Universidade do Minho
Escola de Engenharia

Daniel Bruno Azevedo Pinto

Development, optimization and control of functional bakery products

February 2010

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Dissertation for PhD degree in
Chemical and Biological Engineering

Supervision
Dr. António A. Vicente
Dr. Inês Castro

February 2010

Author: Daniel Bruno Azevedo Pinto

e-mail: danielpinto@cpc.com.pt

Phone: +351 91 431 12 62

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Title of Thesis

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University of Minho, February, the 2nd 2010

à Inês e ao Francisco!

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"Numa qualquer manhã, um qualquer ser, vindo de qualquer pai, acorda e vai.
Vai. Como se cumprisse um dever."

António Gedeão

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"Estabelecer a harmonia entre todos os actos de uma empresa de maneira a facilitar o seu funcionamento e o seu sucesso …"

Henri Fayol (1841-1925)

Sumário

Os alimentos funcionais são alimentos que, para além das suas funções nutricionais básicas, têm um efeito fisiológico benéfico ou reduzem o risco de aparecimento de doenças crónicas. Recentemente, o sector agro-alimentar descobriu nos alimentos funcionais uma oportunidade de negócio uma vez que estes produtos têm um elevado valor acrescentado, sendo comercializados a preços muito superiores aos dos produtos similares sem características funcionais (FAO, 2003).

Existem, no mercado, diversos alimentos funcionais nos diferentes sectores agro-alimentares, nomeadamente lacticínios, bebidas, entre outros. Os produtos tradicionais de panificação e pastelaria são dos alimentos mais consumidos em Portugal, por todas as classes sociais e apresentam, *per se*, propriedades nutricionais importantes. No entanto, durante o processo de cozedura desencadeia-se um elevado número de reacções químicas complexas, enzimáticas e não enzimáticas, responsáveis pela coloração e aroma final do produto. Estas reacções, principalmente a formação de produtos resultantes da reacção de Maillard, para além de influenciarem as características organolépticas do produto, são responsáveis pela formação de compostos com propriedades fisiológicas de especial relevância. Alguns desses compostos, como a acrilamida e a 3(2H)-furanona, foram classificados como carcinogénicos. Por outro lado, foram já identificados alguns compostos com propriedades antioxidantes capazes de inibir o crescimento de células tumorais humanas (p.ex. proteínas proniladas – *pronilated proteins* – que parecem possuir importantes propriedades antioxidantes e establilizadoras de reacções químicas adversas).

Ainda na área de produtos de grande consumo, em Portugal e por todo o mundo, um dos exemplos mais relevantes é a batata frita. Face a este facto tornou-se pertinente o estudo e determinação de factores que afectam a formação de acrilamida ao longo do processamento de batatas fritas. Associado ao processamento de batatas fritas e intimamente ligado à segurança alimentar está o óleo de fritura utilizado uma vez que a degradação do mesmo leva à produção de compostos polares considerados carcinogénicos. A sua quantificação ao longo do processo de fritura e a limitação da utilização de óleos degradados é, portanto, um tema de elevada importância em matéria de segurança alimentar.

Os resultados obtidos permitiram confirmar que as batatas fritas e produtos de panificação e pastelaria, devido à sua composição e condições de processamento, têm potencial para a formação de acrilamida. No caso das batatas fritas, verificou-se que a concentração de acrilamida aumenta com o número de frituras efectuadas com o mesmo

óleo, até ao momento em que tende a manter-se constante; atinge-se uma taxa de máxima formação nas 7ª, 5ª e 2ª frituras, para 170, 180 e 190 ºC, respectivamente. Isto permite concluir que há um aumento significativo da velocidade de formação de acrilamida com a temperatura de fritura. A percentagem de compostos polares também aumenta ao longo das frituras e com o aumento de temperatura, sendo que a taxa máxima de formação ocorre nas primeiras frituras. Verificou-se uma relação entre a formação de acrilamida e compostos polares, no entanto esta relação tem diferentes comportamentos com as temperaturas de fritura. Relativamente aos compostos funcionais em estudo, os resultados revelam que existe uma tendência de diminuição da concentração de tocoferóis ao longo do processo de cozedura de produtos de panificação e pastelaria.

A garantia sistemática da segurança alimentar tem vindo a assumir, nos últimos anos, uma importância extrema. A inexistência de meios sistematizados de controlo pode potenciar a ocorrência de toxinfecções alimentares podendo traduzir-se num problema de saúde pública com enormes consequências económicas e sociais. A gestão da segurança alimentar, para além da sua obrigatoriedade, traduz-se em inúmeras vantagens competitivas para a empresa uma vez que poderá solucionar áreas críticas de um processo reduzindo as perdas resultantes de desperdícios e de devoluções de produtos. Por outro lado, exige um constante acompanhamento por técnicos qualificados para seguirem o processo e intervirem em situações "anormais" que se possam verificar. Esta disponibilização de recursos em pequenas indústrias alimentares, como é o caso das panificações, é normalmente bastante problemática quer por motivos económicos quer por razões culturais. Assim sendo, torna-se necessário facilitar os processos documentais que permitem, por exemplo, realizar mapas de produção, garantir a rastreabilidade dos produtos atribuindo automaticamente uma identificação de lotes, ou registar não-conformidades que se verifiquem. Desenvolveu-se um *software* no âmbito da rastreabilidade em indústrias alimentares e em particular na indústria de panificação e pastelaria (PaniGest®) alicerçado na regulamentação Europeia para a rastreabilidade de produtos alimentares. Este *software* está já em fase de comercialização e a resposta do mercado tem sido extremamente positiva.

Por fim, foi desenvolvido um *software* de gestão de laboratório com o objectivo de dar resposta aos requisitos das normas de acreditação de laboratórios de análises de produtos alimentares. O *software* desenvolvido, LabGest, está já completamente implementado no LabMaia e em outros clientes da empresa tendo sido já auditado em sede de acreditação dos laboratórios. A avaliação foi extremamente positiva tendo o *software* dando clara resposta aos requisitos da norma de acreditação de laboratórios NP EN ISO/IEC 17025.

Abstract

Functional foods are foods that, beyond their basic nutritional functions, have a physiological effect or reduce the risk of onset of chronic diseases.

Recently, the agri-food sector found in functional foods a business opportunity as these products have high added-value and can therefore be sold at much higher prices than similar products without functional characteristics (FAO, 2003).

In the actual global market there are many functional foods in different agro-food industries, including dairy, beverages, among others. The products of traditional baking and pastry are among the most consumed foods in Portugal, by all social classes and have, *per se*, important nutritional properties. However, during the production process, there are a high number of complex chemical reactions, enzymatic and non-enzymatic, responsible for color and flavor of the final product (Lindenmeier and Hofmann, 2004). These reactions, especially the Maillard reaction, are responsible for the formation of compounds with physiological properties of particular relevance in addition to their influence on the organoleptic characteristics of foods. Some of these compounds, such as acrylamide and 3 (2H)-furanones were classified as carcinogenic (Hiramoto *et al.*, 1996, 1998, Mottram *et al.*, 1998, Stadler *et al.*, 2002, 2003). On the other hand, some compounds with antioxidant properties were already identified, which are able to inhibit the growth of human tumor cells. Some other works (Lindenmeier and Hofmann, 2002, 2004) report the need to assess further information about the functional properties of pronilated proteins since they seem to have important antioxidant and stabilizing properties against adverse chemical reactions.

Also in the area of consumer products, in Portugal and throughout the World, one of most consumed products is French fries. Considering this, it was relevant to study and determine the factors affecting acrylamide formation during French fries processing. Associated with French fries processing and closely linked to food safety is the frying oil used. Oil degradation leads to the production of polar compounds, considered to be carcinogenic. Their quantification during the frying process is therefore important in order to a limit the use of degradated oils is a matter of high importance in food safety.

The results confirmed that French fries and bakery products, due to their composition and processing conditions, have the potential for acrylamide formation. In addition, in the case of French fries production, it was found that the concentration of acrylamide increases with the number of fryings made with the same oil until a certain maximum level. This level is attained earlier for higher frying temperatures, thus suggesting that for higher

temperatures the rate of formation of acrylamide increases significantly. The percentage of polar compounds also increased with the number of fryings and the increase of temperature; the maximum rate of formation occurs in the first frying. A relationship was found between acrylamide and polar compounds formation; this relationship presented different behaviors with frying temperature.

In terms of the functional compounds stability in bakery products, results show that there is a decrease of tocopherols concentration during baking.

In recent years, the systematic assurance of food safety has been taken to be of an extreme importance. The lack of systematic means of control can enhance the occurrence of food born illness, potentially leading to public health problems with significant economic and social consequences. The management of food safety, in addition to being mandatory, is reflected in numerous competitive advantages for the company since it can address critical areas of a process by reducing the losses by waste generation and products rejection. On the other hand, it requires constant monitoring by skilled technicians who follow the process and intervene in "abnormal" situations that may occur. This availability of resources in small food companies such as bakery industries is usually very problematic, both for economic and cultural reasons. Therefore, it is necessary to facilitate documents managing which, for example, includes production maps, ensuring the automatic traceability of products by assigning an identification of lots, or registration of noncompliances. A software for the implementation of traceability in the food industries and particularly in the bakery industry (PaniGest®) has been developed and is already being marketed. This software is based on European regulations for food traceability. The market feedback has been extremely positive.

Finally, laboratory management software has been developed in order to meet the requirements for the accreditation of laboratories. This software, LabGest, has now been fully implemented in LabMaia and other customers and it has already been audited for accreditation of laboratories; the obtained feedback is very positive and the software provides a clear response to the requirements of the accreditation of laboratories, including NP EN ISO/IEC 17025.

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Chapter 1.

GENERAL INTRODUCTION

1. Motivation and Description of Thesis Structure

Castro, Pinto & Costa, Lda. (CPC) was founded in 2000, and later on was part of the initial group of the first few Spin-off's from the University of Minho. The Company has been expanding and consolidating its activities since then.

Nowadays the company counts with 4 strategic business areas, namely:

- Trading and Production of OleoTest®;
- Consulting/Auditing;
- Research and Development;
- Training.

CPC soon began to introduce in their offer new products and new services, always supported by a policy of sustained growth.

From a technical staff of only 3 employees and no facilities or equipment, the company has now a staff of 15 technicians (including a PhD as well as graduates in various areas). Now located in Maia Industrial Zone (in the North of Portugal), in its own facilities developed from scratch, where in addition to administrative support, training rooms and product warehouses are available, it also has one of the most modern Laboratories in the country.

A major milestone of the company was the development and launch of OleoTest®, the rapid test for quality control in frying oils, which is now the most used in Portugal; it is already present also in Spain, France, Greece, Germany, Austria, United Kingdom and Panama. From the experience gathered with the development of this rapid test, the company realized it needed more scientific data and results to promote this product. This has a strong connection to one of the major objectives of this work: to study acrylamide and polar compounds formation in French fries during frying.

Another milestone was the creation of Consulting/Auditing business area. In particularly this business area has established strong connections with the baking industry, which represents nowadays around 70 % of this business area. Following this significant experience in implementing food safety systems in the baking industry, one of the main objectives of this PhD thesis was the identification, of harmful and functional compounds in bakery products, together with the determination of their formation and degradation rates.

Further, in the framework of its services business area, the company has always promoted the establishment of a relationship of trust and confidentiality with its clients. The

services are designed according to the specific needs of the client, therefore increasing the degree of effectiveness and efficiency.

LabMaia, the first satellite company created by Castro, Pinto & Costa, Lda., provides services in Research & Development and Analytical Control in food, water and environmental areas. This expertise together with the outstanding professionalism of the cadre of analysts and assistants, made of LabMaia the reference laboratory of the northern region of Portugal. In this area of business CPC also started a software development department which soon engaged in the production of software based in clients' needs and specifications. Taking into account the amount of customers and the experience in working with the baking industry, another objective was proposed for this thesis: development of a traceability software to help clients to implement food safety systems – PaniGest®.

The group Castro, Pinto & Costa supports and complies with the requirements of ISO 9001:2008 and is certified since September 2007 and its laboratory is accredited by NP EN ISO/IEC 17025:2005. Due to this accreditation and because of the experience achieved with PaniGest® software development, a new research and development area was created in the software domain: to develop software that would comply with NP EN ISO/IEC 17025 and help laboratories to reduce the amount of bureaucratic paper work in accreditation processes; this was the origin of LabGest®.

Currently, due to the increasing number of collaborative research, development and innovation developed together with other businesses, the company is in the process of certifying its Research, Development & Innovation System under NP 4457:2007.

The work developed under the framework of this PhD thesis has four main chapters:

- Study of acrylamide formation in French fries during frying and polar compounds formation in French fries;
- Identification, formation and degradation of harmful and functional compounds in bakery products;
- Development of traceability software to help clients implementing food safety systems (PaniGest®);
- Development of software complying with NP EN ISO/IEC 17025 and helping laboratories to reduce the amount of bureaucratic paper work in accreditation processes (LabGest®).

2. Introduction

The processing of foods is essential for its microbiological safety, while also contributing to increase the nutritional quality and to reduce the levels of possible toxic combinations. In many foods, the heat treatment is essential for the nutritional properties and sensory characteristics, especially the texture, flavor and color. Recent data indicate that heat treatment can induce the formation of health promoters, such as antioxidants and anti-microbial agents (Buchanan, 1993). Furthermore, processing can also lead to the formation of contaminants, like mutagenic heterocyclic amines (Zochling *et al.*, 2002) and acrylamide (Mottram *et al.*, 2002).

There are various reactions occurring in heated foods and that leads to the formation of new compounds involving fat, carbohydrate, protein and/or amino acids. The oxidative formation of reactive intermediates of fat and carbohydrate has a major role. The routes of possible reactions are related and highly dependent on composition of raw food matrices, the conditions of treatment and also the area of exposed surface of the food. Despite the change of the color of food is a common aspect to most thermal processes, the chemical nature of color formation reactions is different and dependent on the chemical constitution of the matrices (for example matrices rich in fat and carbohydrates have distinct mechanisms of colored compounds formation). The main source of compounds formed when heat treatment of food is undertaken, is the reaction between carbohydrates and amino groups, the Maillard reaction, which causes the development of browning in its advanced stage. The Maillard reaction is driven by low water activity, high temperature and prolonged heating time (Martins, 2003) leading to the formation of the so-called Maillard reaction products (MRPs), responsible for the volatile profile of foods. The Maillard reaction is associated with foods that were exposed to heat during industrial or domestic processing.

The Maillard reaction plays an important role in the improvement and/or appearance of food flavors. It is related to the aroma, taste and color, particularly in traditional processes such as roasting of coffee and cocoa, baking of bread and cakes, heat processing of cereals and meat (Fors, 1983; Friedman, 1996). Additionally, during the Maillard reaction, a wide range of products with high importance to the nutritional value of foods is formed (formation of functional compounds). However, it can also lead to a decrease in digestibility and possibly to the formation of toxic compounds and mutagens. Some studies (Lindenmeier and Hofmann, 2002, 2004) relate the need to obtain further information about the functional properties of pronilated proteins since they seem to have important

antioxidant and stabilizing properties against adverse chemical reactions. Moreover, vitamin E, ascorbic acid and β-carotene are the main elements of the diet that affect the body's antioxidant capacity (Steinberg *et al.*, 1984). More recently, the Maillard reaction was also associated with the formation of potentially carcinogenic compounds such as acrylamide (Mottram *et al.*, 2002 and Stadler *et al.*, 2002) and compounds containing the structure 3(2H)-furanones which have been associated to DNA mutagenic activity (Hiramoto *et al.*, 1996, 1998). Research performed by Baynes *et al.* (1989) clearly indicates that the Maillard reactions *in vivo* are partly responsible for diabetic complications and, in general, for the ageing process.

The reasoning presented above shows the essential need of performing optimiziation of thermal food processes aiming at an improved acceptance by the consumer (as they interfere with the food's organoleptic profile) and in order to comply with existing laws (safety profile), thus fostering the added-value of food products and minimizing the formation of harmful compounds.

3. Functional food - Feeding

Today, the feeding habits and dietary issues are often addressed in the media and among consumers. The daily diet provides energy and essential nutrients and is a complex mixture of various compounds that may be either protectors or offensive for the human body, which can interact among themselves or with other constituents of food (Skog and Alexander, 2006).

With industrialization, economic development and globalization of markets, changes occurred in people's lifestyle and consequently in the diet, preferring the convenience of the products at the expense of concern about the health impact of diet (Valente, 2001). Currently, a large variety and increasing amounts of food are available to consumers. In most cases such foods are industrially processed and personal preferences and taste are the main characteristics that influence consumers's choice (Birch, 1999).

The referred changes have an impact on public health and "nutritional status" of people, mainly in developed countries. Thus, the incidence of chronic diseases such as obesity, diabetes, cardiovascular disease, hypertension and some types of cancer increased significantly.

For these reasons, in the last decade interest has grown in scientific research in the areas of nutrition and analysis of epidemiological data aiming at clarifying the importance of diet in the prevention of mortality due to chronic diseases. Thus, the change in dietary habits may be the key to prevent the possible emergence of diseases such as cancer or cardiovascular diseases (FAO, 2003). This is why functional foods have invaded the market. Besides the original nutrition function they have, allegedly, a physiological effect and they either reduce the risk of onset of chronic diseases or may contribute to the treatment of diseases. The concept of functional food is, for some years, part of the vocabulary of nutrition (Torres, 2005). A food may be called functional "when the beneficial action of one or more functions is satisfactorily demonstrated in addition to adequate nutritional effects, relevant to a state of improved health and / or reduction of risk of disease" (Ashwell, 2002).

These foods, enriched in vitamins, minerals, acids, among others, are a new business opportunity for the agri-food sector because of their high added-value that results in a trade at prices much higher than similar products without functional characteristics. In the United States, for example, that market moves about 15 billion dollars per year.

4. Quality and Food Safety Management Systems

In recent years, the quality of manufacturing products has become one of the most important factors in national and international business and economic patterns (E.H. Drosinos *et al.*, 2006).

A significant number of standards have been developed over the years. The ISO family of standards represents an international consensus on management practices with the aim of ensuring that an organization can deliver products or services that meet the customers' quality requirements. The standards in the ISO 9000 series intend to be generic standards for quality management and quality assurance. The standards are to be applied to any type of organizations; independent to the size of the organizations or the kind of products manufactured or services provided, in private and public organizations, including government services and, of course, also in the food industry. In this last case, product related standard requirements forces food industry to implement HACCP programmes to control product quality and food safety. Also, food manufacturers are obliged by legislation to apply HACCP, while the other management systems are applied voluntarily in the food industry.

Published in September 2005, ISO 22000 *food safety management systems – requirements for any organization in the food chain*, is the newest arrival. The product of an ongoing collaboration between the International Organization for Standardization (ISO) and the food industry, it aims to be an international, auditable standard defining food safety management along the entire food chain – "to ensure that there are no weak links", as the ISO website explains. The intention is that ISO 22000 will sit alongside the other existing food safety schemes and complement them by bringing a common language and understanding of how food safety should be managed all along the food chain. ISO 22000:2005 is a HACCP-type standard based on ISO 9001:2000 especially developed to assure food safety. According to the ISO, ISO 22000 is intended to ensure that there are no weak links in food supply chains. The standard "can be applied to organizations ranging from feed producers, primary producers through food manufacturers, transport and storage operators and subcontractors to retail and food service outlets – together with inter-related organizations such as producers of equipment, packaging material, cleaning agents, additives and ingredients".

While ISO 22000's main difference from other standards lies in its scope and international applicability, it also differs from standards such as BRC and IFS, in that it does not offer a prescriptive list of requirements for good practice.

Instead, in acknowledgement of the fact that it would be impossible to name all requirements for all types of food businesses, it puts the onus on the food company to define the best practice that is most relevant to it.

The working group that developed the standard involved members from 14 different countries and representatives from a number of other organizations, including Codex Alimentarius and the Confederation of the Food and Drink Industry of the European Union (CIAA). Given the global nature of the food industry, it was important that ISO 22000 be developed with international and cross-industry consensus. According to several consulting companies, this approach represents the standard's greatest strength.

The assurance of safe production and the supply of safe food products appear to be the main aims of the food industry. These aims can be attained by adopting a systematic and organizational structure, controlling activities, processes, procedures and resources according to the standards which constitute the basis for the quality and hygiene systems, including HACCP, ISO 9000 and 14000 series (Early, 1995). The implementation of a Quality Management Systems, according to ISO 9000 series in the world of food products, is related to ensuring quality procedures for the food companies and reinforcing legislative requirements (Bolton, 1997).

Many food processing and packaging companies can start on the journey towards world-class quality by building a solid structure using together GMP, HACCP and the ISO 9000 family (Surak & Simpson, 1994).

The most common, in the food industries, is the simultaneous operation of a Quality Management System and a food safety system such as HACCP. The application of HACCP within an ISO 9001 Quality Management System can result in a food management system that is more efective than the application of either ISO 9001 or HACCP alone, leading to enhanced customer satisfaction and improving organizational efectiveness (Sparling, Lee, & Howard, 2001). In 2001, in order to facilitate the implementation of HACCP and ISO 9001 in food and drink organizations, ISO published ISO 15161:2001 which provided guidelines on the application of ISO 9001:2000 for the food and drink industry. ISO 15161:2001 includes sourcing, processing and packaging of food and drink products and it explains the possibility to link the commonalities and the communication between the two systems. It is important to consider that ISO 15161:2001 is not a standard for the implementation of

HACCP and cannot be a reference document at certification, but these guidelines are intended to provide a clear management system supporting HACCP controls for an efective food safety system, under the recognized framework of an ISO 9001 Quality Management System.

Efstratiadis and Arvanitoyannis (2000) mentioned that HACCP as a part of a quality system not only manages to provide safety to the products, but also assures a better and more efective implementation of the whole quality system. The new standard ISO 22000:2005 offers an alternative to food enterprises that do not implement ISO 9001 and want to have an efective food safety management system.

5. Laboratory Quality Management System

ISO/IEC 17025 is a quality standard for testing and calibration in laboratories. The current release was published in 2005. There are two main sections in ISO/IEC 17025: Management Requirements and Technical Requirements. Management Requirements are related to the operation and effectiveness of the quality management system within the laboratory and has similar requirements to ISO 9001 and ISO 22000. Technical Requirements address the competence of staff, testing methodology, equipment and quality and reporting of test and calibration results. The standard is the basis for accreditation from an accreditation body.

Implementing ISO/IEC 17025 has benefits for laboratories but there are also additional work and costs required.

Main benefits are:

- Having ISO/IEC 17025 accreditation status will get direct access to more contracts for testing and/or calibration. Some public and private organizations only establish contracts with accredited laboratories.
- Having ISO/IEC 17025 accreditation status will improve the reputation and image of the laboratory. This will also help to get more contracts from organizations that do not mandate accreditation but give preference to accredited laboratories in competitive situations.
- When correctly implemented, the quality system can help to continually improve the quality of data and effectiveness of the laboratory.
- ISO/IEC 17025 is the basis for most other quality systems related to laboratories, for example, Good Manufacturing Practices and Good Laboratory Practices.

Analytical testing laboratories seeking NP EN ISO/IEC 17025 will be impacted in a couple of areas. The main difference between formal accreditation and ‘just’ good analytical practices is the amount of documentation to be developed. There is no doubt that any good analytical laboratory uses qualified analysts, and for performing tests, checks the performance of equipment used for testing and validates analytical methods. However, many times the outcome of the tests is not fully documented. NP EN ISO/IEC 17025 accreditation requires formal documentation for about everything. It is similar to operating

in a regulated environment; 'what is not documented is a rumor', assessors consider it as 'not being done'.

The overall impact on analytical laboratories can be best illustrated on the entire sample/data workflow in a laboratory.

Figure 1.1 shows a typical workflow of samples and test data along with the NP EN ISO/IEC 17025 requirements on individual steps and the laboratory as a whole.

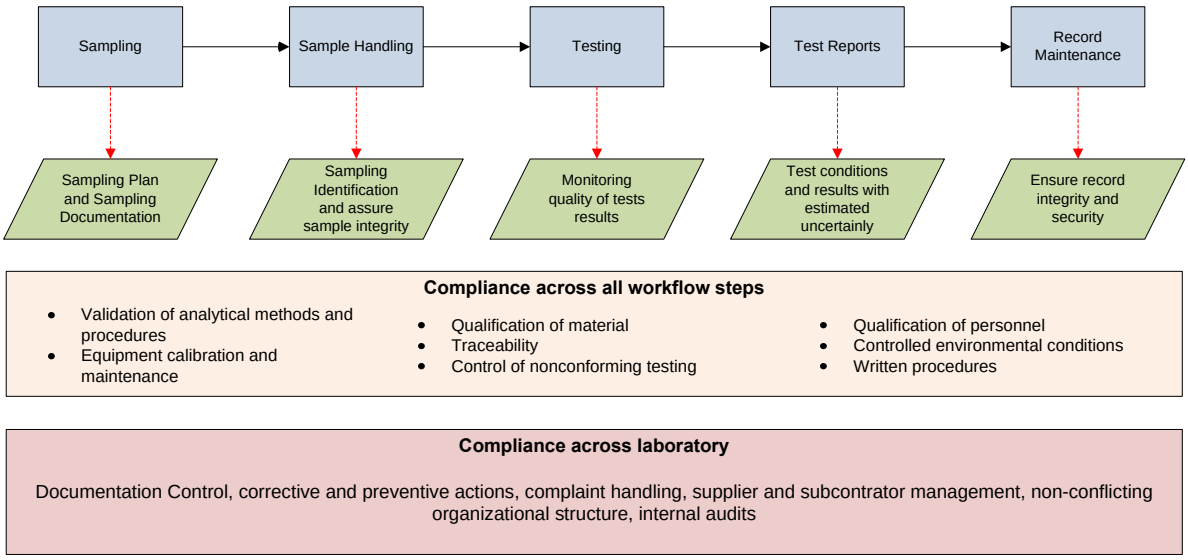

Figure 1.1: ISO/IEC 17025 Requirements for Analytical Laboratories

The ISO/IEC 17025 is an international standard applicable to all organizations performing tests and/or calibrations. It has management requirements about the organization of the lab, its quality system, document control, review of requests, tenders and contracts, subcontracting of tests and calibrations, purchasing services and supplies, service to the customer, complaints, control of nonconforming testing and/or calibration work, corrective actions, preventive actions, improvement, control of records, internal audits and management reviews. The technical requirements are about the personnel, accommodation and environmental conditions, test and calibration methods and method validation, equipment, measuring traceability, sampling, handling of test and calibration items, assurance of the quality of test and calibration results and reporting the results.

This means that having a quality system implemented according to this standard means to have the corresponding documentation, equipment, trained personnel, adequate accommodations, validated laboratory methods, etc., meaning that not only the processes are organized according to the requirements but also the technical competence of the lab must be strictly controlled to guarantee a good product and the customer's satisfaction. To

help laboratories to achieve all this requirements LabGest® was developed, which is a laboratory management software dealing with processes from sample receiving to printing of client analyses certificates.

6. Software Development: How Programming Works

On its own, a computer is not very smart: it is essentially a substantial number of small electronic switches that are either on or off. By setting different combinations of these switches, it is possible to make the computer do something, for example, display something on the screen or make a sound. This is the basic idea of programming: telling a computer what to do.

Understanding which combination of switches will make the computer do what is needed may be a difficult task – that is where programming languages come in.

In the presente work the software presented in Chapters 4 and 5 where developed using Visual Basic.

6.1. Visual Basic Programming language – basic concepts

People express themselves using a language that has many words. Computers use a simple language that consists of only 1s and 0s, with a 1 meaning "on" and a 0 meaning "off".

A programming language acts as a translator between the user and the computer. Rather than learning the computer's native language (known as *machine language*), it is possible to use a programming language to instruct the computer in a way that is easier to learn and understand.

A specialized program known as a *compiler* takes the instructions written in the programming language and converts them to machine language. This means that a Visual Basic programmer does not have to understand what the computer is doing or how it does it; to understand how the Visual Basic programming language works is all that is required.

The written and spoken language has a structure: for example, a book has chapters with paragraphs that contain sentences consisting of words. Programs written in Visual Basic also have a structure: *modules* are like chapters, *procedures* are like paragraphs, and *lines of code* are like sentences.

When speaking or writing, different categories of words are used, such as nouns or verbs. Each category is used according to a defined set of rules. In many ways, Visual Basic is much like the language used every day. Visual Basic also has rules that define how categories of words, known as *programming elements,* are used to write programs.

Programming elements in Visual Basic include *statements*, *declarations*, *methods*, *operators*, and *keywords*.

Written and spoken language also has rules, or *syntax*, that defines the order of words in a sentence. Visual Basic also has a syntax - at first it may look strange, but it is actually very simple. For example, to state "The maximum speed of my car is 55", we would write: Car.Speed.Maximum = 55.

The user interface is the part of a program that users see when they run the program. A user interface usually consists of a main window or form, and several controls, such as buttons, fields for entering text, and so forth. These types of Visual Basic programs are known as *Windows Forms* applications, and the user interface is created using Windows Forms controls.

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Chapter 2.

IDENTIFICATION OF HARMFUL COMPOUNDS IN PROCESSING FRENCH FRIES – EVALUATION OF THEIR FORMATION AND DEGRADATION

Abstract

During thermal processing, most foods are subjected to many reactions that, besides conferring colour and flavour to the final product, are linked to the formation of harmful chemical species, such as acrylamide and polar compounds.

The aim of the work was the determination of factors that affect acrylamide formation during French fries processing at 170, 180, 190 and 200 ºC, relating this parameter with frying oils quality through total polar compounds quantification.

With these results, it was possible to confirm that French fries, because of their composition and processing conditions, have the potential of acrylamide formation. Moreover, it was observed that acrylamide concentration increases with the number of frying batches performed with the same oil until it keeps stable in the subsequent frying batches; the highest formation rate occurs on the 7^{th}, 5^{th} and 2^{nd} frying cycle at 170, 180 and 190 ºC, respectively. This result leads to the conclusion that for higher frying temperatures, the formation of acrylamide rate increases significantly. The percentage of polar compounds also increased with the number of frying cycles and with temperature, and its highest formation rate was observed at the beginning of frying. It was observed that acrylamide and polar compounds formation are related however different relationships were found for different frying temperatures.

1. Harmful Compounds: Acrylamide

The Maillard reaction products are important for the sensory properties of processed food, such as color, aroma and taste. However some of the compounds that are formed may be beneficial or can have a toxic effect to humans. Among these compounds are heterocyclic amines and acrylamide.

According to Wilson *et al.* (2006), acrylamide formation takes place during the processing of food, either during frying, roasting or cooking, however it is not typically found in boiled foods or in those which have been subjected to microwaves. Acrylamide has become a worldwide alert when in 2002 it was discovered that this can be formed in foods during processing (Or *et al.*, 2007). Immediately after its discovery in thermally processed foods (Tarek *et al.*, 2002) rich in carbohydrates, the highest level of production was associated with the thermal degradation of free asparagine in the presence of sugars during the Maillard reaction (Mottram *et al.*, 2002 and Stadler *et al.*, 2002). In fact, several experimental studies have shown that the structure of carbon and nitrogen of the acrylamide amide group derived from the amino acid asparagine (Mottram. *et al.*, 2006). Identification of asparagine as a precursor of acrylamide was then supported by several observations (Amrein, 2005):

- The side chain of asparagine is similar to that of acrylamide, as shown in Figure 2.1; the decarboxylation or deamination of asparagine may lead to acrylamide;
- The highest concentrations of acrylamide are found in french fries and the potatoes are rich in asparagines;
- The free asparagine may also be found in other plants such as cereals, which may explain the frequent occurrence of acrylamide in thermally processed products (e.g. bakery and pastry).

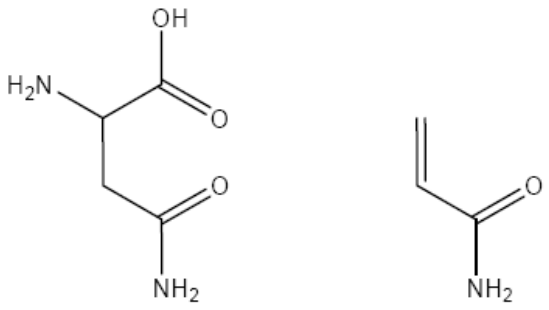

Figure 2.1: Structural forms of asparagine and acrylamide (Adapted from Amrein, 2005)

Asparagine needs carbohydrates to generate acrylamide. According to Pedreschi *et al.* (2005), from structural considerations alone asparagine can be converted thermally into acrylamide through decarboxylation and deamination reactions. Anyway, the main product resulting from the thermal decomposition of asparagine is maleimide, mainly due to a rapid reaction of intramolecular cyclization which prevents the formation of acrylamide. However, when in presence of reducing sugars, asparagine is capable of producing acrylamide beyond maleimide. The formation of acrylamide thus occurs during non-enzymatic browning due to Maillard reaction of reducing sugars with asparagine (Figure 2.2) at temperatures above 120 ºC (Gökmen *et al.*, 2005).

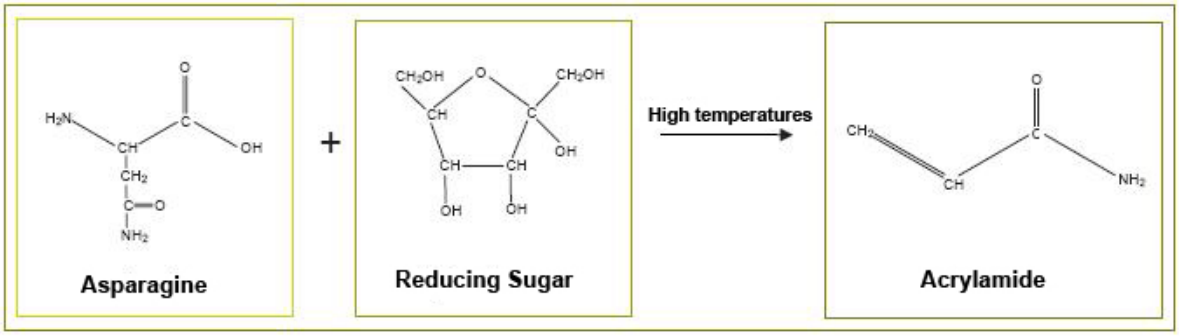

Figure 2.2: Mechanisms proposed for the formation of acrylamide (Adapted from Coughlin, 2003).

The discovery of acrylamide in foods processed with heat attracted attention and stimulated researchers to develop and validate rapid methods for its quantification (Zhang *et al.*, 2007). Reports from FAO/WHO (2002), recommend the use of methods to reduce or eliminate the formation of acrylamide. However it is uncertain whether changes in methods of processing may also influence the organoleptic properties of food and its nutritional composition. Anyway, according to Wilson *et al.* (2006), any effort to decrease the formation of this compound should also include "cooking at home" recommendations.

Zyzak *et al.* (2003) found that asparaginase, a selective enzyme, could be added before processing to reduce the production of acrylamide; according to Mottram *et al.* (2006), suppressing the reaction of Maillard could reduce acrylamide levels. However, since this reaction is responsible for food flavors and color generation, as already mentioned, its presence is essential to ensure the organoleptic quality expected by consumers.

As a secondary product of the Maillard reaction, the acrylamide content is influenced by the same factors that influence flavor and color formation. These factors include the reagents concentrations (food sugars and free amino acids levels), processing time and temperature, moisture content, pH and presence of additives, type and content of reducing

sugars, amino acids and frying oils (Or *et al.*, 2007). The reagents are influenced not only by the type of food but also by the cultivation techniques, sun conditions, harvest time and raw materials storage conditions. Several authors report that acrylamide in food may be reduced through changes in processing technologies (Or *et al.*, 2007).

Ahrné *et al.* (2007) study the effect of crust temperature and water content in the acrylamide formation during bread baking and found that the combination of both parameters (temperature and water content) has a significant effect on the acrylamide formation, and that higher temperatures result in higher acrylamide concentrations. However, it was also observed that higher temperature (around 260 ºC) and low moisture content, the acrylamide concentration decreased. In this case, however, product color and other sensory properties made it unacceptable for human consumption. So, the temperature has an important role in the acrylamide formation and elimination in food, and therefore the temperature, time and humidity are the keys to control the acrylamide formation in foods during heating (Gökmen and Palazoğlu, 2007).

The risk that acrylamide in food represents to health has been considered by various government agencies and national authorities (Gökmen *et al.*, 2005). Acrylamide is found mainly in foods rich in starch and products which are subject to high temperatures during processing, such as frying, baking or roasting (Amrein, 2005). Table 2.1 shows some examples of food and the range of concentrations of acrylamide in them. Changes of acrylamide concentrations in foods reflect the differences in materials, composition, process conditions and/or processing habits.

However, foods are not the only ones that contain acrylamide. To name a few, there is acrylamide in paper, paints, cosmetics and tobacco. This compound is commercially produced as an intermediate in the polyacrylamide production and synthesis. Acrylamide is also useful as a flocculant for clarification and purification of water for consumption, in wastewater treatment, soils stabilization, among others (Or *et al.* 2007). Therefore, tobacco, cosmetics and water may also contribute to the total amount of acrylamide that humans are exposed to (U.S. Department of Health and Human Services, 2005). Tobacco is a particularly important source, because acrylamide levels in the blood of smokers are about four times higher than in non-smokers (Schettgen *et al.*, 2002).

Moreover, there are several important considerations regarding the toxicology of acrylamide once it presents a variety of adverse effects in animals and humans. This compound is known as neurotoxic (causes peripheral neuropathy) in humans after exposure and is a toxic agent affecting the reproduction of in rodents. Acrylamide tests are positive in

a big number of genotoxicity tests, including chromosomal abnormalities, polyploidy, aneuploidy and other mitotic abnormalities in mammalian cells in the absence of metabolic activation (Or *et al.*, 2007).

Table 2.1: Acrylamide concentration in various foods (Adapted from Amrein, 2005)

Foodstuff	Acrylamide concentration (µg.kg^{-1})
Chips (package)	100-3770
Chips	50-680
Fried Meat	15-45
Fried rice biscuits	17-64
Bread	20-40
Roast Bread	25-2800
Gingiber bread	80-7800
Breakfast cereals	25-850
Coffee (beans)	40-480
Roasted nuts	10-2000
Popcorn	160-180
Grape juice	270

According to Gökmen *et al.* (2005), several studies have been performed to understand the health risks associated with acrylamide. However, there are no regulations regarding the limits of this compound in thermally processed foods (Gökmen *et al.*, 2005 and Ou *et al.*, 2007), and the association between acrylamide in the daily diet and the risk of cancer is debatable. Exhaustive research on acrylamide formation mechanism has been performed and, more recently, methods of testing, safety assessment/risk, metabolism in humans and strategies to reduce its content in foods have been extensively studied, (Or *et al.*, 2007).

The consumption of acrylamide in the daily diet of the population has been monitored in several countries. For adults, the estimated average consumption is approximately 0.3 to 0.6 $\mu g.kg_{body\ wheight}^{-1}.d^{-1}$. However, children and adolescents tend to consume higher amounts of acrylamide (Wilson *et al.*, 2006).

1.1. The Maillard Reaction

For as long as food has been cooked, the Maillard reaction has played an important role in improving the appearance and taste of foods. Its control has been a central and major challenge in food industry, since the Maillard reaction is related to aroma, taste and colour, particularly in traditional processes such as the roasting of coffee and cocoa beans, the baking of bread and cakes, the toasting of cereals and the cooking of meat. Moreover, during the Maillard reaction a wide range of reaction products is formed with significant importance for the nutritional value of foods. These advantages can be reduced by the decrease of digestibility and possible formation of toxic and mutagenic compounds, but may otherwhise be improved by the formation of antioxidant products. The chemistry underlying the Maillard reaction is very complex. It encompasses not one reaction pathway but a whole network of various reactions. The original comprehensive reaction scheme of Hodge (1953) has been developed and elaborated by food technologists ever since, so the understanding of the reaction is advancing steadily. Nevertheless the Maillard reaction is notoriously difficult to control. Various factors involved in food processing influence it and they can be considered as food processing variables. The kinetic approach tends to present a complementing view of this mechanism, because it considers the rate determining steps of the reaction. It is a powerful approach because rate-determining steps provide control points.

The Maillard reaction was first described by Louis-Camille Maillard in an article for the French Academy in 1912. However, only in 1953, Hodge presented the first outline of the original reaction understandable where it was shown that the chemistry that underpins the Maillard reaction is very complex (Hodge, 1953). It was not only a reaction but a number of different reactions. Since then, the original scheme has been continuously investigated, contributing to better understanding and attempt to control these reactions. The control of this series of reactions is very important in the food industry since the qualitative parameters of food are widely affected.

The Maillard reaction has been named after the French chemist Louis Maillard (1912) who first described it but it was only in 1953 that the first coherent scheme was put forward by Hodge (Figure 2.3). In essence, it states that in an early stage a reducing sugar, like glucose, condenses with a compound possessing a free amino group (of an amino acid or in proteins mainly the α-amino group of lysine, but also the α-amino groups of terminal amino acids) to give a condensation product N-substituted glycosilamine, which rearranges to form

the Amadori rearrangement product (ARP). The subsequent degradation of the Amadori product is dependent on the pH of the system.

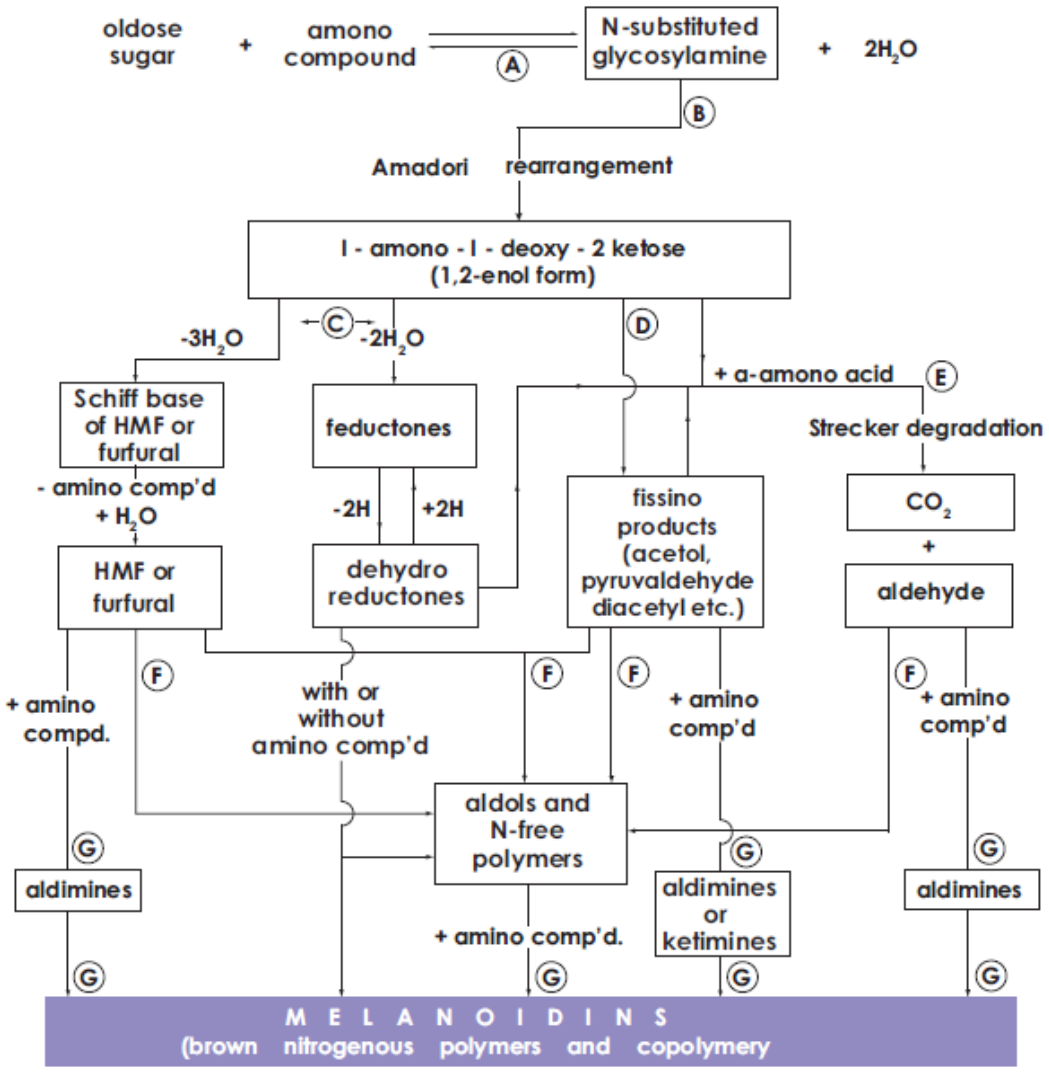

Figure 2.3: Maillard reaction scheme adapted from Hodge (1953)

The rate of the Maillard reaction and the nature of products formed are mainly determined by the conditions of reaction (Kaanane, 1989). These include chemical composition, pH and water activity, the presence of oxygen and metals, the combination of temperature/time during heating and the presence of reaction inhibitors (for example sulfur dioxide). However, the combined influence of time, temperature and pH, the speed of reaction, are very important in food matrices. These factors interact in determining the development of Maillard reaction during food processing, thus providing a high impact on the quality of processed foods.

The Maillard reaction strongly affects the food quality. Quality, from a technological point of view, depends on the control of chemical, physical and microbiological properties

and on their changes during processing and storage. To monitor these food changes it is important to study the reactions of interest in a quantitative manner.

1.1.1. Maillard reaction stages

At pH 7 or below, Maillard reaction undergoes mainly 1,2-enolisation with the formation of furfural (when pentoses are involved) or hydroxymethylfurfural (HMF) (when hexoses are involved). At pH > 7 the degradation of the Amadori compound is thought to involve mainly 2,3 enolisation, where reductones, such as 4-hydroxy-5-methyl-2,3-dihydrofuran-3-one (HMFone), and a variety of fission products, including acetol, pyruvaldehyde and diacetyl are formed. All these compounds are highly reactive and take part in further reactions. Carbonyl groups can condense with free amino groups, which results in the incorporation of nitrogen into the reaction products. Dicarbonyl compounds will react with amino acids with the formation of aldehydes and α-aminoketones. This reaction is known as the Strecker degradation. Subsequently, in an advanced stage, a range of reactions takes place, including cyclisations, dehydrations, retroaldolisations, rearrangements, isomerisations and further condensations, which ultimately, in a final stage, lead to the formation of brown nitrogenous polymers and copolymers, known as melanoidins.

The complexity and the variety of the Maillard reaction products has, throughout the years, raised the interest of scientists in different fields of research (Ericksson, 1981, Waller and Feather, 1983; Fujimaki, Namiki and Kato, 1986; Finot *et al.*, 1990; Labuza *et al.*, 1994; Ikan, 1996, O'Brien *et al.*, 1998). New important pathways, not accounted for by Hodge, have been established. McWeeny, Knowels and Hearne (1974) reported that the most important intermediates in colour formation are 3-deoxyosuloses and 3,4-dideoxyosulos-3-enes, which in the case of glucose is 3-deoxyhexosulose (DH) and 3,4-dideoxyhexosuloses-3-ene (DDH).

Later, Ghiron *et al.*, (1988) stated that 3-deoxy-2-hexosuloses, 1-deoxy-2,3-hexodiuloses and other α-dicarbonyl intermediates can undergo nucleophilic addition reactions with amino acids with subsequent decarboxylation to produce the so-called Strecker aldehyde (RHC=O). In 1990, Huber and Ledl isolated and characterised 1-deoxy- and 3-deoxyglucosones from heated Amadori products. Later, in agreement with the previous reports, Tressl, Nittka and Kersten (1995) using 13C-labeled sugars, have given a new perspective to the reaction mechanism. It involves different reaction pathways, in

which the key intermediates are the 1-, 3- and 4-deoxyhexosuloses. Moreover, a major influence of the pH is expressed. Along with enolization reactions, the Amadori product and its dicarbonyl derivatives can undergo concurrently retro-aldol reactions producing more reactive C2, C3, C4 and C5 sugar fragments, such as hydroxyacetone derivatives, glyceraldehyde and diketones. Retroaldol reactions become more important at higher pH values. Yaylayan and Huyghues-Despointes (1996) confirmed that observation.

ARP could generate acetic acid and pyruvaldehyde and other lower sugars in addition to free amino acids. As a result, high pH is suggested to be the main pathway to flavour formation. In addition to retroaldol reactions, three redox mechanisms have been identified, in which α-hydroxy carbonyls, α-dicarbonyls and formic acid are involved. Berg and Van Boekel (1994) reported formic acid as a main degradation reaction product for the Maillard reaction of lactose. Also, Van Boekel and Brands (1998) reported formic acid and acetic acid as two main degradation products for the Maillard reaction of glucose and fructose. In line with Tressl's perspective, Yaylayan (1997) proposed a classification for the Maillard reaction named the chemical pools. This approach is based on the observation that during the Maillard reaction the amino acids and the sugars also undergo independent degradation, in addition to the conventional degradation where Amadori product is formed. Berg (1993) concluded that isomerization and degradation reactions of the sugar are, from a quantitative point of view, more important than the Maillard reaction, for conditions such as those found e.g. in heated milk. Finally, the central importance of the Amadori product, formerly supposed to be the main intermediate of the reaction, has been questioned in both food (Molero-Vilchez and Wedzicha, 1997) and medical fields (Fu *et al.*, 1994). In spite of all the work that has been done, the mechanism of the Maillard reaction is still a controversial issue.

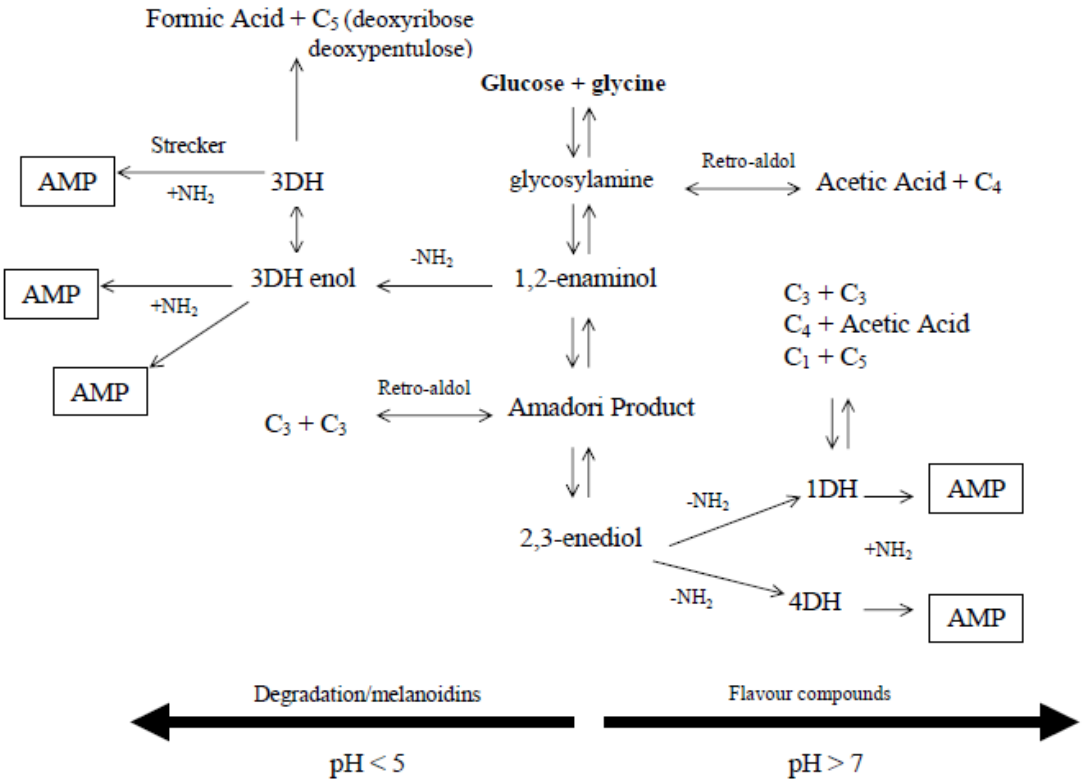

Figure 2.4: Scheme of glucose/glycine Maillard reaction adapted from Tressl *et al.*, (1995). AMP (Advanced Maillard Products); 1-DH (1-deoxy-2,3-diketose); 3-DH (3-deoxyaldoketose); 4-DH (4-deoxy-2,3-diketose)

1.1.2. Influence of Maillard reaction products on food properties

The origin of volatile compounds responsible for flavour is still relatively difficult to determine, due to the existence of multiple possibilities. The interest shown by food industry stems from a desire to produce and control the characteristic aromas and colours obtained on cooking, baking and roasting. Once the analytical technique of combined gas-chromatography-mass spectrometry was developed for the separation and identification of relatively volatile substances, the search for compounds with specific odours was greatly intensified. The results have been summarised in a series of review articles (Buckholz, 1988; Ho, 1996; Taylor and Mottram, 1996; Chuyen, 1998). Like in brown colour formation, it is clear that both quantity and quality depend on the precursors, thermal processing parameters, pH, and quantitative ratio of amino nitrogen to reducing sugar. For example, Lane and Nursten (1983) reported a thorough study on odours produced in Maillard reaction systems. They identified 12 amino acids, five to seven of which were thought to be related to bread, crusty biscuits, cake or toast aroma at each of the 4 temperatures studied, using single amino acid/glucose combinations at different

temperatures. Also, Fors (1983) published a literature review of the sensory properties of volatile Maillard products and related compounds. It includes qualitative aroma and flavor descriptions and sensory threshold values for various compounds, classified according to their chemical structure. More recently Teranishi, Wick and Hornstein (1999) have reviewed the thermal generation of Maillard aroma.

From the fact that we also eat with our eyes, the significance of Maillard browning in processed foods in terms of consumer acceptance is obvious. The degree of browning (usually measured via absorbance at 420 nm) is often used analytically to assess the extent to which the Maillard reaction has taken place in foods. