of official controls

Official controls must be carried out within a strategic and forward-looking approach. Each Member State must therefore draw up and regularly update a multi-annual national control plan (MANCP) and annual reports on the results, covering all the regulated areas of official control of the chain.

The competent authorities must carry out official controls on a regular basis, on the basis of risk and with appropriate frequency, on all the sectors and in relation to all operators, activities, animals and goods governed by food chain legislation.

Official control must be carried out based on a strategic and forward-looking approach.

Competent authorities must carry out internal audits of their official control systems, which shall be subject to independent review.

Adequate operator records and documented procedures and mechanisms must be in place to ensure effective and consistent action, and to take corrective action when shortcomings are identified. Operators will also be entitled to appeal against the decisions taken by the competent authorities.

Competent authorities must carry out internal audits of their official control systems, which shall be subject to independent review. It also states that competent authorities may delegate some of their tasks to other bodies, while ensuring that the impartiality, quality and consistency of the official controls and of the other official activities are preserved. In particular, the delegated body must be accredited in accordance with the International Organisation for Standardisation (ISO) standard for the conduct of inspections.

The competent authorities must regularly publish information on the official controls and the results obtained.

Audits carried out by experts from the European Commission and third countries focus on the reliability and quality of official controls.

Commission experts must be able to carry out checks, including audits in the Member States to verify the implementation of regulations and the functioning of national control systems and the competent authorities.

02.04 Confidentiality, transparency and public cooperation

The competent authorities shall be accountable to operators and the general public for the efficiency and effectiveness of the official controls they carry out, and must regularly publish information on the official controls and the results obtained.

The Regulation also refers to public cooperation. Adequate arrangements must be in place to enable any person to alert the competent authorities to possible infringements of this Regulation and to protect that person from retaliation.

Aims of the Catalan Food Safety Plan (II). Source: ACSA.

03. Conclusions

Official control therefore occupies a prominent place in the strategy of public intervention in food safety. Its correct application is a cornerstone in guaranteeing food safety and consumer rights and supporting the export profile of Catalan companies operating in the food chain, as it contributes to generating confidence in national and international food markets. The audits carried out regularly by experts from the European Commission and third countries focus on the reliability and quality of official control as essential factors for companies being able to continue their activities.

Further reading

Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products (...): https://eur-lex.europa.eu/legal-content/ES/TXT/?uri=CELEX-%3A02017R0625-20191214

Catalan Food Safety Plan: https://acsa.gencat.cat/ca/agencia/pla-seguretat-alimentaria

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THE OFFICIAL AGRIFOOD CONTROL SYSTEM IN CATALONIA (II).
Food quality and the fight against fraud

01. Official control measures and the fight against fraud in the European Union

EU food legislation made few references to food fraud until 2013, when specific countermeasures were considered as a result of the horse meat crisis. These included:

- Review the legal framework on official controls applicable to the food chain and broaden the approach to include food fraud, leading to Regulation (EU) 2017/625.
- Define "food fraud" at the European level for harmonised control in the Member States. There is now a consensus on defining criteria.
- Reinforce assistance and administrative cooperation of competent national fraud control authorities with the network of contact points (the Food Fraud Network).
- Enable an IT tool for the exchange of information and alerts on food fraud (like the RASFF). The "ACA System" for administrative assistance and cooperation in reporting cross-border fraud and incidents was launched in 2015.
- Engage in a collaborative and multidisciplinary approach to involve law enforcement, judicial authorities and public-private and private institutions in monitoring.
- Improve inspection techniques and base controls on risk analysis.
- Develop and apply technologies and analytical methods to detect fraud.
- Impose deterrent financial penalties that are effective, dissuasive and proportionate to the benefit obtained by the fraudster.
- Encourage private initiatives to establish anti-fraud programmes, such as the obligation to report fraudulent behaviour to the authorities.
- Consider publishing the results of controls, with easily accessible and understandable information for consumers, with the twofold aim of transparency for consumers and discouraging fraudsters.

EU food legislation made few references to fraud until 2013, when the horse meat crisis led to the introduction of specific measures to combat the problem.

- Establish monitoring programmes coordinated by the European Commission involving targeted national monitoring bodies: detection of horse DNA (2013, 2014), adulteration of honey with added sugars (2015-2017), fish species substitution (2015), food e-commerce (2017) and condiments and spices (2019).
- Implement annual OPSON operations coordinated by Europol and Interpol, with the participation of national law enforcement and administrative control authorities, and in particular: controls of canned tuna marketed as fresh (2018) and controls of conventional foodstuffs marketed as organic (2019).
- The Farm to Fork (2020) strategy for a fair, healthy and environmentally friendly food system which among other goals, aims to combat food fraud and considers the implementation of deterrence measures and intensified control to be essential in order to achieve a level playing field between operators.

02 Legal framework for official food quality control and the fight against fraud

Regulation (EU) 2017/625 on official controls and other activities to ensure the application of food and feed law applies has been in force in the EU since December 2019, and extends the scope of control to the entire agrifood chain and including developments in the fight against fraud; it stipulates that Member States must carry out regular, unannounced, risk-based checks; it imposes financial penalties reflecting the financial benefit to the fraudster or a percentage of their turnover, and provides for the establishment of authenticity and integrity centres to support Member States in their activities to prevent and detect fraudulent and deceptive practices (e.g. Knowledge Centre for Food Fraud and Quality) and encourage administrative assistance and cooperation between states in cases of cross-border non-compliance.
At the national level, Law 28/2015 for the defence of food quality is the basic legislation on the control and penalty system in the field of quality. In addition to official control aspects, it also governs operator self-monitoring and monitoring by sectoral associations and mechanisms for cooperation be-

tween administrations, such as the Food Quality Coordination Board, affiliated to the Spanish Ministry of Agriculture, Fisheries and Food and made up of representatives of the Spanish government and the governments of the autonomous communities.

In Catalonia, Law 14/2003 on agrifood quality regulates the main aspects of official control and establishes the conditions for quality assurance, the obligations of agrifood operators, inspection rights and obligations, precautionary measures, the penalty system, etc.

Apart from the general control legislation mentioned above, sector regulations governing specific control aspects must also be taken into account for some products.

03. Framework of competence for the official control of food quality and the fight against fraud in Catalonia

The Generalitat de Catalunya has exclusive competence as regards the quality and traceability of agricultural and livestock products and the fight against fraud in agrifood production and marketing, and these responsibilities have been assumed by the Ministry of Climate Action, Food and Rural Agenda (DACC). The Directorate-General for Agrifood Companies, Quality and Gastronomy is the competent authority for official quality and anti-fraud control.

The control measures are included in the long-term Spanish Plan for the Official Control of the Food Chain (PNCOCA), which is the general Spanish planning instrument, and includes food control measures in other areas (health, consumption, borders, differentiated quality, animal health, etc.). The current PNCOCA covers the period 2021-2025; anti-fraud inspections are included in the Official Food Quality Control Programme of the PNCOCA.

04. Main aspects investigated

The main aim of the inspections is to identify and limit fraud, deception, counterfeiting, adulteration, unauthorised or prohibited practices, and any other infringement of quality regulations in order to protect the economic interests of the actors in the agrifood chain and to ensure fair competition in commercial transactions.

The investigation focuses on agrifood products (their nature, identity, substantial qualities, composition, origin and provenance, quantity, etc.), the identity and activities of operators, and the appropriate use of designations of origin and other official quality marks. Law 14/2003 excludes seeds, medicines, animal health products, plant health products, medicated feedstuffs, infant and dietetic foods, cosmetics, tobacco, live animals and pre-harvest plants from the definition of "agrifood products".

The scope of the investigation excludes aspects related to health, environment, taxation, public subsidies, certification of operators and the DOP, PGI or other classifications products, which fall under the competence of other control units.

Controlled foodstuffs. Photo: Sub-Directorate for Agrifood Inspection and Control.

Controlled foodstuffs. Photo: Sub-Directorate for Agrifood Inspection and Control.

Facilities and practices subject to control. Photo: Sub-Directorate for Agrifood Inspection and Control.

In general, anti-fraud inspections are carried out on the premises of industries where products are processed and/or dispatched before entering the commercial circuit, and exclude direct sales outlets to the end consumer (retail and e-commerce), restaurants and institutions, wholesale markets etc., because they fall under the competence of other bodies.

Official fraud control aims to protect the economic interests of the actors in the agrifood chain and to ensure fair competition.

05. Quality and anti-fraud actions

The following types of inspections and controls take place in the area of quality and fraud prevention:

- Inspections previously scheduled within the General Inspection Plan in companies selected by risk analysis.
- Unplanned, unscheduled inspections arising from unexpected events or suspected infringements: complaints, justified proposals from other control bodies, requests for assistance from other Member States, etc.
- Exploratory controls, which are systematic control measures to categorise food businesses according to risk elements assessed by the inspection staff (activity, self-monitoring, traceability, etc.).

The main steps in anti-fraud inspections are:

- The drafting of an inspection report as an official record of the facts and proof or evidence of non-compliance.
- An investigation and audit methodology based on specific techniques including physical and documentary checks.
- Unannounced inspection visits.
- The system contains penalties; penalties are stipulated if infringements are detected. However a warning may be issued for minor infringements.
- Depending on the seriousness of the infringement, precautionary measures such as the immobilisation of products or labelling, suspension of activities, withdrawal of products from the market, prohibition of advertising, etc. may be taken.

Inspection techniques combine physical checks and documentary checks such as examination of the identity of products (foodstuffs, raw materials, prohibited substances, etc.); examination and assessment of the manufacturing processes; sampling for official analysis; checks on presentation and labelling; flow measurement of products in order to apply the mass balance technique; metrological or quantity control to confirm the real content of packaged foods; verification of government authorisations for the operator's activities; verification of accompanying documents and commercial documentation; examination of records of material accounting (inputs, outputs, production, treatments, etc.) and commercial and financial accounting; verification of the traceability system; evaluation of quality self-monitoring systems; verification of official declarations, etc.

The inspections are unannounced, subject to penalties if infringements are detected and precautionary measures are taken depending on their seriousness.

06. Risk analysis

Risk analysis is applied at three levels in quality control and anti-fraud:

1. Risk analysis of food sectors: this consists of an assessment of all sectors using Delphi and Las Vegas methodology, and the sectors with the highest scores (High Risk) are included in the General Inspection Plan.
2. Company risk analysis: an objective and weighted assessment of risk factors of all companies, on the basis of which the companies to be inspected are selected. More than 70 risk elements are considered, which are grouped into the risk factors listed in table 1.

3. Risk analysis of elements for control on site in companies: during the inspection, it will be necessary to decide which aspects will be checked, depending on the activity, evidence found, indications of fraud, history of infringements, etc.

07 Responsibility

Food operators are responsible for compliance with the legislation in all aspects related to their activity; for example, in the field of quality emphasis must be placed on justifying the veracity of food information in labelling and advertising, having a self-monitoring system to ensure the conformity of products placed on the market, establishing adequate and comprehensible traceability systems, planning for fraud prevention, and extreme caution with regard to raw materials, etc.

When applying penalties and corrective measures, it is necessary to determine the party responsible for infringements, which for packaged products is generally the company that appears on the label, with some exceptions such as falsification, poor preservation of the product or collusion with the manufacturer; and for bulk products, the party responsible is the person who owns or physically possesses the product.

In administrative proceedings, Law 14/2003 and Law 2/2020 (on wine) establish penalties according to the seriousness of the infringements and other criteria such as intentionality, the damage caused, repeated infringements, turnover, etc. Additional penalties such as temporary closure of the company, suspension or cancellation of the right to use a Protected Designation of Origin (PDO), confiscation of goods, etc. are also envisaged. Criminal prosecution is also envisaged for certain infringements, as certain conduct such as offences against public health, falsification of PDO products and fraud involving more than a certain amount, is considered a criminal offence.

Inspection, infringement and penalty history	1 Number of inspections 2 Rate of minor infringements 3 Rate of serious infringements 4 Rate of penalties 5 Number of warnings 6 Number of fines by amount
Marketing activities and formats	7 Administrative authorisations for activities 8 Markets and marketing channels
Company self-monitoring	9 Self-monitoring
Traceability	10 Identification of bulk products 11 Labelling 12 Accompanying documents 13 Product registration and volume
Company size	14 Turnover 15 Technology ratio
External control	16 External control

Table 1. *Risk analysis of companies (risk factors). Source: Sub-Directorate for Agrifood Inspection and Control.*

08 Conclusions

In conclusion, we must reflect on our vulnerability to fraud. Factors such as high profits, low risk of detection and minimal penalties in the context of a globalised market with a long and complex food chain, primarily national controls, economic crisis, geopolitical conflicts and government austerity measures, create the conditions for increasingly sophisticated fraud.

To address this issue, the official control methodology must be adapted to new food production and marketing techniques and be able to use all the possible technologies available (analytical, digital, etc.).

Industries must also adopt preventive measures to exercise extreme caution, especially for products at risk, and implement a control plan to minimise fraud in their safety and quality management systems.

Further reading

http://agricultura.gencat.cat/ca/ambits/alimentacio/lluita-frau-alimentari/

https://ec.europa.eu/food/safety/agri-food-fraud_enhttps://ec.europa.eu/food/safety/agri-food-fraud_en

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THE ROLE OF THE AGRIFOOD LABORATORY in official controls

Agrifood Laboratory facilities. Photo: AFL

01. Introduction

The Agrifood Laboratory (AFL) of the Ministry of Climate Action, Food and Rural Agenda, accredited according to the UNE-EN ISO/IEC 17025 standard, is an official service and the technical cornerstone of the Catalan Government in terms of analytical needs for the control and monitoring of agrifood quality and safety. Based in Cabrils, it is involved in the official control of agrifood products and means of production by analysing samples from inspection procedures, litigation and control campaigns in the field of agrifood intervention and foreign trade.

The Official Tasting Panel of Virgin Olive Oils of Catalonia (PT) is located in Reus. It is functionally affiliated to and completes the responsibilities of the AFL, with the sensory evaluation of olive oil.

The main tasks of the AFL are to:

- Conduct analysis in litigation proceedings and in control campaigns.
- Participate in projects and issue opinions, reports and certificates related to the quality and safety of agrifood products.
- Design, develop and apply analysis methods to control the quality and safety of agrifood products, in accordance with the criteria required by the European Union and international regulations.
- Participate in Spanish and international projects aimed at improving and optimising inspection and control processes for quality and safety in the agrifood sector.
- Establish agreements with companies and associations in the agrifood sector to carry out studies and projects of shared interest.
- Establish collaboration agreements with training centres to host trainees.

> The AFL is an official service and the technical cornerstone of the Catalan Government in terms of its analytical needs for the control and monitoring of agrifood quality and safety.

The AFL provides its services to public administrations, and to individuals and companies only when they are subject to government subpoenas (contradictory analyses, exports, etc.). If the latter require analyses for information purposes, they should contact private laboratories on the Catalan Register of Agrifood Laboratories. The AFL can only offer informative analyses to individuals or companies in cases where private laboratories do not offer an accredited analysis.

02. Legal framework

In the European Union, the current regulatory framework for official controls on the quality and safety of agrifood products is Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products (hereinafter the Official Controls Regulation).

The previous regulation governing official controls in these areas, Regulation EC 882/2004, established the obligation for Member States to have multi-annual national control plans.

The current regulatory framework for official controls on the quality and safety of agrifood products is EU Regulation 2017/625.

Sampling and official analysis methods are governed by regulations. A tiered system for selecting methods is established if the analysis methods are not described in Community legislation. The official control samples may be informative or regulatory.

In response to this requirement, the Spanish Plan for the Official Control of the Food Chain (PNCOCA) was published, which sets out the systems for official controls along the entire food chain, from primary production to the points of sale to the end consumer. This plan is valid for five years and is updated periodically. As we will see in the section on laboratory designation below, the PNCOCA includes analytical laboratories.

03. Official sampling and analysis

Both official sampling and analysis methods are regulated by legislation. Commission Regulation (EC) 152/2009 of 27 January 2009 lays down the sampling and analysis methods for the official control of feed.

As regards sampling, the minimum number of sampling points (incremental samples) varies depending on the total amount of feed (batch size) and whether the distribution of the analyte concerned is uniform or not to ensure representativeness.

The official methods must be described in Community legislation and failing that, a tiered system for selecting the method established, in the following order:

- methods according to internationally recognised standards or protocols, including those accepted by the European Committee for Standardization (ECS), or developed or recommended by the European Union Reference Laboratories (EURL) and validated according to internationally accepted scientific protocols,
- methods complying with national standards,

Samples kept at the Agrifood Laboratory. Photo: AFL.

- methods developed or recommended by National Reference Laboratories (NRLs) or developed or recommended with method validation studies performed by the laboratory or by several laboratories, validated according to internationally accepted scientific protocols in both cases.

However, in cases where analysis is urgently needed and none of the above methods are available, other methods may be used by the NRL or failing that, by any other designated laboratory until an appropriate method is validated according to internationally accepted scientific protocols.

Samples from official controls may be of an informative or regulatory nature.

Informative or prospective samples provide information on the products analysed, and do not lead to penalties in cases where the results are non-compliant.

According to the Official Control Regulation, operators are entitled to a second expert examination and in the event of a dispute between the competent authority and the operator, another analysis by another official laboratory. To that end, the regulatory samples, which are made up of three specimens (initial, counter and final), can lead to the application of the infringements and penalties system established by law. These samples must be properly sealed to ensure they cannot be opened. The counter copy, held by the feed or food businesses, may be used in the event of disagreement with the result of the initial one and where appropriate, the administration may use the final sample.

04. The designation of laboratories

According to the Regulation on official controls, the competent authorities must designate official laboratories to carry out laboratory analyses of samples taken during official controls.

To enforce this requirement, the PNCOCA contains a specific procedure for the designation of laboratories in the agrifood area, whereby the competent authority designates a specific laboratory to carry out the analyses for which it is responsible. It foresees the designation of both public and private laboratories, provided that with some exceptions due to a lack of supply and urgency, they have accreditation for the required tests according to the UNE-EN ISO/IEC 17025 standard (general requirements for the competence of testing and calibration laboratories). The designation is made for specific determinations or in overall terms if they are the same as all the tests listed in the technical annex of the laboratory's accreditation.

Commission Delegated Regulation (EU) 2021/1353 of 17 May 2021 complements the Official Controls Regulation with regard to the cases and conditions under which competent authorities may designate official laboratories which do not fulfil the conditions in relation to all the methods they use, in areas affecting AFLs, such as food additives, flavourings and additives for feed, which may be delegated to laboratories using non-accredited methods, provided that they have a quality assurance scheme in place and make use of methods characterised by the relevant criteria set out in Annex III of the Official Controls Regulation (accuracy, precision, repeatability, reproducibility, detection/quantification limits, retrieval, uncertainty, etc.).

Analytical methods carried out in the agrifood Laboratory. Photo: AFL

The Catalan Register of Agrifood Laboratories provides information on public and private agrifood laboratories that carry out analyses and controls on the quality, conformity and traceability of agrifood products.

The competent authorities have to designate official laboratories to carry out laboratory analyses of the samples taken during official controls.

05. The Catalan Register of Agrifood Laboratories

The Catalan Register of Agrifood Laboratories, which was created and regulated by Decree 123/2009 of 28 July 2009, registers laboratories that carry out agrifood analyses for third parties.

This Register has a catalogue of public and private agrifood laboratories that carry out analyses and controls on the quality, compliance and traceability of agrifood products in Catalonia.

There are two types of registration:

- As a laboratory accredited by an official accreditation body, in accordance with compliance with the ISO UNE-EN 17025 standard.
- As a recognised laboratory, which, despite not being accredited according to the ISO standard mentioned above, is competent to carry out the activities for which it is registered.

This catalogue, which is regularly updated, contains a list of the groups of activities for which each laboratory is registered, stating whether it is recognised or accredited. The same agrifood laboratory may be accredited for some activities and recognised for others.

Exports of agrifood products to third countries are the responsibility of the Spanish government, but the AFL is involved in the certification of analytical aspects.

