UNDERSTANDING WHAT IS NEEDED TO PRODUCE QUALITY DATA

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Overview of the presentation

1. Quality Control: Why?
2. Basic accreditation: ISO/IEC 17025
3. Understanding what is needed to produce quality data
   3.1 Method validation: a quick intro
4. QA in practice
5. About Food Chemistry Lab.
Quality Control: Why?

1. You want to deliver correct results
2. Your customer demands it Accreditation
3. Marketing purposes
## Quality Control: Why?

### ISO Certification Standards

<table>
<thead>
<tr>
<th>ISO 9001</th>
<th>ISO 17025</th>
<th>ISO 15189</th>
</tr>
</thead>
<tbody>
<tr>
<td>CERTIFICAÇÃO</td>
<td>ACREDITAÇÃO</td>
<td>ACREDITAÇÃO</td>
</tr>
<tr>
<td>Quality management systems -- Requirements</td>
<td>General requirements for the competence of testing and calibration laboratories</td>
<td>Medical laboratories -- Requirements for quality and competence</td>
</tr>
</tbody>
</table>
Quality Control: Why?

QS: based on the Deming cycle (continuous improvement / living system)
Quality Control: Why?

The quality assurance cycle

- Pre-Analytic:
  - Patient/Client Prep
  - Sample Collection

- Analytic:
  - Data and Lab Management
  - Safety
  - Customer Service

- Post-Analytic:
  - Reporting
  - Record Keeping
  - Quality Control Testing

- Sample Receipt and Accessioning

- Personnel Competency Test Evaluations

- Sample Transport
ISO/IEC 17025

General requirements for the competence of testing and calibration laboratories

- Scope
- Normative References
- Terms and Definitions
- Management requirements
- Technical requirements
## ISO/IEC 17025 Management requirements

- Policy
- Organisation
- Document control
- Review of requests, tenders and contracts
- Subcontracting of tests
- Purchasing services and supplies
- Service to the customer
- Complaints and non conformities
- **Corrective and preventive actions**
- Control of records
- Internal audits and management review
ISO/IEC 17025

ISO/IEC 17025 Management requirements

Complaints and non conformities
- Registration of the deviation
- Causal analysis
- Corrective and preventive actions
- Evaluation
- Continuous improvement
ISO/IEC 17025 Technical requirements

- Personnel
- Accommodation and environmental conditions
- Equipment
- Selection of methods
- Measurement traceability
- Handling of samples
- Assuring quality of test results
- Reporting of results
ISO/IEC 17025 Technical requirements

<table>
<thead>
<tr>
<th>Personnel</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Competence – relevant knowledge and experience</td>
<td>OK</td>
</tr>
<tr>
<td>Training and development</td>
<td></td>
</tr>
<tr>
<td>Job descriptions</td>
<td>OK</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Accommodation and environmental conditions</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Suitable for the correct performance of the tests</td>
<td>OK</td>
</tr>
<tr>
<td>Monitor environmental - storage conditions</td>
<td></td>
</tr>
<tr>
<td>Controlled access</td>
<td>OK</td>
</tr>
</tbody>
</table>
ISO/IEC 17025 Technical requirements

**Equipment**

- Comply with specifications, relevant to the tests
- Operating instructions
- Identification
- Preventive maintenance
- Calibration
ISO/IEC 17025

**ISO/IEC 17025 Technical requirements**

- Test methods and method validation
  - Appropriate methods (fit for purpose)
  - Preferably international or national standards
  - Validation of in-house methods
  - Estimation of uncertainty of measurement
  - Control of data (calculations and data transfers)
ISO/IEC 17025

ISO/IEC 17025 STAC principle

- **S**ee (look – observe)
- **T**hink (start brain flow)
- **A**ct (notes – SOP’s)
- **C**ommunicate (share the knowledge)
UNDERSTANDING WHAT IS NEEDED TO PRODUCE QUALITY DATA

• With so many fictional crime solving dramas on television it’s easy to believe that analytical results can be determined instantaneously.

• Without adequate quality control data, results can be misleading or incorrect.
Use the Correct Method

• Choosing the appropriate method is the **first critical step** in ensuring accurate and reliable results.

• Methods **published by industry-recognized organizations** is a good start.

• Most widely used methods come from the AOAC, ISO or CEN and are often called “Standard Methods.”
Use the Correct Method

The following factors must be considered when choosing a method:

- objective of the test,
- type of matrix,
- detection limit,
- turnaround time needs.
Verify or Validate the Method

• **Method verification** is a process in which a lab proves they are able to perform a Standard Method correctly.

• **Method validation** is the process in which a lab proves a non-standard method, such as a method developed in-house, actually works as intended and meets the requirements of a client.

In short: know what it can do and what to expect
Verify or Validate the Method

Table 1. Analytical performance parameters

<table>
<thead>
<tr>
<th>Analytical Performance Parameter</th>
<th>Assays</th>
<th>Quant.</th>
<th>Limit test</th>
<th>Specific tests</th>
<th>I.D.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Accuracy</td>
<td>Yes</td>
<td>Yes</td>
<td>*</td>
<td>*</td>
<td>No</td>
</tr>
<tr>
<td>Precision</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
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<tr>
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<td>Yes</td>
<td>Yes</td>
<td>Yes</td>
<td>*</td>
<td>Yes</td>
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<tr>
<td>LOD</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
<td>*</td>
<td>No</td>
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<tr>
<td>LOQ</td>
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<td>No</td>
<td>*</td>
<td>No</td>
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<tr>
<td>Linearity</td>
<td>Yes</td>
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<td>No</td>
<td>*</td>
<td>No</td>
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<tr>
<td>Range</td>
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<td>Yes</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Robustness</td>
<td>Yes</td>
<td>Yes</td>
<td>No</td>
<td>Yes</td>
<td>No</td>
</tr>
</tbody>
</table>

+ measurement uncertainty

» * May be required, depending on the nature of the specific test.

Table 1 below, summarizes the parameters required to validate a method.
Elements of Method Verification or Validation

Accuracy and Precision

• **Accuracy** is the *closeness of the test results obtained by the method to the actual or true value.*

• **Precision** is the *degree of agreement among individual test results when the procedure is applied repeatedly to multiple samplings of a homogeneous sample.*
Elements of Method Verification or Validation

Figure 1: The Relationship Between Accuracy and Precision

- **Accurate** (The average is accurate, not precise)
- **Precise** (Not accurate)
- **Accurate and Precise**
Elements of Method Verification or Validation

Accuracy

Accuracy can be determined by comparing the results of a series of samples to the true value.

The true value can be:

- published results of a certified reference material,
- calculated by consensus of multiple laboratories, or comparison of the results of fortified samples, also referred to as spiked samples, against the published method.
Elements of Method Verification or Validation

**Precision**

*Precision can be determined by calculating the standard deviation of a series of like samples.*

**Selectivity/ Specificity**

*Selectivity refers to the extent to which a method can determine particular analytes in mixtures or matrices without interferences from other components.*
Elements of Method Verification or Validation

IUPAC: “Specificity is the ultimate selectivity” and “Sometimes the term specificity is used (100% selectivity (or 0% interferences).

If you have a sample, with matrix and ingredients (e.g. more than one ingredient), and you can only detect one ingredient. That method is specific for that ingredient.

If you can separate and detect more ingredients without interferences, than that method is selective.
Elements of Method Verification or Validation

Detection Limit (LOD)
Lowest concentration of analyte in a sample that can be detected (not necessarily quantitated)
Not useful in practice

Quantification Limit (LOQ)
Lowest concentration of analyte in a sample that can be determined with acceptable precision and accuracy

The rule is:

- LOD is 3 times the noise (standard deviation) above the blank response, and the LOQ is 10 times the noise.
- LOD and LOQ are determined by calculating the standard deviation of a series of samples at the suspected LOD.
Elements of Method Verification or Validation

**Linearity:** compares the relationship between an instrument response and concentration of an analyte. The ideal situation is when there is a one to one linear response.

![Example Standard Curve to Demonstrate Linearity](image)

To determine the linearity, a series of analytical standards at different concentrations is analyzed.
Elements of Method Verification or Validation

Range
Range is the upper and lower concentration limits of the analyte that can be determined with precision, accuracy, and linearity.

Range is determined by fortifying a blank matrix with a representative analyte at the highest expected value of your samples.

Robustness
Measure of the capacity to remain unaffected by small (deliberate) variations in method parameters.
Use Appropriate Standards

Many methods require analytical standards to properly identify the analyte and/or determine the concentration.

No matter the source, all standards should have a certificate of analyses to prove authenticity.

» Selection of reference materials ("standards")
» Internal standard(s)
» External standard(s)
» Calibration standards
Participate in Proficiency Programs

- ISO 17025 accredited labs must participate in proficiency testing (PT) programs on a yearly basis.
- PT programs enable laboratories to assess their performance by comparing their data with other laboratories.
Perform Internal Quality Control

Once the **right method and standards have been chosen and validated** and the lab has proven its proficiency, the next step is **having systems in place to ensure that everything goes right on any given day**.

This can be done through the use of **Laboratory Control Samples (LCS)**, also referred to as **Quality Control (QC) samples**.
Perform Internal Quality Control

- LCS are samples with known true values.
- Running LCS with every batch of samples will demonstrate accuracy every time the test is run.
- The type of LCS is chosen to reflect the qualities of common client samples.
- Spiking of client samples may also be performed for difficult or uncommon matrices to check for sample matrix effects.
Perform Internal Quality Control

There are several types of LCS that can be used in a food testing laboratory to prove accuracy:

- **Food samples with a known true value of the component of interest (Standard Reference Materials (SRM) / Reference Materials)**

- **Fortified samples**: Fortifying a sample is to add known amounts of test components to the clean sample at the beginning of the test, and then prepare the fortified sample for analysis like all other samples.
Perform Internal Quality Control

LCS results are plotted on control charts to demonstrate that results are within the acceptable range and prove the method’s accuracy.

Figure 3: Example Chart Demonstrating Accuracy

Figure 4: Example Chart Demonstrating Precision
Confirm Results

The last quality check is to confirm results. This is accomplished by performing a second review of all data by a qualified analyst.

Looking Forward and being Prepared

Quality data does not come easily or instantaneously. It takes time and dedication to get the “correct” answer.
Looking Forward and being Prepared

ISO 17025 provides the road map for laboratories to develop a comprehensive and robust quality management system.

Having quality management systems in place throughout the lab can ensure that the lab has taken the time and is dedicated to getting the “correct” answer for all tests.
Food and Nutrition Department

About the Food Chemistry Lab

Coordenador
Maria Antónia Calhau

Estruturas de suporte
Gestão da Qualidade

Unidade de I&D
Helena Soares Costa
Identificar as necessidades de I&D em alimentação e nutrição
Desenvolver Programas e Projectos em alimentação e nutrição
Produzir e Divulgar conhecimento
Formação em I&D

Unidade de Observação e Vigilância
Luísa Oliveira
Riscos Biológicos
Riscos Químicos
Riscos Alimentares e Nutricionais

Unidade de Referência
Margarida Saraiva
Química
Mariana Santos
Microbiologia
Margarida Saraiva
Cristina Belo Correia
Materiais de Referência
Isabel Castanheira
Food and Nutrition Department

About the Food Chemistry Lab

The Food Chemistry Lab. develops activities in the areas

- Food and nutrition analysis,
- Food composition,
- Food chemical contaminants (mycotoxins, mercury, nitrates)
- Food Aditives,
- Packaging and Food Contact Material
- Food allergens (Determination of total allergen gluten/gliadin)

Our lab received the accreditation according to EN ISO/IEC 17025 on January 21, 2008, by the Portuguese Accreditation Institute (IPAC).
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About the Food Chemistry Lab
Food and Nutrition Department

About the Food Chemistry Lab

Nutritional Analysis - Why?

- All food products that are offered on a market have to comply with the respective regulation.
- Consumers are increasingly concerned about the quality and nutritional content of food.
- Nutritional information must be confirmed and validated by analytical techniques in order to be accurately included on labels and packaging.
New Food Information Regulation (FIR) –

• This was published by the European Union in 2011 and was designed to make food labelling easier to understand for consumers.

• The regulation combines rules on general food and nutrition labelling into a single EU regulation.

• Most requirements will not apply until 2014, with nutrition labelling becoming compulsory in 2016.
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New Food Information Regulation (FIR) – EU Regulation 1169-2011

The current rules specify the nutrients that can be included. The information has to be presented per 100g/ml, but could also be provided per portion.

Further information can be added to labels such as the amounts of polyunsaturates, monounsaturates, starch, cholesterol, vitamins or minerals.

<table>
<thead>
<tr>
<th>Format 1: ‘Big 4’</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy (kJ and kcal)</td>
</tr>
<tr>
<td>Protein (g)</td>
</tr>
<tr>
<td>Carbohydrate (g)</td>
</tr>
<tr>
<td>Fat (g)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Format 2: ‘Big 4 and Little 4’</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy (kJ and kcal)</td>
</tr>
<tr>
<td>Protein (g)</td>
</tr>
<tr>
<td>Carbohydrate (g)</td>
</tr>
<tr>
<td>of which sugars (g)</td>
</tr>
<tr>
<td>Fat (g)</td>
</tr>
<tr>
<td>of which saturates (g)</td>
</tr>
<tr>
<td>Fibre (g)</td>
</tr>
<tr>
<td>Sodium (g)</td>
</tr>
</tbody>
</table>
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New Food Information Regulation (FIR) – EU Regulation 1169-2011

<table>
<thead>
<tr>
<th>Current back of pack nutrition panel</th>
<th>New back of pack nutrition panel</th>
</tr>
</thead>
<tbody>
<tr>
<td>Energy 1500kJ/356kcal</td>
<td>Energy 1500kJ/356kcal</td>
</tr>
<tr>
<td>Protein 9.9g</td>
<td>Fat 7.4g</td>
</tr>
<tr>
<td>Carbohydrates 58.1g, of which sugars 16.8g</td>
<td>of which saturates 1.1g</td>
</tr>
<tr>
<td>Fat 7.4g, of which saturates 1.1g</td>
<td>Carbohydrates 58.1g, of which sugars 16.8g</td>
</tr>
<tr>
<td>Fibre 8.9g</td>
<td>Protein 9.9g</td>
</tr>
<tr>
<td>Sodium Below 0.1g</td>
<td>Salt Below 0.1g</td>
</tr>
</tbody>
</table>
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Our nutritional analysis include:

- **Lipids, Fats & Oils**: Fatty Acid Profiles
- **Proteins**: Amino Acid Profile, Proteins
- **Carbohydrates**: Total Dietary Fiber; Starch, Sugar and Sugar Profiles,
- **Vitamins**: Water-soluble and fat-soluble
- **Minerals**: Commonly labeled minerals; Individual Element Analyses
- **Basic Composition / Proximates**: Ash, Fat, Dietary Fiber, Moisture, Protein and Salt
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About the Food Chemistry Lab

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Moisture</td>
<td>Air Oven (AOAC 925.10)</td>
</tr>
<tr>
<td>Ash</td>
<td>Ash furnace (AOAC 923.03)</td>
</tr>
<tr>
<td>Total N-proteins</td>
<td>Kjeldahl (AOAC 920.87)</td>
</tr>
<tr>
<td>Total fat</td>
<td>Acid hydrolysis (AOAC 954.02)</td>
</tr>
<tr>
<td>Total dietary fibre</td>
<td>Enzymatic-Gravimetric (AOAC 985.29)</td>
</tr>
</tbody>
</table>
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**Amino Acid Profile**  
Waters Acquity UPLC

**Vitamins Analyses**  
Waters HPLC

**Individual Element Analyses**  
Inductively coupled plasma optical emission spectrometry (ICP-OES)
Food Composition

Information about food composition is necessary for:

- the assessment of diet quality,
- providing a useful tool for the field of public health nutrition.
- Food composition data (FCD) are important to food manufacturers, food retailers, governmental agencies, health professionals and consumers.
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Food chemical contaminants
(mycotoxins, mercury)

Total mercury (Hg) in a Direct Mercury Analyser DMA 80
(Milestone Inc.; CT, USA)
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Food additives must comply with specifications which should include information to adequately identify the food additive, including origin, and to describe the acceptable criteria of purity.
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Testing of Packaging and Food Contact Material

Food packaging and food packaging materials should be safe and should not transfer their components into food in unacceptable quantities.

(In European Union, the COMMISSION REGULATION (EU) No 10/2011 of 14 January 2011 on plastic materials and articles intended to come into contact with food)
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About the Food Chemistry Lab

Food allergens (Determination of total allergen gluten/gliadin)

• Nowadays, as the number of people with gluten intolerance increases, the food industry shows a greater concern to produce food that can be consumed safely by this group of people.

• In accordance with the European Regulation No. 1169/2011, which came into force on 13 December 2014, it is required to indicate on the label substances or products causing allergies or food intolerances.
Analysis for detection and quantification of gluten in food and raw materials:

Enzyme-linked Immunoassay (ELISA) R5 (RIDASCREEN® Gliadin)

Foodstuffs intended for particular nutritional uses have their own Regulation No. 41/2009, applicable since January 2012
Thanks for your attention

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