As a member of the LNR Association for additives for use in animal feed, the AFL participates at an EU level in the evaluation of analysis methods in this field.

06. Official certification

Exports to third countries of products for animal feed, human consumption or phytosanitary products are the responsibility of the Spanish government, which issues the appropriate official certificate, which contains a series of health attestations agreed with the authorities in the destination country. In the case of feed, food and live animals, the requirements for each country and type of product or animal can be consulted using the public search engine on the CEXGAN website of the Spanish Ministry of Agriculture, Food and the Environment.

However, in order for these certificates to be issued, prior certification by the competent authorities of the autonomous communities is required in some cases. The AFL is involved in the official certification or attestation from the analytical point of view.

Samples are analysed at the AFL or the client provides reports from accredited laboratories or those on the Catalan Register of Agrifood Laboratories, according to which compliance with the analytical requirements of the products is verified and the appropriate certificate is issued.

07. The AFL as a national reference laboratory

The AFL is part of a consortium of National Reference Laboratories (NRL) for additives for use in animal nutrition, as referred to in Annex II of Commission Regulation (EC) No 378/2005 of 4 March 2005 on detailed rules for the implementation of Regulation (EC) No 1831/2003 of the European Parliament and of the Council as regards the duties and tasks of the Community Reference Laboratory concerning applications for authorisations of feed additives.

In Spain, the Laboratorio Arbitral Agroalimentario in Madrid is also a member of the association; the EU Reference Laboratory (EURL) is the IRMM - *Joint Research Centre* in Geel (Belgium).

The EURL is assisted by the Association of National Reference Laboratories for some of its tasks assigned listed in Annex II to Regulation (EC) No 1831/2003 of the European Parliament and of the Council of 22 September 2003 on additives for use in animal nutrition. In particular, the AFL supports the EURL in the following areas:

- Evaluating additive analysis methods, based on the data submitted in the applications for authorisation.
- Submitting evaluation reports to the European Food Safety Authority (EFSA).

Laboratory records. Source: AFL.

- Coordinating the validation of additive analysis methods.

The Association's laboratories are therefore responsible for contributing to the initial evaluation report for the authorisation of additives, which is drafted by one of the laboratories, by sending comments within 20 days of receiving the report. The AFL evaluated a total of 43 initial reports in 2020, and 36 in 2021.

08. Conclusions

The AFL is part of the system of official controls in the food chain. In addition to being accredited according to the UNE-EN ISO/IEC 17025 standard, it is subject to compliance with EU, national and autonomous regional legislation. Compliance with all regulatory requirements gives the analytical results obtained the necessary quality and reliability. In addition, with the Register of Agrifood Laboratories, the AFL ensures that there is an adequate range of analyses available, and it supports the commercial activities of economic operators with its certification. As a member of the Association of National Reference Laboratories, it has a number of responsibilities that give it prestige and enhance the quality of its services in the field of animal nutrition.

2021: 131 CELAC	13 companies	11 feedstuffs (100 CELAC)
		1 food (11 CELAC)
		1 phytosanitary treatment (20 CELAC)
	10 countries	Algeria (75 CELAC)
		Sudan (22), Philippines (17), Russia (5), Indonesia (5), USA (2), Singapore (2), Jordan (1), Morocco (1), Saudi Arabia (1)
	91 samples analysed in the AFL (69%)	- Animal components, microbiology and radioactivity (50 CELAC) - Physical chemistry, microbiology and radioactivity (41 CELAC)

Export certificates (CELAC) issued by the Agrifood Laboratory in 2021. Source: AFL

Further reading

Agrifood Laboratory
http://agricultura.gencat.cat/ca/ambits/alimentacio/laboratori-agroalimentari/

Regulation (EU) 2017/625 of the European Parliament and of the Council of 15 March on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products
https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:02017R0625-20191214&qid=1631604667093&from=ES

Commission Delegated Regulation (EU) 2021/1353 of 17 May 2021 supplementing Regulation (EU) 2017/625 of the European Parliament and of the Council with regard to the cases and conditions under which competent authorities may designate official laboratories which do not fulfil the conditions in relation to all the methods they use for official controls or other official activities
https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32021R1353&from=ES

Commission Regulation (EC) No 152/2009 of 27 January 2009 laying down the methods of sampling and analysis for the official control of feed
https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:02009R0152-20170524&from=EN

Spanish Plan for the Official Control of the Food Chain (PNCOCA)
https://www.aesan.gob.es/AECOSAN/web/seguridad_alimentaria/seccion/pncoca.htm

Decree 123/2009 of 28 July 2009, on the Catalan Register of Agrifood Laboratories
https://portaljuridic.gencat.cat/ca/document-del pjur/?documentId=480654&language=ca_ES

Catalan Register of Agrifood Laboratories
http://agricultura.gencat.cat/ca/ambits/alimentacio/laboratori-agroalimentari/registre-laboratoris/

Catalogue of the Catalan Register of Agrifood Laboratories
https://analisi.transparenciacatalunya.cat/en/Ind-stria/Registre-dels-laboratoris-agroalimentaris-de-Catal/4i2z-4vrq/data

Foreign Livestock Trade (CEXGAN) https://servicio.mapama.gob.es/cexgan/publico/publico/Buscador.aspx

Commission Regulation (EC) No 378/2005 of 4 March 2005 on detailed rules for the implementation of Regulation (EC) No 1831/2003 of the European Parliament and of the Council as regards the duties and tasks of the Community Reference Laboratory concerning applications for authorisations of feed additives
https://eur-lex.europa.eu/legal-content/ES/TXT/?uri=CELEX%3A02005R0378-20151022&qid=1635331092471

Regulation (EC) No 1831/2003 of the European Parliament and of the Council of 22 September 2003 on additives for use in animal nutrition
https://eur-lex.europa.eu/legal-content/ES/TXT/?uri=CELEX%3A02003R1831-20210327&qid=1635332233194

National Reference Laboratories (NRL) and European Union Reference Laboratories (EURL)
for the study of food and feedstuffs
https://www.mapa.gob.es/es/alimentacion/temas/laboratorios-agroalimentarios/listaaecosanlaboratoriosnacionalesdereferencia_tcm30-448729.pdf

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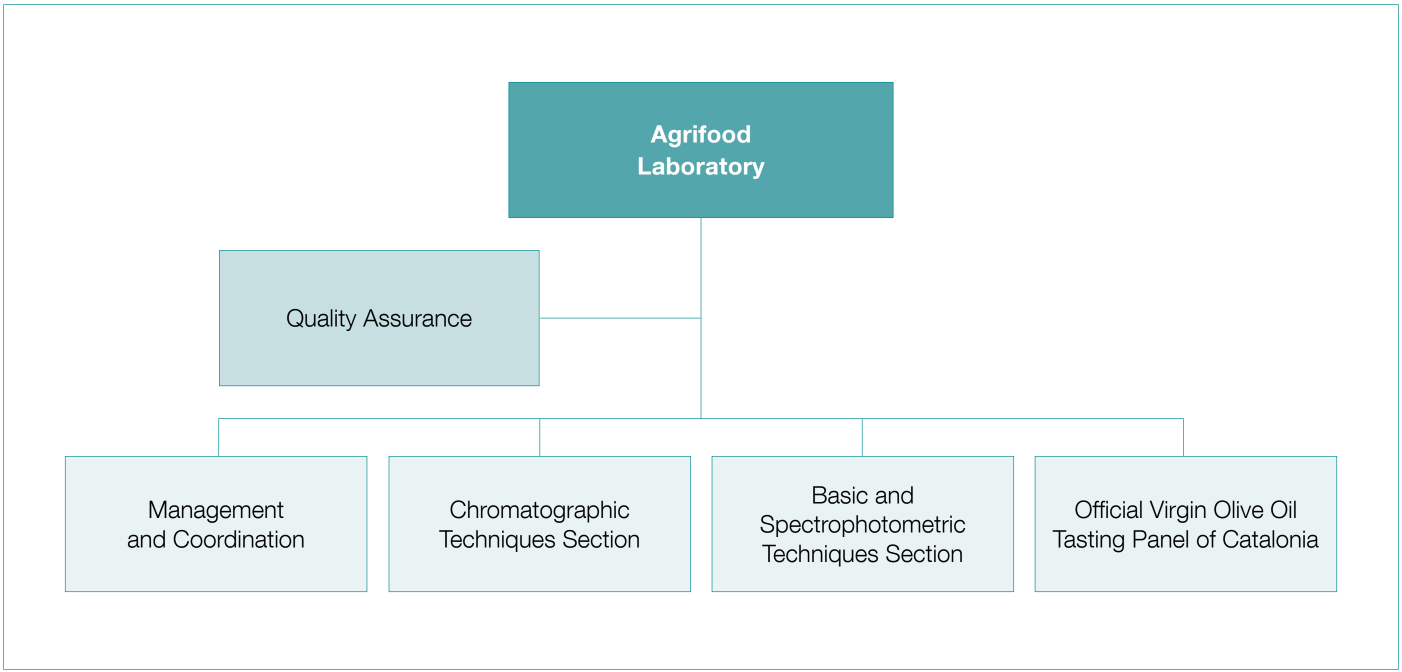
THE LABORATORIES OF THE MINISTRY OF CLIMATE ACTION, FOOD AND RURAL AGENDA.

Infrastructure, technical means and resources



Organisation chart of the AFL. Source: AFL.

01. The Agrifood Laboratory

01.1 Organisation chart

The Agrifood Laboratory (AFL), with a status similar to a service, is a body attached to the Ministry of Climate Action, Food and Rural Agenda. It reports to the Sub-Directorate General for Agrifood Transfer and Innovation.

The AFL, which is located in Cabrils, is accountable to the Official Virgin Olive Oil Tasting Panel of Catalonia, located in Reus.

AFL building. Photo: AFL.

The AFL, which has the status of a service, is a body affiliated to the Ministry of Climate Action, Food and Rural Agenda and reports to the Sub-Directorate General for AgriFood Transfer and Innovation.

01.2 Infrastructure

The AFL is located on the outskirts of Cabrils, in a detached building owned by the Ministry of Climate Action. It occupies a total of 3,271 m^2 on the ground floor and three upper floors. Almost half (1,401 m^2) of this area is given over to laboratories and sample preparation rooms. The rest is allocated to rooms for administrative use, meeting rooms, the library, toilets and changing rooms, an office and canteen, storerooms and installations rooms, hall, corridors, stairways, terraces and courtyards.

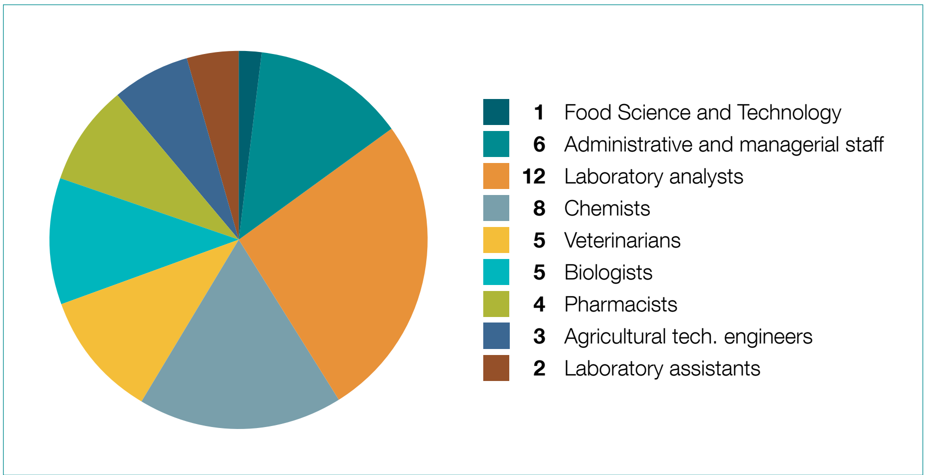
The ground floor is occupied by the Interprofessional Dairy Laboratory of Catalonia (ALLIC). The remaining floors are occupied by the AFL.

01.3 Staff

The AFL is composed of 46 people, including technical, administrative and management staff (Table 1).

Staff	No.	%
Women	35	76
Men	11	24
Total	46	100

Table 1. *AFL staff, by gender. Source: AFL.*



AFL staff, by training. Source: AFL.

AFL staff carrying out an analysis method. Source: AFL

01.4 Equipment

The analytical and instrumental equipment needed to apply the various techniques is extensive and varied. The analytical equipment at the AFL is mainly:

Small instruments
- Optical (e.g. polarimeters, refractometers, microscopes, lamps, magnifying glasses)
- Gravimetric (e.g.: triple beam balances, scales, analytical scales)
- For measuring temperature (e.g. baths, stoves, muffles, autoclaves)
- For preservation/freezing (e.g. refrigerators, freezers, ULT freezers)
- Volumetric (e.g.: burettes, automatic pipettes, dispensers, syringes)
- Evaporation/distillation (e.g: Turbovap, Rotavapor, Gibertini, Kjeltec)
- Homogenising/grinding (e.g: Stomacher, Ultrasound, Ultraturrax, Agytax)
- PH meters
- Microwaves (e.g. ultraclave, ultrasound)
- Fat extractor (e.g.: Soxtec)
- Centrifuges (e.g.: centrifuges, refrigerated ultracentrifuges)
- Anton Paar Digital Density Meter
- Automated culture media preparation and placement units
- Capillary electrophoresis
- Other small instruments

Large instruments
- Gas chromatography:
  - 1 GC-ECD/FID
  - 1 GC-FID/FID
  - 1 GC-MSD
  - 1 GC-FID
  - 2 GC-MS/MS
- Liquid chromatography:
  - 3 HPLC/DAD/F
  - 1 HPLC/DAD/IR
  - 1 LC/DAD/MSD
  - 3 LC-MS/MS
- Spectroscopy
  - 1 ICP-AES
  - 1 ICP-MS
- Spectrophotometer
  - 1 UV/VIS
- Thermocyclers (2 PCR) and real-time thermocyclers (2 qPCR)

- Dumas nitrogen autoanalyser and mercury autoanalyser

Reference equipment
- Filters
- Flowmeters
- Masses
- Temperature probes
- Thermohygrometers

Personal protective equipment
- Glass cases (e.g. gases, acids)
- Cabinets (e.g. laminar flow, gas filtration)

Protection of networked equipment
- Particularly sensitive and costly equipment protected by UPS
- Generator for power outage protection throughout the building
- Fire protection system with control unit

The analytical and instrumental equipment necessary for the application of the various techniques in the AFL is extensive and diverse.

02. The Catalan Animal Health Laboratory and the Catalan Agriculture and Plant Health Laboratory

02.1 Organisation chart

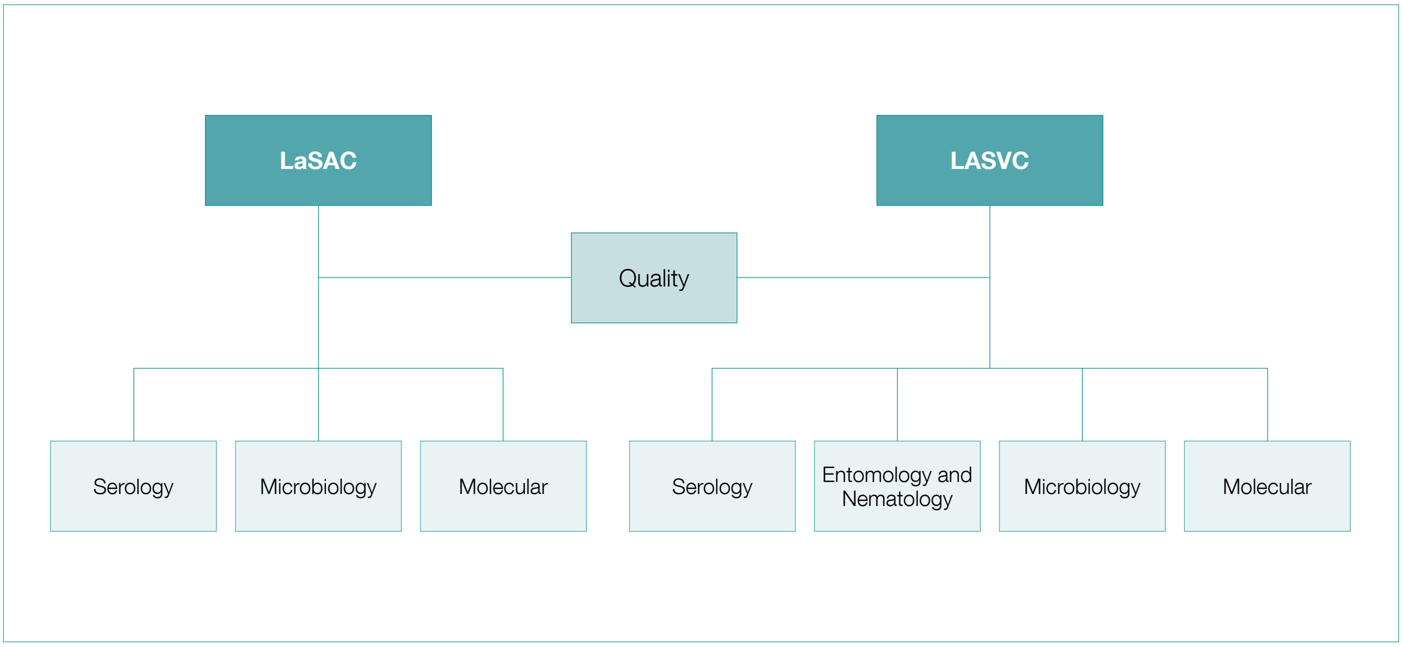
The Catalan Animal Health Laboratory (LaSAC) and the Catalan Agriculture and Plant Health Laboratory (LASVC) are the official technical support laboratories for the diagnosis of diseases included in the official animal and plant health surveillance, monitoring, control and eradication programmes of the Government of Catalonia.

Both laboratories are affiliated to the Directorate-General for Agrifood Companies, Quality and Gastronomy and are located in Lleida on the UdL Campus of the ETSEA (Agronomists).

02.2 Infrastructure

The laboratories of the Directorate-General for Agriculture and Livestock are located in a three-storey building. The two upper floors of 2,000 m^2 are given over to the laboratory facilities, and the ground floor houses the staff entrances, loading and unloading bay and waste management.

FID gas chromatography at the AFL. Photo: AFL.



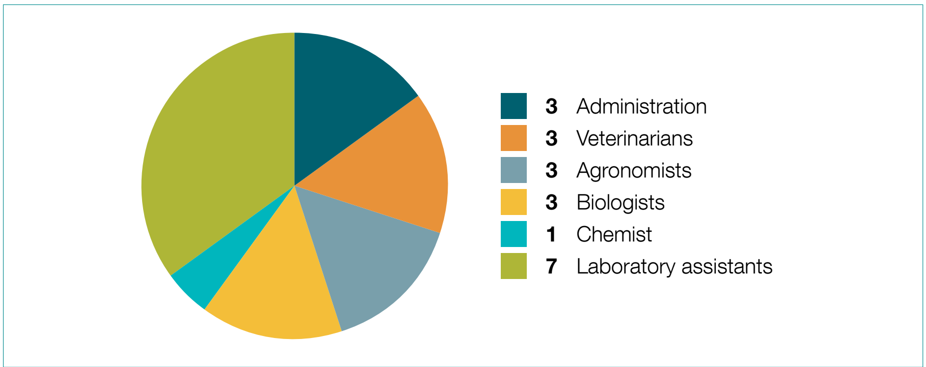
Organisation chart of the LaSAC and LASVC. Source: LaSAC/LASVC.

LaSAC and LASVC building. Photo: LaSAC/LASVC.

The LaSAC and the LASVC are the official technical support laboratories for the diagnosis of the diseases included in the official animal and plant health surveillance, monitoring, control and eradication programmes of the Generalitat de Catalunya.

Staff	No.	%
Women	16	80
Men	4	20
Total	20	100

Table 2. *LaSAC and LASVC staff, by gender. Source: LaSAC/LASVC.*



LaSAC and LASVC staff, by training. Source: LaSAC/LASVC.

The area of the laboratory is divided into a sample reception area, two administration rooms, various rooms for each of the laboratories' departments, a biological containment laboratory, chambers and different rooms used by both laboratories, changing rooms, meeting rooms, storage rooms, etc.

02.3 Laboratory staff

LaSAC and LASVC employ 20 people, including technical, administrative and management staff (Table 2).

02.4 Laboratory equipment

The analytical and instrumental equipment necessary for the application of the different techniques is very varied and extensive. The analytical equipment at LaSAC-LASVC includes:

Small instruments
- Optical (e.g. small microscopes, lamps, magnifying glasses)
- Gravimetric (e.g.: triple beam balances, scales, analytical scales)
- For measuring temperature (e.g. baths, stoves, muffles, autoclaves, incubators)
- For preservation/freezing (e.g. refrigerators, freezers, two ULT freezer units at -80 °C)
- Volumetric (e.g.: burettes, automatic pipettes, dispensers, syringes)
- Homogenising/grinding (e.g. Stomacher, ultrasound, Polytron)
- PH meters
- Microwaves (e.g. ultraclave, ultrasound)
- Fat extractor (e.g.: Soxtec)
- Centrifuges (e.g.: centrifuges, refrigerated ultracentrifuges)
- Automated culture media preparation and placement units
- Capillary electrophoresis
- Thermoblock
- ELISA plate washer
- Millipore Water Purifier
- Electronic and motion sensing Bunsen burners
- Other small instruments

Large instruments
- High-resolution optical stereo and microscopy equipment with image handling and storage systems for the laboratory quality system
- Multichannel pipette calibration equipment
- Gas detector calibration equipment
- Homex sample homogenisers (two)
- Grinding mill-homogenising mill
- Sample processing robots
  - 1 with two 4- and 96-pronged arms
  - 1 with a 4-pronged arm
- Gas chromatograph for MIS bacterial identification
- ELISA photometers (3)
- Nucleic acid extraction equipment
  - 8-column QIAcube (2 units)
  - Magnetic ball biosprint
- DNA and RNA concentrator
- Nanodrop
- End-point thermal cyclers (2 PCR kits) and real-time thermal cyclers (4 qPCR kits)
- Transilluminator
- Centralised temperature control system for refrigerators, cookers, freezers, cold rooms and incubators, by means of external temperature probes

Reference equipment

- Standard masses
- Temperature probe
- Hygrometer
- Photometer verification plate

Personal protective equipment

- Fume cupboards (for acids, bases, toxic and volatile products)
- Biological safety cabinets (I and II), with horizontal and vertical flow
- PCR mini-cabinets

Protection of networked equipment

- Particularly sensitive and expensive equipment, standards and reference materials, protected by UPS
- Generator for power outage protection throughout the building

03. The LIMS in the work of laboratories

The Laboratory Information Management System or LIMS software is a computer application for the efficient management of the entire volume of laboratory data and results.

Both laboratories manage all their samples using this program, from entry into the laboratory until the results report is issued, as well as all related processes.

The LIMS is a software application for efficient management of the entire volume of laboratory data and results.

The LIMS mainly works in the following areas:

- Sample management
- Workload management
- Management of the results and traceability of analyses
- Integration with measuring equipment and instruments

Equipment at the LaSAC and LASVC. Photo: LaSAC/LASVC.

- Validation of results
- Generation of results reports
- Access to the web platform for clients to consult or download results reports
- Exploitation of data and statistics on laboratory activities
- Reagent stock and store management
- Invoicing management
- Equipment management: maintenance, calibrations, breakdowns, etc.
- Quality records management

04. Conclusions

The laboratories of the Ministry of Climate Action, Food and Rural Agenda need a robust infrastructure and equipment to carry out all the official controls, as well as a qualified team of technical and administrative staff.

The demands of official control and the need to comply with all the increasingly stringent regulatory requirements mean that it is necessary to periodically renew equipment and to continuously train staff, especially with regard to new analytical methods and techniques.

Further reading

Agrifood Laboratory: https://agricultura.gencat.cat/en/ambits/alimentacio/laboratori-agroalimentari/index.html

Samples recorded in the laboratory and material for preparation prior to analysis. Photo: LaSAC/LASVC.

Catalan Agriculture and Plant Health Laboratory: https://agricultura.gencat.cat/en/ambits/agricultura/dar_sanitat_vegetal_nou/laboratori-agricultura-sanitat-vegetal-catalunya/index.html

Catalan Animal Health Laboratory: http://agricultura.gencat.cat/ca/ambits/ramaderia/sanitat-animal/dar_actuacions_transversals/dar_laboratoris_sanitat_ramadera/

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THE ANALYSIS PROCESS.
Flowchart. An analysis method, a project

Agrifood Laboratory facilities. Photo: AFL.

01. Introduction

This article explains the analysis process in the Agrifood Laboratory (AFL) of two samples in which pesticide residue and genetically modified organism (GMO) determinations are requested, from their reception to the issuing of the analysis report.

The determinations chosen are part of the official controls in the food chain, and are examples of two different procedures. Pesticide residue testing involves the use of chromatography coupled with mass spectrometry techniques, and GMO testing requires real-time PCR amplification of DNA extracts. Chromatography and molecular biology techniques are essential tools in official agrifood control.

02. Reception and preparation of samples

The different clients, which are mostly the Ministry of Climate Action, Food and Rural Agenda (DACC)'s own inspection services, as well as others from the Spanish Government and Spain's autonomous communities, send the samples to the AFL and they are received by the internal Management and Coordination unit.

After the information on the application form and the sample itself has been reviewed, it is identified with a registration number and the sample information and the determinations to be carried out are entered into the LIMS, the AFL management software.

Chromatography and molecular biology techniques are essential tools in official agrifood control.

The preparation of the sample must ensure its representativeness, and this is governed by applicable legislation.

It includes grinding for solid samples, and filtration for liquid samples, where necessary. The samples are divided into as many sub-samples as types of determinations requested by the customer, in order to distribute them to the technical staff for analysis.

The preparation of the sample must ensure its representativeness, and this is governed by applicable legislation.

03. Determination of pesticide residues in unprocessed plant products

03.1 Regulatory framework

Regulation (EC) 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin is the regulatory framework in this area.

The maximum residue level (MRL) is the highest permitted legal concentration level of a residue of a plant protection product, and is expressed in mg/kg in food and feed. MRLs are listed in the aforementioned Regulation for each plant protection product in each plant product, and are updated regularly as scientific knowledge comes to light. MRLs are available for more than 1,000 phytosanitary treatments in more than 300 products.

In this context, the analysis methods necessary to monitor the presence of pesticide residues in these products must be sufficiently sensitive and specific to be able to identify and quantify a very large number of products at very low concentrations, amounting to parts per billion.

03.2 Preparation of the sample

Regulation (EC) 178/2006 amending Regulation (EC) 396/2005 lists the food and feed products to which maximum levels for pesticide residues apply.

This regulation lists the part of the products to which the maximum limits apply. For example, in citrus fruits, the MRLs apply to the whole product whereas in the case of olives for oil, the MRL applies to the entire product after removal of the stems (if any) and soil (if any).

The first operation to be performed after determining the parts of the sample where the MRLs are to be applied is quartering, which is performed done by dividing each piece into two parts and arranging them randomly on a flat surface. The surface is divided into four parts and two opposite quarters are taken. The quartering operation is repeated until the aliquot for analysis of approximately 250 g is obtained. The sample is then ground.

After the sample has been prepared, the pesticides are extracted.

03.3 Pesticide extraction

The AFL has classified the samples into the following groups according to their characteristics:

- G1: Fruit (vegetables with high water and medium/high sugar content)
- G2: Vegetables (vegetables with high water and low sugar content)
- G3: Cereals and legumes (vegetables with low water and low fat content)
- G4: Citrus fruits (vegetables with high water, medium/high sugar and high acid content)
- G5: Nuts and oilseed crops
- G6: Complex matrices and others
- G7: Olives and oily fruits (vegetables with high fat and high water content)
- G8: Flowers
- G9: Leaves
- G10: Trunks

Depending on the group the sample belongs to, extraction with organic solvent (ethyl acetate) is used in groups G1 and G2 or G4 when the determination is performed by gas chromatography

Samples kept at the Agrifood Laboratory. Photo: AFL.

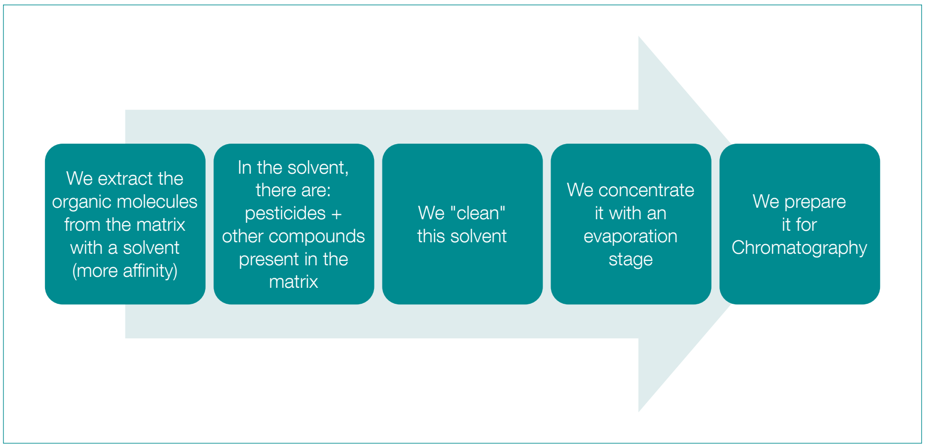


Figure 1. *Pesticide extraction process. Source: AFL.*

Weigh 5 g sample of olives

Prepare the preserved control sample

Add the internal standard to all samples and controls

Add 10 ml of extraction reagent and shake

Centrifuge for 5 minutes at 3,000 rpm

Freeze at -20 °C for at least 12 hours

Take a 6 ml aliquot of the organic phase

Place the aliquot obtained in a clean-up tube (PSA/GCB/Magnesium sulphate)

Shake and centrifuge

Measure 4 ml and evaporate in the TurboVap evaporator at ± 50 °C

Reconstitute with 1 ml ethyl acetate (GC) or 1 ml MeOH/H_2O 80/20 (LC) and homogenise

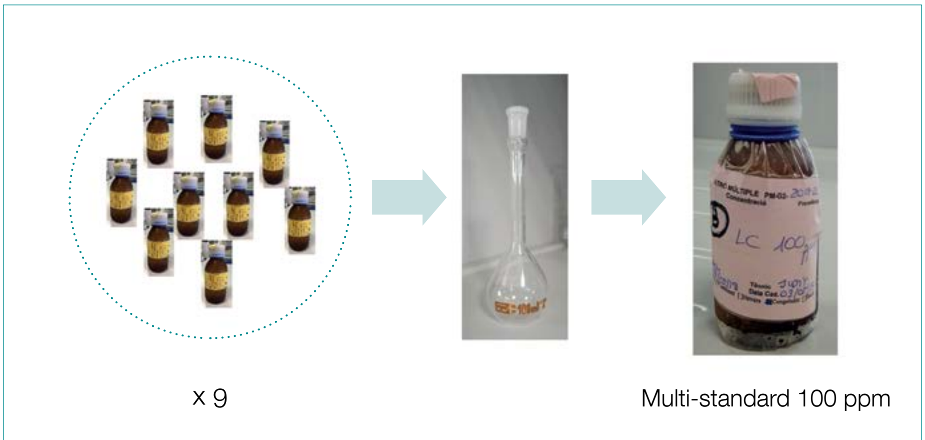
Filter at 0.22 µm if the extracts are analysed by LC or at 0.45 µm if the extracts are analysed by GC

Encapsulate in chromatography vials

Prepare the calibration line with the matrix extract

Analysis procedure for the extraction of pesticides from olives. Source: AFL.

Pesticide analysis. Photo: AFL.



Standard preparation. Source: AFL.

with mass spectrometric detection (GC-MS/QQQ), or with dispersive solid phase extraction (Quechers) in group G4 and in groups G5 to G10 when the determination is performed by liquid chromatography with mass spectrometric detection (LC-MS/QQQ).

The extraction process provides an organic solvent extract for determination by high performance liquid chromatography or high performance gas chromatography with triple quadrupole mass spectrometry detection.

03.4 Detection and quantification of pesticides

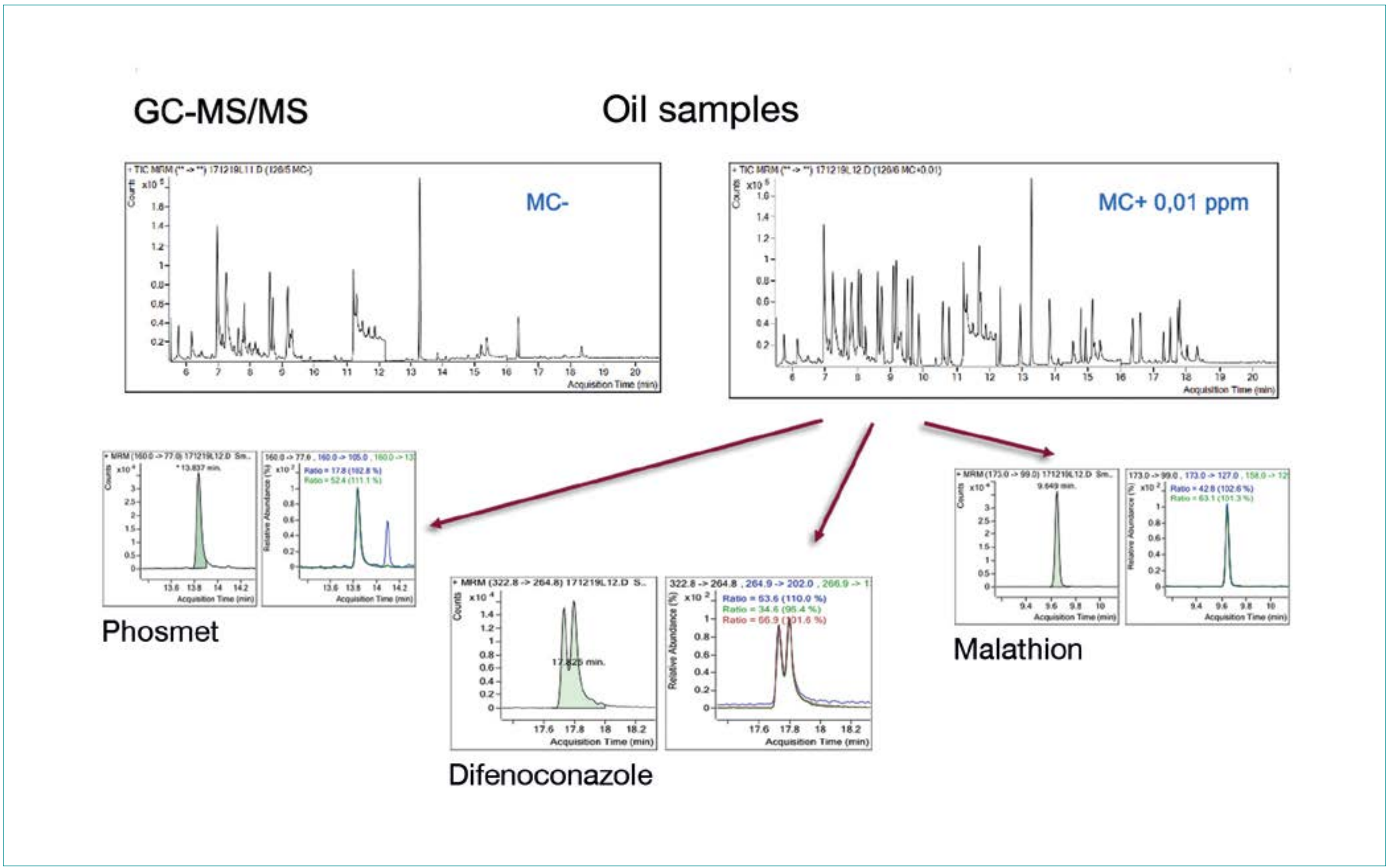
Each pesticide will require a different chromatography system depending on its chemical characteristics. For thermolabile products, the technique of choice is liquid chromatography (LC) and for the others, gas chromatography (GC). Some analytes also respond well to both separation systems. The detection system in both cases is triple quadrupole mass spectrometry (MS/QQQ).

Chromatography techniques coupled with mass spectrometry are very sensitive, and enable pesticides to be identified and quantified.

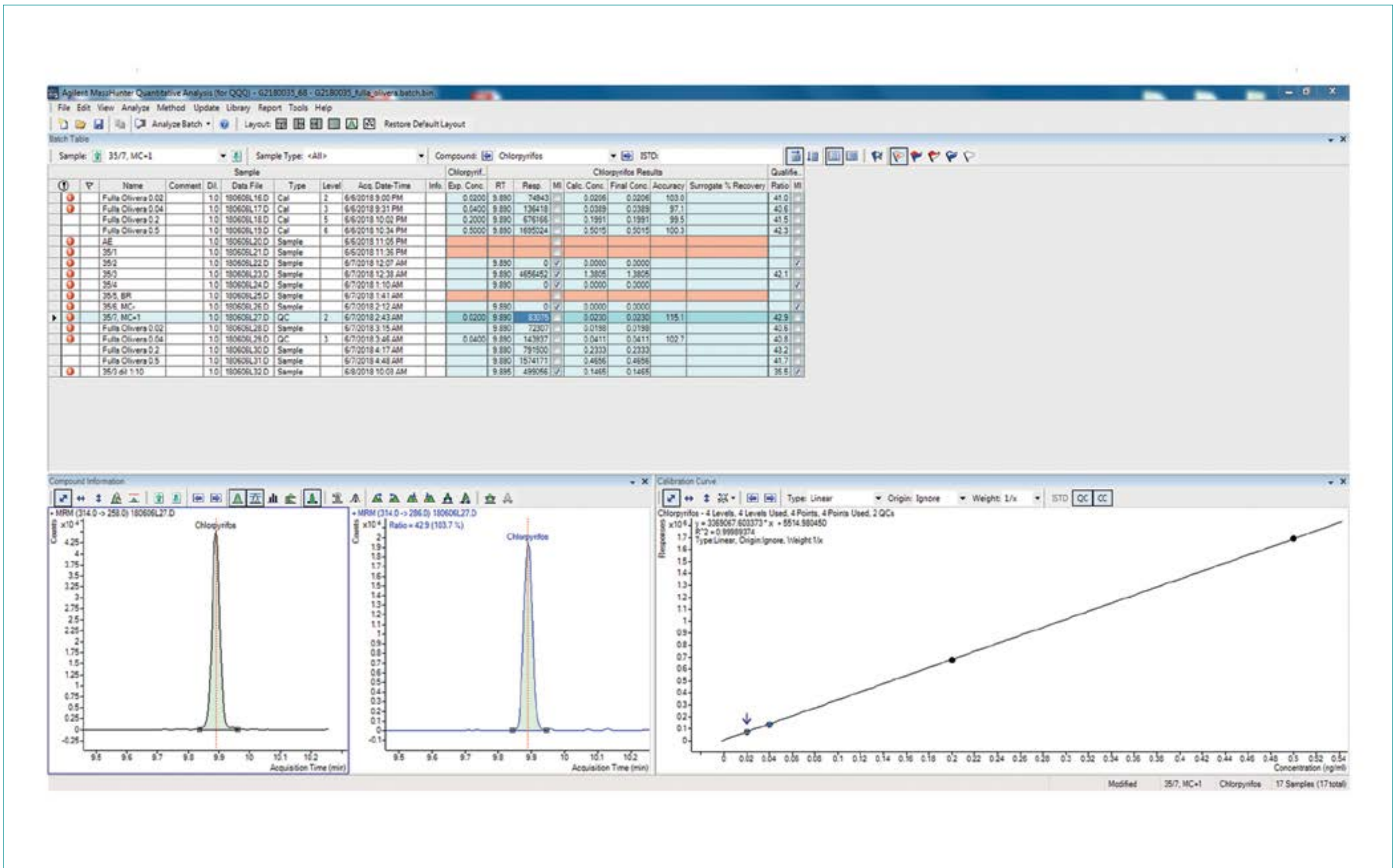
The AFL service catalogue contains a total of 173 pesticides in unprocessed plant products, of which 83 are determined by LC-MS/QQQ and 90 by GC-MS/QQQ.

In the determination and quantification of pesticides, there must be individual standards for each substance to be analysed, with their corresponding certificate of richness. A stock solution of each standard is prepared by weighing and dilution. Various standards are prepared after the 173 individual solutions have been prepared.

Samples without any pesticide content or 'blank samples' are also necessary to act as control samples, in



GC-QQQ chromatographic signal of an oil sample. Source: AFL.



Quantification of chlorpyrifos in olive leaf. Source: AFL.

which pesticides will be determined to conclude that they are not present in the samples, and known amounts of the standards can therefore be added to these blank samples to enable quantification of the test samples and evaluation of the recovery of the extractions.

After the standards, controls and samples have been injected into the appropriate chromatographic systems, chromatographic profiles are obtained to identify and quantify the pesticides.

Chromatography techniques coupled with mass spectrometry are very sensitive, and enable pesticides to be identified and quantified.

The results obtained are listed in the analysis report and interpreted as appropriate by the inspection bodies, taking the applicable regulatory MRLs into account.

04. Detection and quantification of GMOs in animal feed

Regulation (EC) 1829/2003 of the European Parliament and of the Council of 22 September 2003 regulates the limit levels for the labelling and sale of products containing GMOs. Accordingly, in animal feed, feed materials and compound feed containing, consisting of, produced by or produced from GMOs must be labelled as genetically modified products if the content per ingredient is higher than 0.9%. Examples of each type include:

- Foods that are GMOs: genetically modified maize.
- Foods containing GMOs: soy sauce prepared from genetically modified soybeans.
- Food produced from GMOs: genetically modified soybean oil.
- Foods containing ingredients produced from GMOs: a preparation containing soya lecithin from genetically modified soya beans.

It will therefore first be necessary to determine whether or not the feed contains any genetically modified components, and then to quantify the components that have been detected in order to determine whether or not the labelling is correct.

All the analytical methods used are in the database of methods validated by the European Reference Laboratory and are applied in most European laboratories, which means that the results are comparable.

Certified materials mainly from European reference materials are used as reference controls.

04.1 Preparation of the sample

The samples prepared as described in section 02 may be whole grain, broken grain, flour and compound feed, of different grain sizes and types. They should have a fine flour texture to start DNA extraction with the maximum efficiency and homogeneity.

04.2 Separation of workspaces

It is essential that the different work-

Sample of maize in grains and after milling. Source: AFL.

All the analytical methods used are in the database of methods validated by the European Reference Laboratory.

spaces are separated sufficiently to prevent contamination, so there are various separate spaces:

- Sample preparation room
- DNA extract extraction and handling room
- PCR reagent preparation room
- PCR amplification room

The room where the sample is prepared must be well away from the analysis rooms to avoid possible environmental contamination, as the sensitivity of the technique used, PCR, can detect traces and grinding can produce very fine particle dust that could contaminate the environment. For this reason, an environmental monitoring record is included in each milling series to indicate whether or not the environment has been contaminated.

04.3 DNA extraction

A commercial kit (Nucleospin® Food) that has been validated at the AFL is primarily used to perform DNA extraction with the different types of matrices to be analysed (grain, meal, plant material and compound feed). This facilitates unification of the procedure, and allows better control of the reagents.

The extraction consists of cell lysis to release the DNA together with the cell contents, centrifugation to remove solid debris, fixation and precipitation to separate the DNA from the other components of the liquid fraction, fixation to a mini-column with a special silica membrane in which the DNA will be retained, washing to remove soluble substances that may interfere with the analysis, and recovery and solubilisation of the purified DNA. The quantity and quality are subsequently evaluated by means of a microspectrophotometer and the working concentrations are adjusted before analysis.

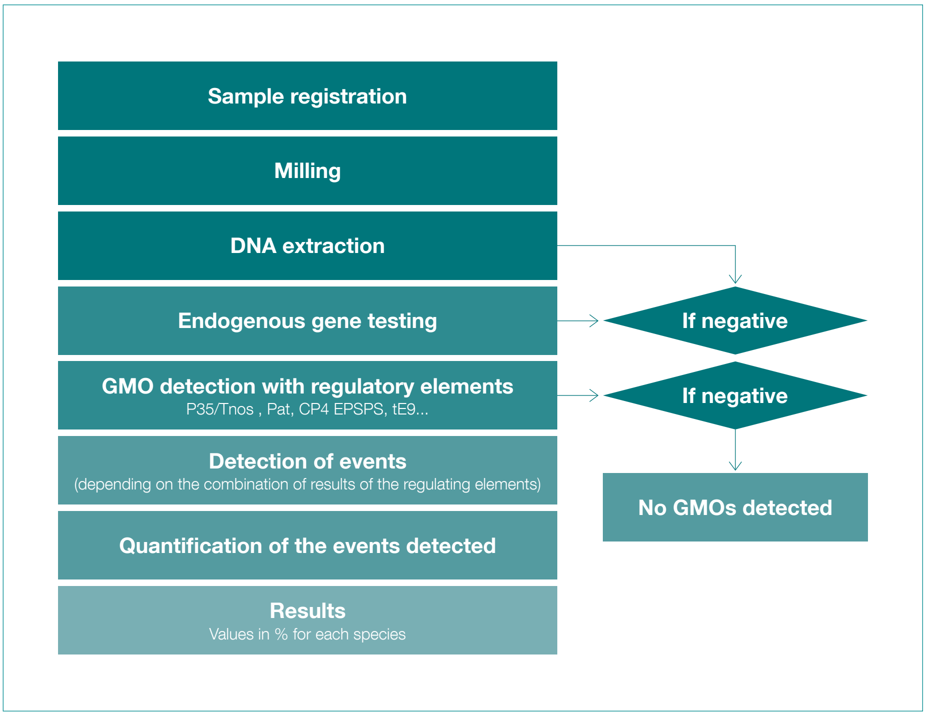
04.4 GMO detection or screening

PCR analysis can now begin with the DNA purified and the concentration adjusted. Both detection and quantification analyses of GMOs are performed by real-time PCR (qPCR) with Taqman probes, which provide high levels of sensitivity and specificity.

First, there is an analysis of regulatory elements (promoters, terminators, building blocks, etc.), which are genes that accompany the structure of the DNA inserted in the modification and are sufficiently widespread so that most and in some cases all genetically modified strains are detected.

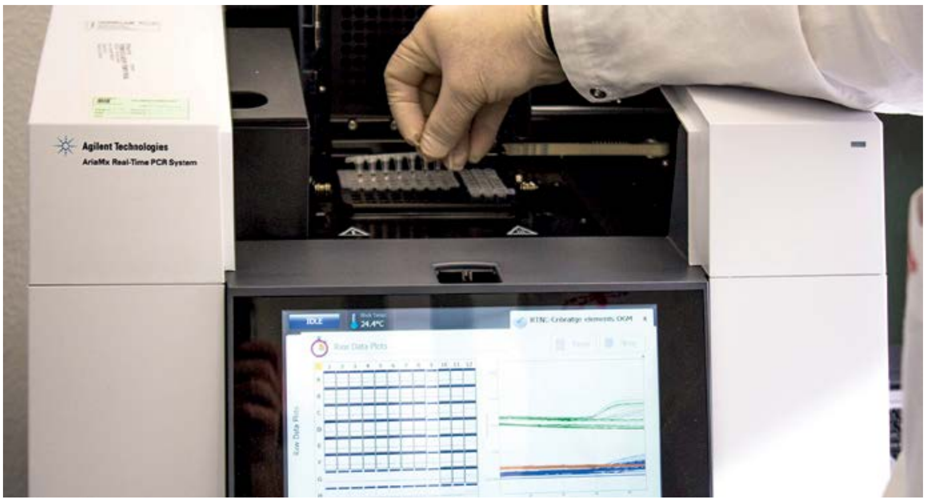
The AFL analyses five regulatory elements (P35s, Tnos, Pat, CP4epsps and tE9), which identify GMOs in materials containing maize, soybean, oilseed rape and/or cotton. Analysis of regulatory elements enables more than 80% of genetically modified varieties to be identified.

Depending on the result of the presence/absence of the elements, the detection of the possible varieties continues according to the screening with the identification of the incorporated events. An event is the series of inserted genes that give the modified variety a specific characteristic. At the AFL, detection is performed using the DNA strand that is called a "specific event", i.e. a DNA sequence that includes part of the plant species' own genome and part of the DNA inserted in the modification, as a target. This ensures detection of the modified gene in a specific plant species.



GMO analytical sequence. Source: AFL.

PCR preparation for GMO analysis. Photo: AFL.



Loading of samples into a real-time thermal cycler. Photo: AFL

Working code	Event	P35s	T-nos	Pat	CP4epsps
1	Bt176 Maize (SYN-EV176-9)	Yes	No	No	No
2	Bt11 Maize (SYN-BT011-1)	Yes	Yes	Yes	No
3	MON810 Maize (MON-00810-6)	Yes	No	No	No
4	GA21 Maize (MON-00021-9)	No	Yes	No	No
5	NK603 Maize (MON-00603-6)	Yes	Yes	No	Yes
6	MON863 Maize (MON-00863-5)	Yes	Yes	No	No
7	TC1507 Maize (DAS-01507-1)	Yes	No	Yes	No
8	MIR604 Maize (SYN-IR604-5)	No	Yes	No	No
9	59122 Maize (DAS-59122-7)	Yes	No	Yes	No
10	MON88017 (MON-88017-3)	Yes	Yes	No	Yes
11	MON89034 (MON-89034-3)	Yes	Yes	No	No
G1	GTS-40-3-2 Soy (MON-04032-6)	Yes	Yes	No	No

Relationship of transgenic events to the presence/absence of regulatory elements. Source: AFL.

** The tE9 terminator is not present in any of the events included in this table.*

After the events present have been detected, the next step is quantification.

04.5 Quantification of GMOs

Quantification makes it possible to establish whether the GMO content is consistent with the information declared on the product label. Since the regulation specifies that the quantification must refer to each ingredient, the quantification is relative, i.e. the values are given as a percentage for each plant species present. Quantification therefore involves an analysis of the inserted gene to be determined, and an analysis of the presence of the taxon (using endogenous genes specific to the species concerned).

The quantification scale is performed with genomic copies, and is calculated using regression lines with serial dilutions based on certified reference material relating the cut-off cycles (Ct or Cq) obtained in qPCR and the concentration in copies. The final calculation is:

$$\% \, OMG = \frac{n^{o} \text{ de còpies del gen modificat (event)}}{N^{o} \text{ de còpies del gen endògen (taxó)}} \times 100$$

Eleven maize and one soybean variety can currently be quantified in the AFL.

The result of the quantitative analysis must report the % of the *modified* event present, its uncertainty and must specify the taxon to which the percentage refers.

05. Conclusions

Pesticide and GMO analyses detect very low amounts of analytes, to confirm that MRLs are not exceeded and to detect possible accidental contamination, respectively, as required by the applicable legislation. These controls contribute to improving the quality and safety of agrifood products.

Further reading

Regulation (EC) No 396/2005 of the European Parliament and of the Council of 23 February 2005 on maximum residue levels of pesticides in or on food and feed of plant and animal origin and amending Council Directive 91/414/EEC

Pesticides Database https://ec.europa.eu/food/plants/pesticides/eu-pesticides-database_en

EUR-Lex. (2022) "Glossary of the synthesis of genetically modified organisms (GMOs)". EU online publications.

Regulation (EC) No 1829/2003 of the European Parliament and of the Council of 22 September 2003 on genetically modified food and feed.

https://gmo-crl.jrc.ec.europa.eu/gmomethods/. Website listing EU-validated and approved methods for the analysis of genetically modified organisms.

https://crm.jrc.ec.europa.eu/. Website of EU-certified reference materials available for the analysis of genetically modified organisms.

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AGRIFOOD LABORATORIES AND ACCREDITATION and official control

01. Introduction

The growing interest of consumers in food safety, together with the increase in both purchasing power and gastronomic culture, has led the market to demand greater and more effective controls from both the competent administrations and the production sectors. Today, accreditation is a necessity for any activity in this sector (both regulatory and voluntary) involving conformity assessment.

The agrifood sector is one of the sectors in which accreditation by the Spanish National Accreditation Body (ENAC) is most consolidated. The existence of accredited bodies (laboratories, certification and inspection bodies) to assess the conformity of products is essential for both manufacturers and the administration: it facilitates access for manufacturers to the global market, as they can demonstrate in a unique, reliable and recognised way to both domestic and international consumers that their products comply with the requirements applicable to them, and for the government it provides assurance of the safety and integrity of product evaluation activities.

02. ENAC and the food sector

ENAC accreditation in the agrifood sector provides a guarantee that certain products and services placed on the market meet specific safety requirements and a series of distinguishing quality characteristics.

The Spanish National Accreditation Body is the institution designated by the Government to operate in Spain as the sole national accreditation body, in application of the Regulation (EC) No 765/2008 of the European Parliament, setting out the requirements for accreditation in Europe.

ENAC's mission is to create confidence in the market and in society by using a system in accordance with international standards to evaluate the technical competence of testing and calibration laboratories, inspection bodies, certification bodies and environmental verifiers operating in all sectors: industry, energy, environment, health, food, research, development and innovation, transport, telecommunications, tourism, services, construction, etc. It thereby contributes to public safety and well-being, the quality of products and services, the protection of the environment and by doing so, to an increase in the competitiveness of Spanish products and services and a reduction in the costs to society arising from those activities.

Analytical balance. Photo: AFL.

Certified weights. Photo: AFL.

The ENAC mark is the means to distinguish whether or not a certificate or a report is accredited. It is the guarantee that the organisation issuing it is technically competent to carry out the work it performs, and that it is competent both in Spain and in the 100 countries where the ENAC mark is recognised and accepted thanks to the recognition agreements that ENAC has signed with the accreditation bodies of those countries.

03. The accredited laboratory - a key player in food safety

The food sector is a strategic sector for the Spanish economy. As a result, accredited testing services are the most useful and powerful tool to meet the business challenges it faces on a continuous basis with the maximum safeguards. Testing is key in various areas, such as food safety and export for the demonstration by manufacturers and other food operators of compliance with the regulatory specifications of the European Union or third countries where the agri-foodstuffs and products are sent.

Accredited testing laboratories are essential for establishing and controlling the safety and quality of foodstuffs.

Accredited testing laboratories are essential for establishing and controlling the safety and quality of foodstuffs, both in the voluntary area of production control and in the regulated area of official control. The work of the more than 375 laboratories accredited by ENAC contributes both to guaranteeing food safety and to verifying characteristics with a view to the possible sales designations of products and their classification into certain qualities, providing the various agents in the sector (the Government, professional associations, regulatory boards, distributors, producers, technology centres, etc.) with technically competent analytical services for a wide variety of products, techniques and parameters:

- Physical-chemical analysis: compositional analysis of specific products such as oils, wine, milk, cereals, meat and meat products, animal feed, physical-chemical analysis of water for human consumption, irrigation water, fertilisers, agricultural soils, analysis of pesticide and veterinary residues, determination of persistent organic compounds (PCBs, PAHs, dioxins), mycotoxins, heavy metals, etc.
- Microbiological analysis of ready-to-eat foods and raw materials, analysis of drinking water; other more specific analyses such as those related to *Salmonella* control at primary production stages (e.g. faeces, environmental samples), analyses for hygiene control of working surfaces.
- Molecular biology analysis: identification of spices, analysis of transgenic products, detection and identification of microorganisms.
- Sensory analyses such as those carried out to detect shortcomings (odour, taste or colour) and to sensorially characterise certain foods, and hedonic sensory analyses carried out to establish consumer preferences.
- Disease control and diagnostic tests carried out by animal health laboratories to ensure animal health and thereby generate confidence in the products obtained. The diagnostic and detection capacity provided by these laboratories is a key factor in dealing with potential crises and health scares such as mad cow disease, brucellosis, salmonellosis, trichinosis, botulism, tuberculosis, anisakis, the influenza A virus, and bluetongue.

Laboratory equipment. Photo: AFL.

04. Europe is committed to food control accreditation

The European Union, the main market for Spanish food operators, has long had a legal framework that establishes a set of clear regulations aimed at preventing, eliminating and reducing the level of risk to human health throughout the food chain, which includes all processes, products and activities related to the production and handling of food and feed, and involves both the competent authorities and private operators (producers, manufacturers, distributors, importers, etc.).

After designing the official food chain control system, the European authorities are committed to accreditation as one of the cornerstones of their food chain strategy. This is evident in the requirement set out in Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004, which made it compulsory for competent authorities to designate only accredited laboratories for official controls, in accordance with ISO/IEC 17025. At present, practically all the official laboratories of the government bodies that carry out official controls in Spain are accredited by ENAC. In addition, numerous accredited private laboratories participate in official controls in application of the European Regulation mentioned above.

The free movement of agrifood products in the European Union requires a high level of protection in areas such as health and food safety, as well as in other areas such as consumer protection against fraud and non-compliance with quality requirements and/or the right to receive truthful and accurate product information.

All this prompted a change in the European legislation and the publication in 2017 of the new Regulation (EC) No 2017/625 on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products.

> The European Commission relies on accreditation as the only valid tool to demonstrate the technical competence of laboratories performing official controls.

The new Regulation introduced important new provisions such as its extension to the whole agrifood chain, more specific rules to tackle fraud including the obligation for Member States to carry out regular, unscheduled and risk-based controls, as well as the application of financial penalties proportional to the operator's economic benefit or a percentage of their turnover, and the creation of EU reference centres to ensure integrity and authenticity throughout the food chain, among other provisions.

It also included some changes to the accreditation requirements for conformity assessment activities related to market surveillance. The European Commission thereby continues to rely on accreditation as the only valid tool for demonstrating technical competence, but has also introduced more stringent requirements for laboratory accreditation.

In order to ensure the reliability and consistency of the controls to be carried out, the laboratories carrying out the analyses required by the established controls previously had to have the appropriate expertise, equipment, infrastructure and staff to carry them out. This requirement is retained in the new Official Control Regulation, which states that those laboratories must be accredited according to EN ISO/IEC 17025, but adds the requirement that all their analytical results must be covered by accreditation to ensure the necessary reliability.

Laboratory equipment. Photo: AFL.

Laboratory records. Photo: AFL.

These increased requirements are reflected in a number of articles aimed at laboratory accreditation. These include Article 37(5), in which the Commission requires the laboratory to accredit every method of analysis necessary to carry out the required controls. This clause improves the clarification of the scope of accreditation compared to the previous Regulation, by referring to the methods of analysis used rather than to the laboratory as an organisation.

05. Avoiding risks in the food chain

When an operator or a company is considering using analytical services to carry out the required controls and wishes to have the maximum safeguards of technical competence, without running any risks, it must select either internal or external accredited laboratories, because only those laboratories have the proven technical competence needed to provide operators with the flexibility and confidence demanded by clients and the international market. Accreditation and the use of accredited evaluators also provide food companies with support against potential liability claims.

Finally, this creates confidence for consumers, who can rely on products and services covered by accredited reports and certificates, as international accreditation agreements ensure that they meet quality and safety standards regardless of the country of origin.

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DATA AND STATISTICS of results

01. What do you need to know about the Agrifood Laboratory?

The Agrifood Laboratory (AFL) works in various sectors of activity. These can be summarised as food quality (food characterisation, composition, origin and authenticity), food safety (microbiology, natural contaminants and undesirable substances, permitted chemicals and their maximum limits and banned substances). It is also involved in the environmental field with analyses for the preparation of fire risk maps. In order to gain a more in-depth understanding of the analytical work done at the AFL, the following sections describe the services it offers, the type of products and samples it analyses, the techniques it applies and the analytical determinations used. It also sets out the control plans in which it participates, and lists the main users of the service.

02. What does it do? Who does it work for? Control plans and sectors of activity

The analytical services of the AFL are determined by the regulatory framework of the European Union, and at Spanish level they are set out in the National Plan for the Official Control of the Food Chain.

Table 2 lists the control plans and programmes, the users, the types of control, the types and number of samples and the analytical determinations performed.

> The analytical services of the AFL are determined by the regulatory framework of the European Union, which are set out in the Spanish Plan for the Official Control of the Food Chain.

Table 1 and Figure 1 present the details and graphic representation of the samples analysed, according to the origin and type of user of the AFL service.

Apart from the regulatory control of agrifood products, the AFL carries out other activities already described in the article entitled "The role of the Agrifood Laboratory in official controls".

03. What accreditation does it have according to UNE-EN ISO/IEC 17025:2017?

The AFL's accreditation according to the UNE-EN ISO/IEC 17025:2017 standard on "General requirements for the competence of testing and calibration laboratories" came into force on 19 February 1999, with number 157/LE309.

Users		No. samples	% samples	No. users	% clients
Public sector	Generalitat de Catalunya	5106	75	23	32
	Other government bodies	927	14	28	39
	Subtotal	6033	89	51	71
Private sector		743	11	21	29
Total		**6776**	**100**	**72**	**100**

Table 1. *Number of samples according to user classification. Figures for 2021. Source: AFL.*

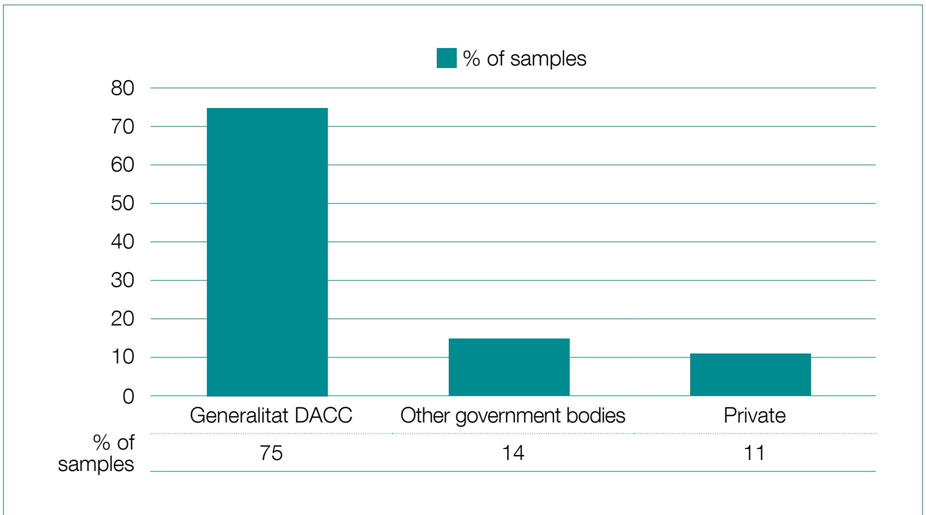

Figure 1. *Percentage of samples analysed according to their origin. Average figure for the last three years. Source: AFL.*

Control plan	User/Client	Type of control	Type of sample	No. samples	Analytical determinations
Plan for inspection and official control of quality and the fight against agrifood fraud	Subdirectorate General for Agrifood Inspection and Control DACC	Nature Identity Composition Species Origin Quality	Food	150-200	Those determining whether the samples comply with the regulatory characteristics of the products
Catalan food residues research plan.	Animal feed and production safety service for livestock production DACC	Illegal use of substances in the rearing of animals for human consumption Correct use of veterinary medicinal products	Feedstuffs Animal urine Animal drinking water Animal hair Fish, crustaceans, molluscs and by-products	2000-2500	Beta-agonists Hormones and lactones Chloramphenicol Corticosteroids Stilbenes Antimicrobials Heavy metals: Hg
Official Animal Feed Control Programme	Animal feed and production safety service for livestock production DACC	Verify compliance with the conditions set out in the applicable regulations	Feedstuffs and their raw materials (including additives and premixes)	750-1100	Components of animal origin: terrestrial animal derivatives, fish derivatives, ruminant DNA Undesirable substances: Heavy metals; Inorganic pollutants and nitrogen compounds; Organochlorate pesticides; Mycotoxins; Inherent plant toxins; Coccidostats Additives: Antioxidants; Vitamins; Trace elements; Urea; Coccidiostats Medicinal substances: Authorised, Not authorised; Microorganisms Antimicrobials Nutritional composition Macrominerals GMOs Pesticides
Official control of hygiene and use of plant protection products	DACC Plant Health Service	Compliance with regulatory requirements for the sustainable use of plant protection products	Fruits, vegetables, cereals, legumes, nuts, flowers, leaves, trunks, etc Culture substrates Pheromone diffusers Irrigation water	400-500	Pesticides; Pheromones
Monitoring plan for the sale of plant protection products	Plant health service DACC	Control of establishments where plant protection products are manufactured, stored or sold	Phytosanitary products Application mixtures for phytosanitary treatments	15-20	Pesticides
Programme for monitoring the quality of the waters and seas in production areas in Catalonia.	IRTA DG Fisheries DACC	Pollution monitoring Compliance with regulatory requirements	Fish, crustaceans, molluscs and by-products	25-50	Heavy metals: As, Cd, Pb, Hg, Ni, Cu, Zn, Cr, Mn
Action plan for the control and awareness of safe consumption of agrifood products	Catalan Consumer Agency	Nature Identity Composition Species Origin Quality	Food	80-120	Those determining whether the samples comply with the regulatory characteristics of the products
Fire Prevention and Firefighting Plan	Forest fire prevention service DACC	Forest fire hazard assessment	Plant material	500-600	Humidity Moisture/Dry Matter Ratio
Farm hygiene plan	Plant Health Service Agricultural Management Service DACC	Verify compliance with the conditions set out in the applicable regulations	Fruit and vegetables Waters	200-240	*Salmonella* *E. coli* *Listeria monocytogenes*
FruitNet Project	IRTA-Fruitcentre Lleida IRTA-Mas Badia Girona DACC	Optimisation of pest and disease control and the use of plant health products in fruit production. Determine the presence period and level of residues of plant health products applied pre-harvest	Fruit (peach, nectarine, pear, apple)	100-150	Phosphonic acid, Acequinocyl, Acetamiprid, AMPA, Bupirimate, Cyfluthrin, Cyprodinil, Deltamethrin, Difenoconazole, Dithianon, EPA, Spirodiclofen, Spirotetramat, Etheophon, Etoxazole, Fenbuconazole, Fludioxonil, Fluopyram, Fluvalinate, Fluxapyroxad, Fosetyl, Fosetyl-Al, Glyphosate, Glufosinate, Glufosinate Ammonium, Isopyrazam, Kresoxim methyl, L-Cyhalothrin, Myclobutanil, MPP, NAG, Penthiopyrad, Pymetrozine, Natural pyrethrins, Pyrimethanil, Spinetoram, Sulfoxaflor, Tebuconazole, Thiacloprid, Pesticide multiresidues
Horta Net Project	IRTA DACC	Develop sustainable production systems related to improved protection against pests, diseases and weeds	Vegetable garden (tomato, lettuce)	8-10	Pesticide multiresidues Spiromesifen Spinosyn A Spinosyn D Spinosad

Control plan	User/Client	Type of control	Type of sample	No. samples	Analytical determinations
Agricultural fertilisation programme	Soil and environmental management service for agricultural production	Improved agricultural fertilisation and organic matter management	Plant material (grasses, fodder, feed materials) Agricultural waters Soils and culture substrates Slurry	140-170	N, P, K Forage quality (DM, Ash, Starch, EE, FB, FAD, FND, LAD, LAD, Digestibility, PB, PD, ENL, UFL, P, Ca, P, Mg) Mycobiology: E. coli, Salmonella, Listeria Mycotoxins: Aflatoxin (B1, B2, G1, G2, Ochratoxin A, Zearalenone)
Fertiliser product control plan	Soil and environmental management service for agricultural production DACC	Control of compliance with current regulations (declared nutritional value, labelling and packaging)	Fertilisers	20-30	Fertiliser composition
Programme to improve the quality of virgin olive oils with Catalan PDOs	"IRTA - Mas Bové DACC"	Technological improvement of olive oil production by mills and controls and the quality of the oils to enhance the profile and presence of Catalan PDO oils in domestic and international markets.	Olive oils	200-250	Quality parameters: Degree of Acidity, Peroxide Value, K232, K270 Moisture and volatiles Insoluble impurities Polyphenols Fatty acid ethyl and methyl esters Sterols waxes Organoleptic evaluation
Quality control of virgin olive oil	Companies, Cooperatives, PDOs	Organoleptic analysis: descriptive certification	Olive oils	1400-1600	Organoleptic classification analysis (oil category and optional labelling terminology) Descriptive report: Positive attributes and shortcomings
National programme. Plan for monitoring and control of commercial cultivation of GMOs. GMO seed monitoring and control plan	DACC Agricultural Management Service	Verify compliance with the conditions set out in the applicable regulations	"Corn and Soya Leaves Maize and Soya Seeds Rapeseed and Cotton"	35-55	GMOs
Public health epidemiological studies	IRTA-CRESA	Salmonella serotyping to obtain genetic data of DNA profiles of salmonella strains and for production of on-farm autovaccines	*Salmonella* strains Seagull cloacal swabs Seagull cloacal swab strain Skua cloacal swabs Wild boar faeces Lizard faeces	20-80	Serotyping of *Salmonella spp*
Control plan for granting subsidies for flax fibre and hemp	Integrated control service and payments Agricultural Management Service DACC	Verify compliance with the conditions set out in the applicable regulations	Hemp	10-15	Δ-9-tetrahydrocannabinol (THC)
Import control plan. Iberian Peninsula Border Inspection Points	Spanish Ministry of Agriculture, Fisheries and Food	Verify compliance with the conditions set out in the applicable regulations	Feedstuffs and their raw materials	700-800	Pesticides, Mycotoxins, Heavy metals (As, Cd, Pb, Hg), Zn-oxide, Fluoride, Nitrites, Microbiology (*Salmonella*, Enterobacteriaceae)
Spanish Plan for the Official Control of the Food Chain. AFL Designation	Government of the Balearic Islands Government of Andalusia Government of Galicia Government of Aragon Government of Cantabria Government of La Rioja Government of Navarre Government of Castile and Leon	Verify compliance with the conditions established in the applicable regulations Illegal use of substances in the rearing of animals for human consumption Correct use of veterinary medicines	Feedstuffs and their raw materials Fruit and vegetables	900-1100	Pesticide residues Heavy metals (As, Cd, Pb, Hg) Zn oxide Nitrites Fluorine Additives Ethoxyquin Mycotoxins Microbiology Ruminant DNA Meals derived from terrestrial animal Fishmeal Authorised and non-authorised medicinal substances Antimicrobials (sulphonamides, tetracyclines) Beta-agonists GMOs
Quality control of packaged fruit juices sent free of charge to food banks.	Lleida Industry, Trade and Regulation of Agrifood Markets Section	Verify compliance with the conditions set out in the applicable regulations	Fruit juices (nectarine, peach, orange, clementine)	10-20	Pesticide residues Microbiology (*E. coli*, *Salmonella*, total mesophilic aerobic count, anaerobic count, fungal and yeast count) Heavy metals (As, Pb) Acidity Degrees Brix
Export licences to third countries	Companies	Import requirements of third country consignee	Feed additives and feed materials	100-150	Microbiology Ruminant DNA GMOs

Table 2. *Control plans and programmes in which the AFL participates. Source: AFL.*

The scope, set out in the applicable technical annex which is available for consultation on the website of both the National Accreditation Body (ENAC) and the Ministry, includes:

- Physical-chemical testing of olive oils and olive pomace oils for quality and purity characteristics and organoleptic evaluation of virgin olive oils
- Microbiology
  - Microbiological analysis using methods based on culture medium isolation techniques
  - Serotyping of strains
- Physical analysis
  - Canned fruit and vegetables
  - White rice
  - Phytosanitary products
- Microscopy
  - Analysis using methods based on optical techniques
- PCR techniques
  - Analysis of GMO using PCR-based methods
  - DNA detection
- Physical-chemical analysis using methods based on the following techniques:
  - gravimetric and volumetric
  - optical
  - molecular spectroscopy
  - atomic spectrometry
  - liquid chromatography
  - gas chromatography
- Descriptive sensory analysis

The technical annex to the scope of accreditation specifies the test product or material, the test identification (technique and working range) and the standard or procedure.

The AFL also has a flexible scope of accreditation according to ENAC Technical Note 19 on the accreditation of pesticide residue analysis in agrifood products (open to matrices and closed to analytes); and a flexible scope according to ENAC Technical Note 18 on accreditation by test categories for the determination of pesticides in phytosanitary treatments. The list of tests covered by the accreditation (LPE and LEBA, respectively) is available in both cases.

The closed and open or flexible accreditation scope formats differ in that in the former, the ENAC has to audit and evaluate the testing procedures before the AFL can issue accredited results.

In the open or flexible format, the AFL manages its own scope of accreditation. Technical competence in the development and validation of new test methods enables the AFL to issue accredited results before being audited/assessed.

More than 84% of the analytical results issued by the AFL are covered by the accreditation of the UNE-EN ISO/IEC 17025:2017 standard.

Among other accreditations and/or recognitions, the International Olive Council (IOC) recognised the AFL for the period from 1 December 2021 to 30 November 2022 for both physical-chemical and sensory type B tests.

This recognition obtained by the AFL and the Official Virgin Olive Oil Tasting Panel of Catalonia means that they can be called upon by the IOC to participate in the event of litigation or disputes in international transactions in which the IOC arbitration procedure is used.

The AFL has also implemented an environmental management system based on the UNE-EN ISO 14001 standard, which aims to contribute to sustainability, and it has adopted the following environmental commitments:

- Compliance with all environmental regulations
- Protection of the environment and prevention of pollution
- Efficient use of natural resources (consumption of energy, water and other natural resources)
- Improving the management of the waste produced
- Compliance with the UNE-EN ISO 14001 international standard

04. What types of products does it analyse?

The products analysed by the AFL can be classified into four broad groups:

- Food
- Means of production
- Feedstuffs
- Other animal products

Food		
Water and ice	Condiments and spices	Vegetables and greens
Stimulant foods	Ice cream	Milk and dairy products
Alcoholic beverages	Animal and vegetable preserves	Edible fats
Non-alcoholic beverages	Natural sweeteners and by-products	Eggs and egg by-products
Meat and meat by-products	Flour and flour by-products	Tubers and tuber by-products
Cereals and by-products	Fruits and by-products	Legumes
Means of production	**Feedstuffs**	**Animal products**
Agricultural waters	Raw materials	Urine
Water for animal consumption	Additives	Hair
Forage	Premixes	Animal tissue
Fertilisers	Compound feed	Viscera
Phytosanitary products		Other
Plant material		
Culture substrates		

Table 3. *Products analysed in the AFL. Source: AFL.*

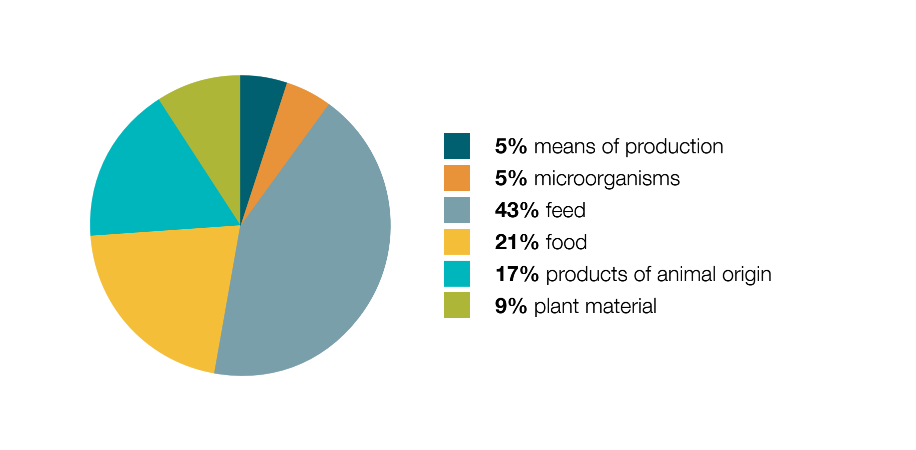

Figure 2. *Percentage of samples analysed by product classification groups. Average figures for the last three years. Source: AFL.*

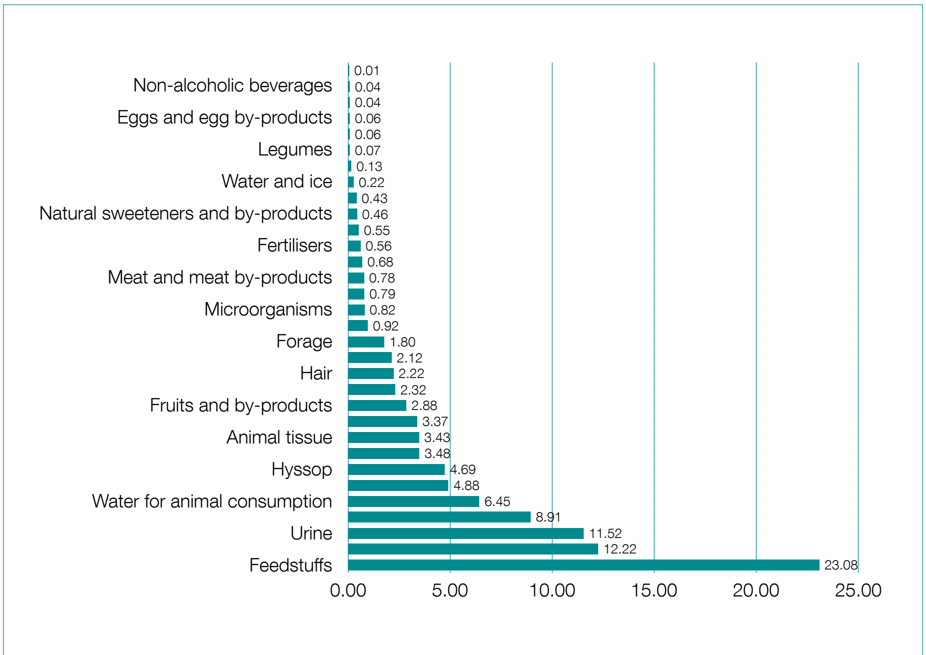

Figure 3. *Percentage of samples analysed by product type. Average figures for the last three years. Source: AFL.*

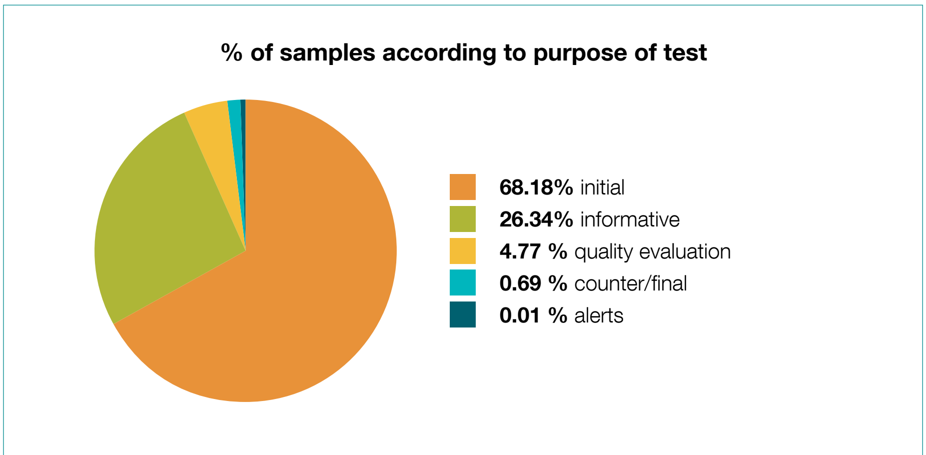

Figure 4. *Percentage of samples analysed according to the purpose of the test. Average figures for the last three years. Source: AFL.*

Table 3 shows the products analysed for each group, and Figure 2 shows the percentages of samples analysed per product group over the last three years.

05. What types of samples does it analyse?

The percentage of samples analysed according to the product classification is shown in Figure 3.

The samples analysed by the AFL according to the purpose of the test can be classified into the following groups:

- Initial (inspection and control processes). Random/targeted/due to suspicion)
- Informative (prospective, studies, projects, for issuing export certificates)
- Evaluation of the quality of the tests (own internal and/or external control)
- Counter/final (inspection and control processes). Random/targeted/due to suspicion)
- From alerts (immobilised consignments, directed, due to suspicion)

06. What analysis techniques does it apply?

The AFL does not specialise in a single technique, but instead its analytical work is very varied and applies a variety of techniques.

The instrumental analytical equipment necessary to apply the various techniques is extensive and diverse, as explained in the article "The laboratories of the DACC. Infrastructure, technical means and resources".

07. What parameters and determinations does it analyse?

The AFL currently offers a wide range of analytical services, including 1,396 different analytical determinations. The average number of determinations analysed per sample in the last three years is 10, and between 4-6 different methods are applied for each sample analysed.

The main groups of analytical determinations that the AFL has in its range of services are listed below:

- Canned food quality requirements (e.g. shortcomings, calibre)
- Rice quality requirements (e.g. shortcomings, size)
- Physical characteristics (e.g. net weight, net volume)
- Major components (e.g. fat, protein, fibre, sugars)
- Minor components (e.g. ashes, impurities, alcohols)
- Mineral components (e.g. Ca, Cu, Fe)
- Heavy metals (e.g. As, Cd, Hg, Pb)
- Additives and vitamins (e.g. ascorbic acid, sulphites, nitrites, vitamin C, vitamin E)
- Microorganisms (e.g. *Salmonella*, *Listeria*, enterobacteriacaea)
- Genetically modified organisms (e.g. qualitative analysis of endogenous genes, detection of regulatory elements for GMO screening, quantitative analysis of GMOs)
- Antimicrobials (e.g. antibiotics, coccidiostats)
- β-agonists and β-antagonists (e.g. clenbuterol, clenproperol)
- Hormones and corticosteroids (e.g. estradiol, dexamethasone)
- Natural pollutants (e.g. mycotoxins, perripolo)
- Phytosanitary products (e.g. glifosat, Folpet, 2,4D, fosmet)
- Pesticide residues (e.g. organochlorines, organophosphates)

08. Results. How are they interpreted?

The agrifood laboratories select the appropriate methods capable of meeting the client's requirements, as set out in the UNE-EN ISO/IEC 17025 standard, which is compulsory and accredited for the laboratories involved in official control.

The methods applied must be fit for purpose, relevant to the client's needs, and consistent with the specified requirements.

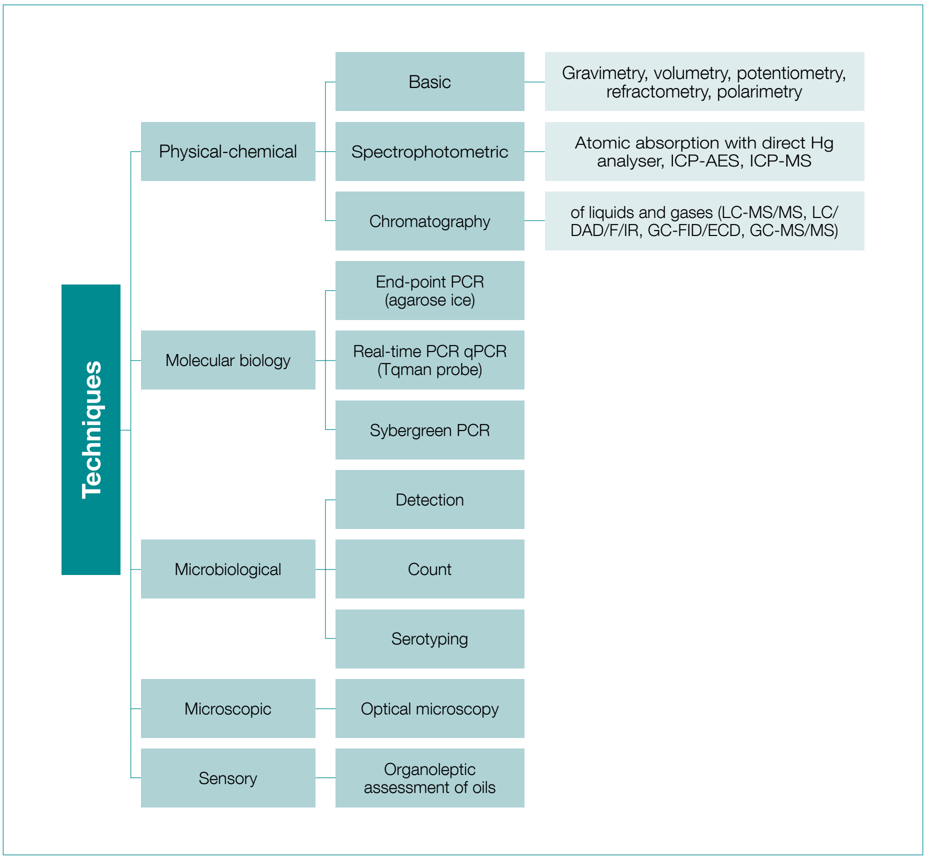

Figure 5. *Working techniques in the AFL. Source: AFL.*

After validation of the method and establishment of its quality controls, the AFL must ensure that it is aware of the applicable standard so that the client can interpret the result correctly (e.g. tolerances, specification limits, maximum limits, MRLs, etc.).

The results must be provided in an accurate, clear, unambiguous and objective manner in the results report. The results report must include all the information agreed upon with the client which is necessary for the interpretation of the results, and all the information required in the method used.

For example, when the AFL carries out analyses for the control of Maximum Residue Levels (MRLs) of pesticides in plant products, it must ensure that it is aware of and controls for compliance with the specific regulations and that the method of analysis it applies is accurate and precise at the limit of quantification (LQ), which must be equal to or lower than the MRL. The pesticide chlorothalonil in maize has an MRL of 0.01 mg/kg and the quantification limit of the method of analysis should therefore be less than or equal to 0.01 mg/kg.

The quantitative result of an analysed sample must include the uncertainty value in order to be able to interpret it correctly, and to determine whether it complies with the established MRL. The definition of uncertainty according to the ISO 3534-1 standard is "an estimate attached to a test result that characterises the range of values within which the true value is claimed to lie".

In order to interpret compliance with a specification correctly, the value of the result obtained and the associated uncertainty must be taken into account.

The possibilities for interpretation of the analytical result can be seen graphically in Figure 6 below with a pesticide residue MRL as an example.

The interpretation of the result, and

ICP-OES spectrophotometer. Photo: AFL.

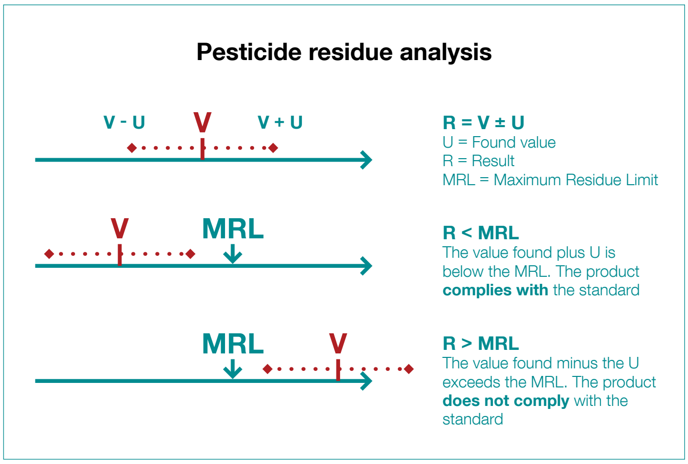

Figure 6. *Interpretation of an analytical result according to the established MRL. Source: AFL.*

thus of the compliance or conformity of the sample with the specification, taking into account the uncertainty of the method, is performed by the unit carrying out the official control. In this case, is not a task for the AFL.

The example of pesticide residues can be extrapolated to many other analytical determinations in which the AFL is involved. Contaminants in foodstuffs, such as heavy metals, mycotoxins and nitrates, also have regulatory limits. The same applies to genetically modified organisms and food and/or feed additives, etc.

In conclusion, the AFL does not receive feedback from customers on compliance or non-compliance with the specifications of the results issued and related to the control plans in which it participates.

For further information, refer to reports and other specific documents dealing with the results of official control plans.

Further reading

Spanish Plan for the Official Control of the Food Chain (mapa.gob.es)

Agri-food fraud (europa.eu)

Catalan Food Safety Agency (gencat.cat)

European Food Safety Authority | Trusted science for safe food (europa.eu)

Introduction. Ministry of Climate Action, Food and Rural Agenda (gencat.cat) Report of the Ministry of Agriculture, Livestock, Fisheries and Food 2019 (gencat.cat)

Agrifood Laboratory scope of accreditation: Search by company - ENAC Portal

ENAC NT 18 and ENAC NT 19: Accreditation documents - ENAC Portal

Agrifood Laboratory information - DACC website. Quality assurance. Ministry of Climate Action, Food and Rural Agenda (gencat.cat)

ISO 3534-1 Standard

Commission Regulation (EC) No 1881/2006 of 19 December 2006 setting maximum levels for certain contaminants in foodstuffs

Commission Regulation (EU) 574/2011 of 16 June 2011 on maximum levels for nitrite, melamine and Ambrosia spp.

Regulation (EC) No 1333/2008 of the European Parliament and of the Council of 16 December 2008 on food additives

Regulation (EC) No 1829/2003 of the European Parliament and of the Council of 22 September 2003 on genetically modified food and feed. Pesticide MRL legislation: EU legislation on MRLs (europa.eu)

SANTE/12682/2019 Guidance document on analytical quality control and method validation procedures for pesticide residues and analysis in food an feed

The methods applied must be fit for purpose, relevant to the client's needs, and consistent with the specified requirements.

In order to interpret compliance with a specification correctly, the value of the result obtained and the associated uncertainty must be taken into account.

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PARTNERSHIPS.
Research and development and innovation projects. Emerging techniques

01. The importance of partnerships with academia as a source of knowledge necessary for an official control laboratory

Food control is essential for protecting the consumer and maintaining consumer confidence in the food chain, as well as ensuring fair practices in the food trade. Due to globalisation, the food supply chain is often long and complex, and rapid breakthroughs in food technology create new types of foods with characteristics that may differ from conventional foods. This situation makes control and detection of potential non-conformities more difficult, increasing the likelihood of food alerts and crises related to food safety or fraud.

The Government, which has to enforce food authenticity and safety, needs its control bodies to have effective strategies in place to meet these new challenges. Research in food analysis and new technologies therefore plays a very important role in food control, and control systems must be kept up to date, incorporating the latest technological and methodological developments. Emerging techniques based on recent instrumental developments, such as chromatographic and mass spectrometric techniques, as well as innovative data analysis strategies, provide a number of advantages that can increase the efficiency and effectiveness of food controls. On the one hand, they provide much more comprehensive information about the food than conventional techniques, often with a much higher level of sensitivity, or with a wider range of analytes in the same analysis. These techniques usually require less sample treatments, with savings in terms of time and reagents, meaning that they can be applied in screening programmes that can cover large sample sizes, and are more representative of real production. However, the instruments, time and resources for the optimisation of these methods are not always within the reach of control laboratories, which largely work on meeting the demand for official analyses.

A prerequisite for the efficient control of agrifood products is therefore co-operation between the various parties involved in the food chain, and particularly between control bodies and research bodies, which can help laboratories to deal with health alerts and reinforce controls by complementing them with screen-

The synergy between the knowledge provided by research and the experience and competence of official control laboratories facilitates the identification and resolution of existing and emerging issues related to food safety and fraud prevention.

Analysis of veterinary drug residues at the AFL. Source: AFL.

ing methods. The synergy between knowledge in the analytical and technological field arising from research, and the experience and expertise of control laboratories in official control procedures and the needs of the food sector facilitates the identification and resolution of existing and emerging issues related to food safety and fraud prevention.

01.01 Promoters of research projects and prioritisation of lines of research

At the European, national and regional level, research into food safety, traceability and authenticity follows priorities established by government bodies, which identify the key lines in this field in their strategic plans, and by the private sector, which focuses on preventing situations that could compromise its competitiveness and image. Research funding therefore concentrates on the priorities identified by these actors, through private, public and mixed funding. Apart from private research, which is usually carried out in companies' research and development departments or in collaboration between business and academia, governments promote research through grants to universities, research centres and technology centres, usually in the form of competitive projects. Some of these funds also co-finance public-private collaborative projects. Food safety and the fight against fraud are among the European and national objectives; in particular, the priorities of the current EU budget (2021-2027) include improvement of the effectiveness, efficiency and reliability of official controls along the food chain with the objective of ensuring the correct implementation and enforcement of EU regulations in this area [1].

Research centres are experts in the workings and management of state and European research and technological innovation programmes, and as such partnerships between control laboratories and academia can help to foster links with control laboratories.

01.02 Transmission of knowledge

Knowledge transfer, which is considered to be the transmission of scientific and technological knowledge generated in universities to the productive and administrative sector, is enriching for all parties involved and therefore beneficial for society, at which the results of these interactions are aimed. As well as academic activities and research, transfer is an integral part of the mission of universities. For this reason, the objectives of the Spanish Plan for Scientific, Technical and Innovation Research (PEICTI) 2021-2023 include facilitating the transfer of knowledge and increasing the capacities for the dissemination and communication of R&D&I in society.

> The transmission of scientific and technological knowledge generated in universities to the production and administrative sector is enriching for the parties involved and therefore beneficial for society.

In the case of partnerships between control laboratories and research centres, this transfer increases the value of research results by giving them practical applications, and can be an important source of innovations and improved laboratory performance. In contrast to university-business knowledge transfer, which primarily involves contracted R&D activities, patent licensing and other intellectual property objects linked to a value chain, the transfer between academia and public oversight bodies focuses on activities of public interest. This may be governed by collaboration agreements like the one signed in 2016 between the University of Barcelona, through the Torribera Food Campus, and the Ministry of Climate Action, Food and Rural Agenda of the Generalitat de Catalunya, covering advisory activities and collaboration between the University and the Agrifood Laboratory on strategic projects and cooperation in teaching. This agreement is the result of the collaborations that the Agrifood Laboratory has historically carried out with the University of Barcelona, through the Faculty of Pharmacy and the Faculty of Chemistry.

Among the various areas of action, educational cooperation between these institutions can have an important return for society. On the one hand, the external work placement programmes established and the places offered to students to carry out bachelor's and master's degree final in the different units of the Agrifood Laboratory allow students to apply the theoretical and practical knowledge from their study programme, and contribute to the completion of their training so that they can enter the world of work in the agrifood sector with greater chances of success. The acquisition of professional skills through the combination of their academic training and professional practice improves the students' training capacity and enhances their career prospects. The tasks carried out by the students during the internship programme are also often of interest to the Laboratory, and in this context it has trained and dedicated people at its disposal to undertake innovative activities that would not be compatible with its routine work. These students also receive scientific support from their academic tutors, who can help with specific technical issues.

Knowledge transfer also encompasses knowledge dissemination activities

Olive Oil Workshop 2019. Photo: LibiFooD.

Knowledge transfer between academia and control laboratories takes place by means of support in carrying out analyses, the organisation of outreach activities and staff training.

aimed at the productive sectors, the scientific community and the general consumer. In this area, the Agrifood Laboratory has been a regular participant in activities organised by the Department of Nutrition, Food Science and Gastronomy, including the various editions of the Olive Oil Workshop (2015-2019):

- The universe of olive oil (2015)
- Sterols in olive oil. The current situation, threshold values and outliers. How do we manage diversity? (2016)
- Sensory analysis of olive oil: the current situation and new challenges (2017)
- Authenticity and control of olive oil, the current situation and new approaches (2019)
- Technical conference on Catalan honey, organised as part of a task force: Valorisation of Catalan honey (2016)

This conference focused on regulatory aspects of these foods, and the dissemination of breakthroughs in analysis to determine their quality and authenticity. At these technical conferences, the synergy between control bodies, the production sector and research in the field of analytical control enabled a definition of the current situation of the control of these products, identification of their strengths and weaknesses that constitute new challenges, and the proposal of possible analytical solutions.

Another noteworthy aspect is the importance of training laboratory technicians in state-of-the-art analytical techniques to ensure that agrifood control systems are constantly updated. Academia plays a key role in this type of training. The Department of Chemical Engineering and Analytical Chemistry of the University of Barcelona offers several courses focusing on the incorporation of the latest technological breakthroughs in current methodological approaches. Some of these are listed below:

- Confirmation of residues of plant and animal health products (2003)
- Mass spectrometry. Analytical alternatives to fraud control: integrity and authenticity (2017)
- Fundamentals of mass spectrometry: GC-MS, LC-MS, MALDI-MS
- Troubleshooting in mass spectrometry (2018)
- High resolution mass spectrometry: ion mobility and ionisation sources Environment (2021)

02. Research groups

Within the Department of Chemical Engineering and Analytical Chemistry and the Department of Nutrition, Food Science and Gastronomy, the main collaborations with the Agrifood Laboratory have been carried out by the Chromatography and Mass Spectrometry: Environmental and Food Analysis (ChroMS-EnviFood) and Lipids and Bioactive Compounds in Food (LiBiFooD) research groups.

LiBiFooD and ChroMS EnviFood logos. Source: LiBiFooD and ChroMS-EnviFood

The Chromatography and Mass Spectrometry Group: Environmental and Food Analysis (ChroMS-EnviFood), which was formerly the Analytical Chemistry-Contaminant Analysis Research Group, is a consolidated research group recognised by the Generalitat de Catalunya (2017SGR0310), with extensive experience in the development of analytical methodologies for the determination and characterisation of organic micropollutants in environmental and food samples. These methodologies use the selectivity and sensitivity provided by chromatographic techniques and mass spectrometry. The research lines focus on the study of (i) the presence, degradation and transformation of organic contaminants in the environment, (ii) the determination of contaminants and adulterants in food for the detection of food safety alerts and food fraud, (iii) the study and application of new breakthroughs in chromatographic and mass spectrometric techniques for the resolution of environmental and food problems. The research group has received continuous funding in competitive calls for projects in the Spanish R&D&I Plan, has led infrastructure projects for the acquisition of high-performance mass spectrometer instrumentation, and has participated in several European projects related to the development of nanoabsorbents for environmental remediation and the study of organic pollutants generated in thermally processed foodstuffs. The group also carries out consultancy work and collaboration activities with companies and public institutions, and engages in technology transfer through various industrial doctorates funded by the Generalitat de Catalunya. Finally, the members of this group maintain a close relationship with scientific societies, as is evident by the fact that two of them have been presidents of the Spanish societies of Chromatography and Related Techniques (SECyTA) and Mass Spectrometry (SEEM).

Chromatography room at the Torribera Food Campus, where the LiBiFooD group carries out its research. Photo: LiBiFooD.

The Agrifood Laboratory actively collaborates with the ChroMS-EnviFood and LiBiFooD research groups, which are consolidated and recognised by the Generalitat de Catalunya.

The LiBiFooD group is a consolidated research group recognised by the Generalitat de Catalunya (2017SGR1269), with extensive experience in the study of food quality and authenticity, and particularly its lipid fraction. Its research lines include (i) food authentication, using lipidomic and chemometric techniques, (ii) the study of food oxidation, stability and quality, and specifically (iii) the study of olive oil and the factors influencing its composition, sensory profile and quality; and (iv) the influence of the use of fats in animal feed on food quality and safety. The group has received continuous funding in various calls for competitive projects and among the most recent projects, the European Horizon 2020 project OLEUM-Advanced solutions for assuring the overall authenticity and quality of olive oil (OLEUM), the Spanish National R&D&I Plan project, TRACENUTS-Development of fraud detection tools for Spanish nuts with a high risk of falsification, and several regional projects, including the Autenfood project: Authentication, traceability and food safety, within the framework of the Research and Innovation Strategy for the Smart Specialisation of Catalonia (RIS3CAT), financed by ACCIÓ – Agency for Business Competitiveness. The group is also works in consultancy and cooperation with companies; within the framework of these technology transfer activities, it has completed an industrial doctorate, partially financed by the Generalitat de Catalunya.

03. Emerging analytical techniques

Analytical control laboratories are confronted with a large variety and complexity of food matrices, and a large number of compounds to be monitored which are generally present in very low concentrations in very complex matrices. The list of new compounds that need to be monitored is also growing continuously (with the development of new products, the generation of transformation by-products in production processes, contaminations that occur during production, transport and/or storage, etc.) and food alerts call for urgent results. For these reasons, control laboratories need to have effective, rapid, sensitive and targeted testing strategies to meet these challenges. Methodologies based on chromatographic and mass spectrometric techniques, together with data analysis strategies, have today become completely and indispensably integrated into the working protocols of analytical laboratories for agrifood control.

The wide range of analytical techniques available today are easily accessible to food control laboratories. Although there are a wide range of spectroscopic, electrochemical and even immunoassay methods that enable controls of a large number of major and minor compounds in agrifood samples, the selective and confirmatory determination of agrifood contaminants at very low concentrations, the identification of new compounds entering the food chain and the detection of food fraud require the potential offered by the combination of chromatographic techniques with mass spectrometry.

03.01 Chromatographic techniques coupled with mass spectrometry

Breakthroughs in chromatographic techniques have made it possible to achieve very efficient separations using capillary columns in high-resolution gas chromatography (HRGC) and columns packed with particles of less than 2 µm in ultra-high performance liquid chromatography (UHPLC). For example, the chromatographic separation of 23 organochlorine pesticides [2] and of ethoxyquin residues and their transformation products in the control of feed production in Catalonia [3] has been possible using HRGC. However, HRGC permitted the development of instrumental methods based on the volatile and semi-volatile fraction of virgin olive oil, for both its commercial classification [4] and for the verification of its geographical and varietal origin [5] -6]. On the other hand, UHPLC has made it possible to determine pigments (carotenoids, chlorophylls, etc.) and cupric dyes (E-141i) [7-8] for detecting fraud in virgin olive oils, and for the exhaustive analysis of the product's phenolic fraction [9]. It has also enabled the analysis of plasticisers including BPA and related compounds in food due to the migration of these substances from packaging [10]. In addition, breakthroughs in the development of innovative stationary phases have improved the selectivities and resolution of chromatographic separations, and offered alternative selectivities to the commonly used C18 columns. Today, columns with fluorinated stationary phases, phenyl-hexyl, zwitterionic and even hydrophilic interaction columns (HILIC, Hydrophylic Interaction Chromatography), have enabled chromatographic separations of a multitude of compound families (ranging from relatively non-polar compounds to

> Breakthroughs in chromatographic techniques have led to improvements in the separation of the various analytes and their detection, and they are now the techniques of choice in many of the determinations required in official agrifood control.

Electrospray ionisation source and ion trap mass spectrometer. Photo: ChroMS-EnviFood.

LC-MS/MS laboratory at the UB (ChroMS-EnviFood research group). Photo: ChroMS-EnviFood.

Mass spectrometric detection is very sensitive and specific, which is essential for the determination of contaminants at low concentration levels in complex agrifood matrices.

the most polar and ionic substances) [11-12]. But these improvements have also made it possible to significantly reduce analysis times and to achieve the determination of a significantly large number of target compounds in a relatively short time, which has helped to improve the productivity of food control laboratories.

Coupling these chromatographic systems with mass spectrometry instruments provides analytical tools with high selectivity and sensitivity, which are particularly necessary for the determination of contaminants at low concentration levels immersed in complex agrifood matrices. However, the biggest advantage of these coupled techniques compared to more classical analytical techniques is their ability to provide unambiguous identification based on both reproducibility of retention time measurements and the selectivity and ability to provide structural information (mass spectra) of the analytes for characterisation. These aspects are very important in current analytical methodologies, as the ability to identify and characterise has become an essential requirement in the analysis of organic compounds, and administrative requirements call for evidence for the confirmation of the presence of the compounds detected in positive samples. Furthermore, the selectivity based on chemical composition and structure provided by mass spectrometry makes it possible to address the interferences that frequently occur in complex food samples, and to reduce and simplify time-consuming and tedious sample treatments prior to analytical determination (GC-MS or LC-MS), as this step often creates a bottleneck in many food control laboratories.

03.02 Mass Spectrometry and Ionisation techniques

In the coupling of gas chromatography and liquid chromatography to mass spectrometry (GC-MS and LC-MS respectively), compounds eluted from the chromatographic column are transformed into gas-phase ions at the ionisation source and transported by electric fields into the mass analyser, where they are separated according to their mass-to-charge ratio (*m/z*) and subsequently sorted (from lowest to highest *m/z*) in the detection system. The ionisation step is therefore a critical phase in achieving maximum signal strength (ion production) and the type of ions generated. The ionisation techniques generally used in GC-MS coupling, such as electron ionisation (EI) and chemical ionisation (CI), operate in high vacuums. EI is a hard ionisation technique in which neutral molecules eluting from the column interact with an electron beam (70 eV) and an energy transfer and the generation of $[M]^{+\bullet}$ molecular ions takes place. Any excess energy that may remain in the molecular ion after ionisation can destabilise the weakest bonds in the molecule and lead to the formation of fragment ions. This fragmentation is often highly characteristic and reproducible, and is the basis for the EI spectral libraries that have been a benchmark for the identification of many of the substances analysed by GC-MS [2,13]. However, some compounds undergo high levels of fragmentation, and the molecular ion completely disappears from the mass spectrum. This poses a problem in the molecule identification process, since it is the molecular ion that provides the elemental composition information of the compound to be identified. Chemical ionisation is therefore used for some families of compounds, as it is a gentler technique, transfers less energy to the molecule and thereby avoids excessive fragmentation. In positive mode chemical ionisation (CI), in addition to the chromatographic eluent, a reactive gas (methane, methanol, ammonia, etc.) is also introduced into the ionisation chamber and this interacts with an electron beam (~12 eV) to generate gas-phase ions that interact with the neutral gas-phase analyte molecules to protonate them or form adduct ions.

Negative ions chemical ionisation (NICI) is also very useful, especially for compounds with highly electronegative functional groups, as it can capture electrons from a low-energy beam (0 -2 eV) and favours charge transfer mechanisms with ionic species generated with the reactive gases.

In contrast, the coupling of liquid chromatography to mass spectrometry was not possible until the development of atmospheric pressure ionisation sources such as electrospray ionisation (ESI) and atmospheric pressure chemical ionisation (APCI). These ionisation techniques make it possible to generate ions in the gas phase from molecules (neutral or ionised) present in the liquid phase. In ESI, a high potential is applied to the capillary of the chromatographic eluent at atmospheric pressure. This high potential causes charged droplets to be generated by an accumulation of ions (positive or negative) at the end of this capillary. The desolvation of the charged droplets, by means of a fogging gas and temperature, encourages the transport of the ions inside the charged droplets in the gas phase. This technique enables ions (single or multiply charged) to be generated from low or very low volatile analytes with masses in a wide range of values (from small ions to macromolecules). ESI is a soft ionisation technique which mainly involves the formation of protonated (positive mode) or deprotonated (negative mode) molecules and/or the formation of adduct ions with sodium, potassium, ammonium, acetate, formate, etc. ESI is currently the technique of choice in many LC-MS-based analytical methodologies [10-11]. However, compounds with functional groups that are not easily protonated or deprotonated in the liquid phase do not usually offer a good response with ESI. Furthermore, when working with ESI, the effects of ionisation suppression (or enhancement) due to competitive processes occurring during ionisation, mainly caused by the co-elution of analytes or the elution of other substances present in complex matrices of agrifood extracts, must always be taken into account, and strategies to offset these adverse effects during quantitative analysis, e.g. using the technique of matrix-matched calibration, are necessary. The use of alternative atmospheric pressure ionisation techniques such as atmospheric pressure chemical ionisation (APCI) and atmospheric pressure photoionisation (APPI) has grown in recent years to solve these problems with ESI. These two techniques are based on gas-phase ionisation mechanisms. The molecules present in the chromatographic effluent are desolvated by thermal and pneumatic assistance from a hot (400-500 °C) nebulising gas. The gas-phase molecules are subsequently ionised with APCI using a needle-shaped electrode (corona electrode) in which a high potential (3-6 kV) is applied to emit electrons that begin a cascade of gas-phase reactions that ultimately ionise the compounds eluted from the LC column. In APPI, this corona electrode is replaced by a krypton lamp that emits 10 eV photons and triggers gas-phase ion-molecule reactions. A dopant, which is a solvent that photoionises easily and improves the final response of the analytes, is also often used in this case. These two techniques are a good alternative when significant problems are encountered with ESI, as they can ionise compounds that are difficult to ionise with ESI and are at the same time less susceptible to matrix effects in the ionisation process, which facilitates the quantification process. For example, APCI-based methods for the determination of natural pigments in olive oil and azo dyes in spices that give better results and are more advantageous for food control laboratories than ESI-based methods have been proposed [7, 14]. In contrast, both APCI and APPI give only single charged ions, and are therefore not suitable for LC-MS analysis of macromolecules such as peptides or proteins, which have to be analysed by ESI. Finally, APCI and APPI sources have been designed for use in GC-MS assembly in recent years, and they provide a good alternative to conventional chemical ionisation. In addition, these atmospheric pressure ionisation techniques facilitate GC-MS coupling with state-of-the-art instruments initially designed for LC-MS, which opens up the possibility of new applications and improved productivity and reduced costs for food control laboratories, as LC-MS and GC-MS methodologies can be carried out using the same instrument. For example, a method for the determination of dioxins and furans by HRGC-Orbitrap using APPI has been proposed [15] which gives similar results to the reference method using equipment with sector mass analysers, making it possible to alternate LC-MS and GC-MS methods in the same equipment (Orbitrap) with minimal modifications.

When applied to complex samples, these GC-MS and LC-MS based methodologies require laborious sample treatments before the generally lengthy chromatographic determinations, which can have a negative impact on the productivity of food control laboratories. A number of new ionisation techniques called Ambient Ionisation Mass Spectrometry (AIMS) have been introduced in the field of agrifood analysis over the last decade. These techniques allow compounds on the surface of a sample to be ionised at atmospheric pressure with no need for treatment of the sample and/or chromatographic separation, and the analysis can be performed in a few seconds. Desorption electrospray ionisation (DESI) and direct analysis in real time (DART) are the two pioneering techniques in this field, [16] and they have now led to the development of a great deal of new AIMS techniques, and many of these are easy to implement in laboratories already working with LC-MS equipment. The UB group (ChroMS-EnviFood), in collaboration with the Cabrils Agrifood Laboratory, has developed methods for the rapid detection of veterinary drugs in animal feed and adulterants in phytosanitary products using DESI coupled to HRMS, [17-18] which enable the rapid detection of positive samples. The advantages of these cutting-edge techniques for laboratory productivity mean that we may start to see them much more frequently in the very near future.

Couplings between chromatographic systems or ambient ionisation sources and mass spectrometry instruments have evolved very rapidly to meet new analytical challenges in recent years.

03.03 Low and high resolution mass analysers

For mass analysers, the general trend to date has been the use of low resolution mass spectrometry systems able to perform tandem mass spectrometry experiments (triple quadrupole and ion traps) for robust and reliable identification and quantification of target compounds (target analysis).

Many official methods now incorporate these technologies, as they enable analytes to be determined at very low concentrations in very complex samples with the guarantee of almost unambiguous identification. However, high resolution systems (sectors, time-of-flight (TOF) and Orbitraps) must be used to monitor certain compounds in order to cope with isobaric interferences (compounds generating ions with *m/z* ratios very similar to the analytes) and to improve selectivity in order to reduce the number of false positives. Most low-resolution methodologies rely on targeted analysis strategies in order to maximise their sensitivity. Mass analysers therefore usually operate in selective ion acquisition modes that are characteristic of target analytes (SIM, selected ion monitoring), generally in GC-MS with quadrupole analysers. As a result, in GC-MS with EI, a significant number of ions are generated for each compound, making it possible to monitor at least three characteristic ions useful for both quantitative determination and confirmation at sufficiently low concentration levels in complex agrifood matrices [3]. On the other hand, to reduce background noise in GC-MS with EI, the soft ionisation sources used in LC-MS and GC-MS require the complementarity of tandem mass spectrometry (MS/MS), usually with hybrid triple quadrupole (QqQ) systems, in order to reduce the background noise and ensure confirmation with a sufficient number of characteristic ions [2-7]. In MS/MS in a QqQ, the precursor ions generated in the ionisation source are isolated in the first quadrupole and sent to the collision cell (usually a second quadrupole), where they are fragmented by collision induced by an inert gas (He, Ar, N_2) and the application of a collision energy. The product ions generated in the collision cell are analysed in the third quadrupole. The most frequently used means of acquisition is MRM (multiple reaction monitoring), in which the first and third quadrupole only allow ions with a specific *m/z* ratio to pass through each of the analytes of interest, and these are called selective "precursor ion-product ion" transitions (typically 2). These MS/MS methods have been very successful in determining a wide range of target compounds for which a standard is available to determine the retention time and to establish the characteristic ions to be monitored, as well as to obtain calibration curves for quantitative analysis [2, 7].

These targeted methodological strategies do not meet the new challenges facing agrifood analysis today, including the detection of suspect compounds and the identification and characterisation of unknown compounds. Methodological breakthroughs in this area have focused on using non-targeted strategies to detect and identify an unlimited number of compounds in a single analysis. High-resolution mass analysers operating in full ion scanning mode with high sensitivity are today the cornerstone for performing this type of non-targeted analysis. Mass analysers such as TOF and Orbitraps have become popular in both LC-MS and GC-MS. These systems provide accurate measurements of the mass of the ions detected (to 4-5 decimal places with less than 5 ppm accuracy in mass measurements) and information on the isotopic distribution, enabling an accurate assignment of the elemental composition of the ions detected, which is important in the structural elucidation of suspected and unknown compounds. The direct application of ESI-MS with an Orbitrap analyser has even made it possible to obtain – without prior chromatographic separation – a triacylglycerol profile of olive oil that is so detailed that large-scale authentication models (more than 1,000 samples) capable of detecting up to 2% and 5% of adulterants high in linoleic acid (sunflower and soybean oil) or oleic acid (hazelnut, avocado, high oleic sunflower oil) in less than 2 minutes have been developed [19].

LC-HRMS: Atmospheric pressure chemical ionisation source (APCI) and hybrid quadrupole-Orbitrap mass spectrometer. Photo: ChroMS-EnviFood.

The different configurations of mass analysers answer the question of whether an analyte is in a sample (target analysis) and in certain configurations (high resolution), which analytes are in a sample (target analysis).

03.04 Ion mobility

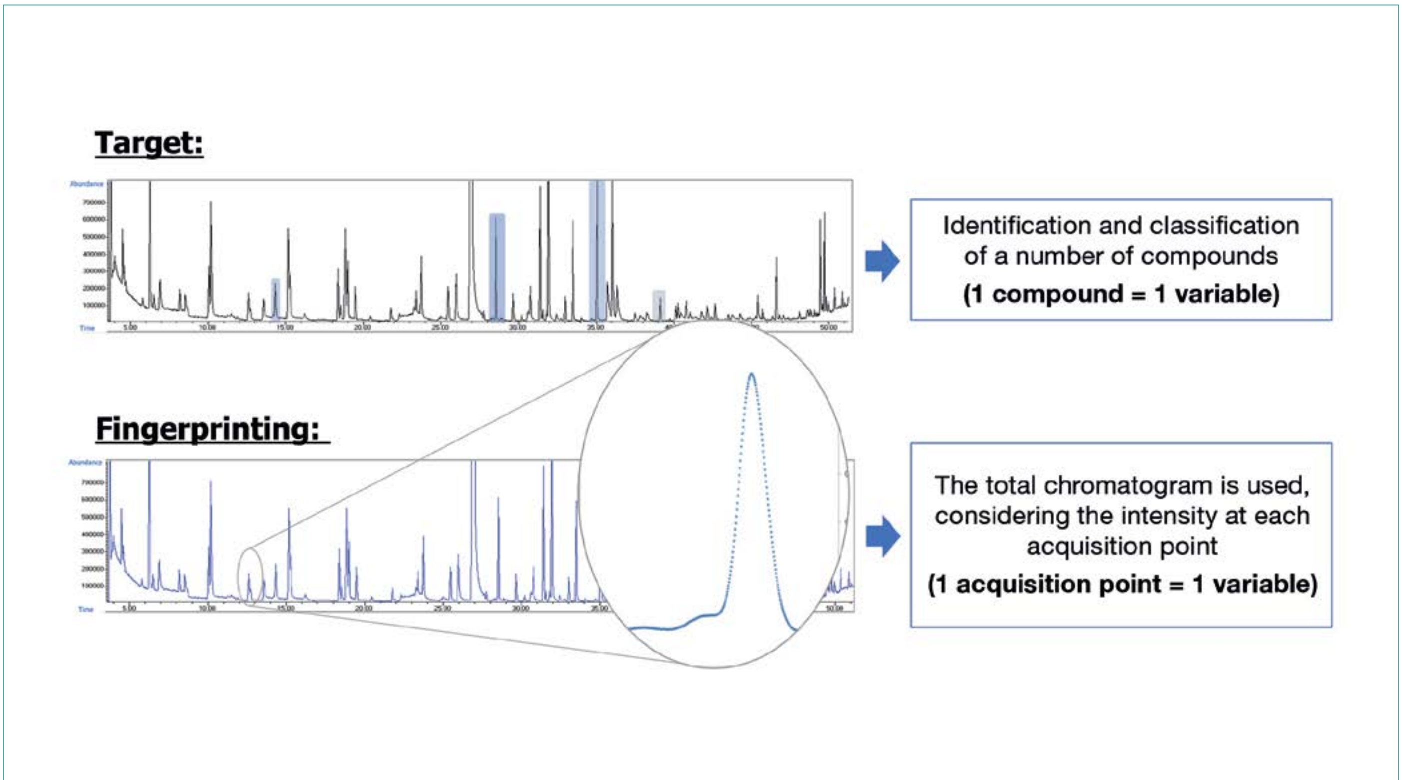
Despite the major advantages offered by high-resolution mass spectrometry, very complex samples and mixtures can present new challenges that may hinder identifications/characterisations and increase background noise, such as the presence of interfering compounds with very similar or identical *m/z* values (isobaric ions) to the analytes of interest, or even the co-elution of isomeric compounds.

> Ion mobility makes it possible to distinguish ions according to their spatial shape as well as their mass and charge, and to provide CCS (Cross Collisional Section) values, which are directly related to the three-dimensionality of the molecules.

Fortunately, technological breakthroughs in the last decade have provided a new tool, ionic mobility, in the agrifood field [20]. This instrumentation, which is generally implemented in Q-TOF hybrid instruments, makes it possible to differentiate ions according to their spatial form as well as their mass and charge, and to provide CCS (Cross Collisional Section) values, which are directly related to the three-dimensionality of the molecules. In these devices, ions enter a pressurised ionic mobility cell with an inert gas to which a potential is applied. As they move through this cell, the ions reach the Q-TOF system according to their aerodynamics. Smaller, less charged and more compact (folded) ions arrive earlier and are larger, and those with higher charges which are more unfolded arrive later. The time taken for the ions to cross the cell is directly related to their three-dimensional shape, and these times are transformed into CCS values, which are incorporated into the GC-HRMS/MS and LC-HRMS/MS data (retention time, precursor ions, product ions), giving them an additional dimension. The chromatograms and mass spectra obtained with this technology can be filtered using the specific CCS characteristic values for each compound, which makes it possible to improve the specificity of the methods and reduce the background noise to obtain much lower detection limits. Ion mobility is today widely used in methods for authentication and fraud identification in the agrifood field [21].

03.05 Emerging analytical approaches: marking (fingerprinting)

In addition to the analytical techniques applied to detection, identification and quantification, how analytical data is processed can play a major role in the assessment of food quality and authenticity. In contrast to traditional methodological approaches based on the determi-



Targeting and fingerprinting approaches to data processing. Source: LiBiFooD and ChroMS-EnviFood.

nation of several markers and comparing them with reference values, innovative approaches such as fingerprinting rely on the raw analytical signal provided by spectroscopic or chromatographic techniques. This approach uses chemometric tools to find specific patterns for a specific type of food (depending on its commercial category, origin, or other distinguishing features) that can differentiate it from foods that do not belong to the same category. Unlike conventional methodologies, which target a limited number of compounds that may be insufficient for authentication, a large amount of information can be taken into account in fingerprinting, and it is possible to distinguish authentic samples, or samples with a certain degree of quality, from non-authentic samples more efficiently. Fingerprinting is being successfully applied in food authentication for this reason. The LiBiFood group has obtained promising results for the geographical and varietal authentication of foods, for the detection of adulteration, and for the evaluation of the sensory quality of virgin olive oils [4- 6]. The major potential of fingerprinting for the volatile fraction of virgin olive oil as an instrumental method to support the tasting panel was demonstrated in the OLEUM project [4] . One of the challenges of fingerprinting methodologies is their validation, as it is necessary to provide a repeatable and reproducible chromatographic signal, and the conventional criteria adopted to evaluate the performance of target methods are not applicable. Ensuring the quality and comparability of analytical results obtained in different laboratories is a requisite for considering the method as a control tool. For this reason, the fingerprinting of the volatile fraction obtained by SPME (Solid Phase Microextraction) coupled to GC-MS was included in the interlaboratory validation activities in the OLEUM project, in which the Agrifood Laboratory was extensively involved. The involvement of both official analytical laboratories and the Agrifood Laboratory and private laboratories was crucial to the success of the validation studies. The inter-laboratory study included a training phase that gave new skills to participants who were not experts in analytical techniques, and a problem sample analysis stage. Feedback from the participants and the results obtained have enabled identification of some of the main reasons for variability between the participants, and the examination of some of the differences between signals observed and proposals for data processing solutions to correct them. The results obtained are an important step in the validation of non-conventional methods such as fingerprinting, which will provide a starting point for future research. However, laboratories have been able to incorporate some of the emerging methodologies developed within the scope of this European research project into their areas of work, demonstrating the importance of cooperation between academia and control laboratories.

Fingerprinting strategies can make it possible to efficiently distinguish authentic samples, or samples with a certain degree of quality from non-authentic samples. They are being successfully applied in the geographical and varietal authentication of foods, the detection of adulteration, and the evaluation of the sensory quality of virgin olive oils.

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CHALLENGES FOR THE FUTURE

01. Introduction

European regulations establish a harmonised framework for the organisation of official controls throughout the agrifood chain. Competent authorities are required to verify that operators comply with European Union (EU) rules, and that food meets specific requirements. As a link in the control chain, official agrifood control laboratories have to work with accreditation according to the UNE-EN ISO/IEC 17025 standard. The aim of this accreditation is to provide confidence: our results must be seen to be valid and we must be seen to be technically competent, i.e. we must not only work well, but also demonstrate this.

We must ensure that the results of the official control laboratories are valid and that we are technically competent, i.e. we must not only work well but also be seen to be doing so.

Official agrifood control covers both the safety and quality of food for both human and animal consumption. Depending on the ministry to which they belong and the distribution of competences, official laboratories focus on different areas to varying degrees. However, official laboratories have close links and shared challenges, and aim to be a useful tool for inspection bodies in the search for banned or undesirable substances in food and feed, and in the fight against agrifood fraud. The ultimate goal of laboratories is to contribute to ensuring food quality and safety throughout the food chain. Our future lies in a common strategy.

In this article, we identify the challenges for the future for the Agrifood Laboratory of Catalonia (AFL). This takes it the internal vision of the Laboratory itself and the external vision of the Barcelona Public Health Agency Laboratory, with whom we share tasks and objectives.

02. The context of official control laboratories

An overview of the global, social and economic context shows us that the world is changing very quickly. The climate emergency, globalisation and the pandemic caused by the SARS-CoV-2 coronavirus are key factors in agrifood control policies and will continue to be so in the future. Global warming is already affecting local food production systems. The EU has set itself the goal of becoming the first CO2 neutral continent. Agrifood control laboratories are involved in the issues of climate change mitigation and adaptation, circularity and sustainability in the design of food production policies, the efficient use of resources, research, innovation and technology transfer.

The pandemic and the development of vaccines against the virus that causes the have shown that investment in research and technology can be responsive and provide rapid solutions to highly complex problems. This work took place in laboratories.

Due to globalisation, foodstuffs produced in other parts of the world are often present in our market, and they must comply with strict European regulations if they are to be consumed. The work of laboratories is essential in establishing new analysis methods for these foods to ensure both their safety and their origin and/or authenticity.

Analysis process. Photo: AFL

The global transformation of the food production system to a large extent depends on alternative sources of protein. As raw materials and a new source of protein, insects and algae will contain undesirable products requiring controls; there are also emerging problems, such as the increasing presence of mycotoxins in feed and food caused by global warming. At the same time, European regulations are lowering the thresholds permitted for various substances such as pesticides and veterinary drug residues in animal feed.

The population's new eating habits entail an increase in the consumption of organic, vegetarian and/or vegan products. Food intolerances and allergies are becoming increasingly common, the market is offering more and more products for this type of consumption. The substitution, dilution, concealment and use of unauthorised substances and fraudulent products must also be monitored.

These are just a few examples of the many issues that have to be taken into account in the work of the official control laboratory when controlling the quality and safety of foodstuffs.

Likewise, despite globalisation, we must consider the dimension of the Catalan food system, its role and its value. Its objectives are: to be sustainable, transformative and based on the circular bioeconomy; local and rooted in the territory; fair, equitable and cohesive, and healthy and trustworthy.

Gas chromatograph with single quadrupole mass spectrometric detector. Photo: AFL

Data processing. Photo: AFL.

The pandemic and the development of coronavirus vaccines have shown that investment in research and technology can be responsive and provide rapid solutions to highly complex problems. This has been done in laboratories.

02.01 The internal working context

As a key player in agrifood control, which is also affected by the climatic emergency, the Agrifood Laboratory must take maximise its environmental management measures. Restricting energy consumption and the waste management of hazardous substances is a key objective and a priority. Investments are therefore necessary in the infrastructure of the laboratory building (climate, lighting, insulation of spaces, working furniture and storage of reagents, materials and their waste). The Laboratory must have an infrastructure with the maximum safeguards of safety, efficiency and respect for the environment.

The development of instrumentation in recent years has been one of the cornerstones of the Laboratory's work in the field of chemical analysis. The

Laboratory's work has considerably expanded, and ranges from the assembly of chromatographic separation techniques to mass spectrometry to benchtop instrumentation. Apart from instrumentation, the development of generic extraction methods for compounds of interest quickly, simply and inexpensively has been of particular interest, as no expensive materials are needed. This generic extraction methodology, associated with the use of new separation and detection technologies, is a powerful tool now available to public laboratories.

Technologies developed more recently include high-performance chromatographic techniques and mass spectrometry, which has evolved rapidly in recent years, in both the low- and high-resolution fields. The use of high-resolution mass spectrometry resolves analytical problems where low resolution shows its limitations, e.g. in the presence of co-eluting isobaric interferences, which can lead to errors when issuing laboratory results.

In the field of microbiology and molecular biology, the pandemic has highlighted methods for the detection and confirmation of the presence of the SARS-CoV2 virus in samples of human origin. Compared to the situation prior to the pandemic, the public is much more knowledgeable about immunochromatography methods ("rapid tests") and the PCR (Polymerase Chain Reaction) method for detecting the presence of the virus. The latter is the increasingly common method used in the field of microbiological analysis as a screening method to obtain rapid results and to anticipate the results from culture methods, as its turnaround time can be hours compared to official methods; this is the case with Legionella analysis in water samples, for which the culture method requires 10 days, whereas the PCR method provides results within hours. However, if the PCR result is positive, the culture method must still be completed, as this is the established method for official controls of Legionella and enables the isolated microorganism to be obtained, which makes subsequent epidemiological studies possible. Because PCR is a technique that is based on the identification of a genomic sequence, it provides high levels of sensitivity and specificity. This is a very useful technique in the field of non-culturable pathogens, as is currently the case for the norovirus.

Molecular techniques mean that part of the process can be automated. Automated extractors are used on an increasingly widespread basis in official laboratories to speed up the extraction of nucleic acids for the subsequent detection of microorganisms. One of the future avenues for the PCR technique may lie in the digital-PCR technique, which provides quantification without the use of a standard line, thereby saving on analysis time and reagent costs.

Another possible future direction in the field of molecular biology is the application of NGS (Next Generation Sequencing) techniques, through which many more microorganisms can be detected simultaneously in a single sample analysis.

Apart from molecular techniques, attempts are also being made to obtain results more quickly and easily in the field of culture methods. For example, the alternative method for rapid monitoring of bathing water quality for coliforms and *E. coli* has already been adopted in Catalonia. Some of these methods have already been certified as alternatives to ISO standards, and can therefore now be used in official controls.

A future challenge for official control laboratories is to be equipped with the latest technology on the market in order to be able to deliver highly reliable results as quickly as possible.

This latter point is linked to one of the cornerstones of work in the laboratory, which is the need for highly qualified personnel with expertise in analytical techniques and food matrices. When working in the laboratory, the most important factor is the people involved. Another future challenge for laboratories is therefore both to have the capacity to recruit qualified staff and to retain trained staff who add value. Innovation in the public procurement model is required in order to be able to recruit and retain talent and knowledge.

Robot for extracting nucleic acids. Photo: Barcelona Public Health Agency.

Moreover, laboratories are regularly assessed for their compliance with the UNE-EN ISO/IEC 17025 standard by means of internal and external audits. The Spanish accreditation system is very inflexible, and not adapted to the changing circumstances mentioned above. In order to be a useful tool for control of the food chain, a broad and flexible scope of accreditation is required that enables results to be issued without the need for going through the entire process, and avoiding gaps in the accredited range of official control laboratories.

In addition, laboratories' responsiveness is a prerequisite in routine work and in urgent situations (targeted actions, suspicions or food crises). It is therefore essential to have a large, stable and qualified workforce; state-of-the-art analytical equipment (which is efficient, sensitive, accurate and robust), and an open and flexible scope of accreditation. They must be ready and prepared for what is required of them, so that the effectiveness of official controls are not compromised.

Prospective studies to identify potential fraud and/or emerging risks prior to the establishment of control plans are necessary. The state of the art in this area must be determined. Laboratories must together on the promotion and participation of this type of campaign, developing methodologies and providing crucial information for control bodies.

02.02 The external working context

The changing environment of climate emergency, globalisation and pandemic that we are currently experiencing highlights the need for cooperation between different branches of government. As agrifood control cannot be achieved by a single laboratory, specialisation is necessary and obvious. Specialised laboratories optimise resources and make their work more cost-effective; in short, they are more efficient. Specialised laboratories can therefore form a network to provide a rapid response to the challenges presented by food control and food alerts, within the framework of the European Union, and the free movement of goods between Member States. Work and collaboration between laboratories must be reinforced to provide the highest levels of service.

The Agrifood Laboratory must therefore invest in state-of-the-art analytical equipment. It must be able to apply the techniques set out in the recommendations and EU regulations without any restrictions, but it must also go a step further. It must position itself as a benchmark in its field of expertise, in its speciality.

In the field of animal feed, Catalonia is very important considering that it is at the forefront of feed production in Spain, and Spain being the leading producer of compound feed in the EU and the tenth largest in the world.

Official control laboratories must have an infrastructure with the maximum safeguards of safety, efficiency and respect for the environment; the latest technology available on the market, in order to be able to deliver highly reliable results as quickly as possible; and qualified personnel, which requires innovation in the public procurement model, and a broad and flexible scope of accreditation, which allows results to be issued without having to go through the entire established process and avoids gaps in the accredited range. In addition, it is necessary to be ready in order not to compromise the effectiveness of official control and to assist in the identification of fraud and/ or emerging risks, develop methodology and provide key information.

Liquid chromatograph coupled to high resolution mass spectrometry (UHPLC-HRMS). Photo: Barcelona Public Health Agency

Specialised laboratories optimise resources and make their work more cost-effective. Work and collaboration between laboratories must be reinforced to provide the highest levels of service. A major challenge for the AFL is to become a national reference laboratory in the field of feed and its raw materials. Working to become a benchmark is the path to excellence.

Another objective is joint participation in studies and projects and collaboration in the training of future professionals from universities and technology centres.

Feed and feed materials, premixtures and/or additives (technological, zootechnical, nutritional, organoleptic, coccidiostatic and histomonostatic) have a very wide range of compounds and substances requiring control, from their composition to residues, including medicinal products (e.g. antibiotics), pesticides (MRL), undesirable products (e.g. mycotoxins) or pollutants (e.g. heavy metals). The same applies to microorganisms (e.g. Salmonella) and genetically modified organisms.

A major challenge for the Laboratory is to become a national reference laboratory; in this case, in the field of feed and feed materials.

Among other tasks, the national reference laboratories, among other functions must collaborate with the European Union reference laboratories, participate in the training courses and interlaboratory tests organised by them, and coordinate the activities of the designated official laboratories in order to harmonise and improve analysis methods. Contact with European reference laboratories leads to a continuous updating of analytical methodology and instrumental techniques, and participation in the development of new European standards. Working towards being a benchmark is therefore the path to excellence.

Links with universities and research and technology centres also need to be strengthened. Joint participation in studies and projects and collaboration in the training of future professionals (degrees, masters, doctorates) is a win-win situation. Enhancing the visibility and disseminating the work done is essential: papers must be published and presented by attending scientific events; in short, the task work must be visible. All this will enable us to obtain resources and knowledge, and to position the laboratories at the highest level.

03. Conclusions

The challenges mentioned in the context described in this article can be summarised as follows:

- Have an equipped, safe and environmentally friendly infrastructure.
- Have efficient, sensitive and robust analysis equipment adapted to the latest technological developments in the market.
- Have a sufficient, stable and qualified workforce and be able to attract and retain talent.
- Have the responsiveness required for emerging problems, and be a useful tool to meet the demands of inspection bodies.
- Promote visibility and presence, which requires closer links between laboratories and the creation of an operational network including universities and research centres.
- Work towards specialisation through cooperation between laboratories and to become benchmarks.
- Have accreditation under the UNE-EN ISO/IEC 17025 standard with a broad and flexible scope, adapted to the needs of food control.

Further reading

Agrifood Laboratory
http://agricultura.gencat.cat/ca/ambits/alimentacio/laboratori-agroalimentari/presentacio-laboratori-agroalimentari

UNE-EN ISO/IEC 17025 Standard
https://www.en.une.org/encuentra-tu-norma/busca-tu-norma/norma/?c=N0059467

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Strategic Food Plan for Catalonia 2021-2026
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Barcelona Public Health Agency Laboratory
https://www.aspb.cat/arees/laboratori/introduccio

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We talk to: DR CHRISTOPH VON HOLST

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Christoph von Holst works at the European Commission's Joint Research Centre (JRC) in Geel (Belgium). He studied chemistry and received his doctor degree in analytical chemistry from the Technical University Munich (Germany). In 1997 he joined the JRC, working on various topics of food and feed analysis such as pesticides, meat and bone meal, chloroorganic compounds, mycotoxins and feed additive. Since 2003 he is charge of the European Union Reference Laboratory (EURL) for feed additives. He is very much interested in statistical data treatment, especially in the field of chemometry and is co-author of about 90 articles in peer reviewed journals.

"The EURLs help the implementation of EU Regulations in the area of food and animal nutrition, focusing on the control tasks of the Member States"

What is the role of the European Union Reference Laboratories (EURL)?

EURLs exists in various fields, but in this interview I am going to focus on EURLs that have been established in the area for food and feed, separately for the various topics such as pesticides, contaminants, genetically modified food and feed, animal proteins and feed additives. EURLs are important, because many Regulations on this topic are established at EU level, while the EU Member States are in charge of correct implementation of these rules. For instance, the European Commission sets maximum levels of contaminants in food or feed and the official control laboratories in the Member are then analysing samples from the market to check, whether these criteria are fulfilled. For a well-functioning of the EU market it is therefore of paramount importance that the official control tasks are conducted in a harmonised manner and that the laboratories in the Member States can demonstrate sufficient expertise in the analysis of food and feed samples. One of the major objective of EURLs is to ensure that these laboratories enforce the rules set at EU levels by producing analytical results with the required quality.

One of the major objective of EURLs is to ensure that these laboratories enforce the rules set at EU levels by producing analytical results with the required quality

Can you briefly describe their relationship with the national reference laboratories? How do you rate this relationship?

EURLs establish networks with National Reference Laboratories (NRLs) that are officially nominated by the corresponding Member States. The EURLs and the Directorate General for Health and Food Safety of the European Commission set up annual programmes that specify e.g. for which substance and in which food or feed matrix proficiency tests (PTs) are organised. In such PTs, the EURL prepares samples containing the substance at the level of interest, sending them to all NRLs with the request for analysis. The analytical results are then reported back to the EURL, which conducts a statistical assessment of the results to get an estimate of the laboratory specific performance in this exercise. Such an information is a key criterion for the NRLs to demonstrate their expertise but also to improve if required. Furthermore, the EURL organises annual workshops with the NRLs to discuss the results of the PTs and other relevant topics.

How would you describe these laboratories' current situation? What resources are they lacking?

The results from the high number of PTs organised by the EURLs in the various fields demonstrate the performance of the NRLs regarding the analysis of food and feed samples. On the other hand, the work of the EURL helps identify topics, where specific laboratories have to improve. In annual workshops organised by the EURL, rather technical discussions take place to foster the exchange of knowledge, thus continuously improving the competence level of all laboratories.

The results from the high number of PTs organised by the EURLs in the various fields demonstrate the performance of the NRLs regarding the analysis of food and feed samples

With regard to animal nutrition, what matrices and analytical determinations are currently used?

The final feed given to the animal is often a complex mixture comprised of feed materials, premixtures and feed additive preparations. In principle, all of these matrices may be included in monitoring programmes of official control laboratories, in order to check for correct labelling information on the food and feed and the compliance with legal limits. Given the enormous diversity of target analytes, also the analytical methods differ a lot. For instance, for the determination of veterinary drugs liquid chromatography coupled to mass spectrometry is applied, while some elements are analysed with inductively coupled plasma atomic emission spectrometry.

With regard to the European Union's objective to become the first carbon neutral continent, what role do the EURLs play?

The EURLs help the implementation of EU Regulations in the area of food and animal nutrition, focusing on the control tasks of the Member States. This will certainly also apply to all measures planned within the frame of the European green deal.

What is the trend in analytical techniques? What investments do you consider to be priority?

This is a very difficult question, given the extreme variety of the analytical disciplines involved. However, there is certainly the trend, to apply more and more multi-analyte methods using instrumentation that enable the simultaneous determination of a high number of different compounds with similar physical-chemical characteristics such as mycotoxins, plant toxins or pesticides. Likewise there is a need for low cost and easy to use instrumentations that allow for rapid screening of samples. Some of these tests are even used on-site, thus ensuring that only the smaller fraction of suspect positive samples from all screened samples are subjected to confirmatory analysis requiring expensive instrumentation. All these efforts will certainly improve the efficiency of official control with the resources available.

There is certainly the trend, to apply more and more multi-analyte methods using instrumentation that enable the simultaneous determination of a high number of different compounds with similar physical-chemical characteristics

What are the future challenges for agri-food laboratories?

One of the major challenge is to keep abreast with ever changing topics and requirements for control laboratory. For instance, the climate change may impact the presence of plant pathogens or natural contaminants in food and feed samples, thus requiring the laboratories to implement new method to measure these substances. Also control programmes in the area of food and feed authenticity are getting more and more important, reflecting the increasing demands from the consumer. Another aspect concerns new control requirements due to the use of alternative feed materials such as insects. Finally, I would like to highlight that these are just examples for a high number of quite different topics laboratories are facing.

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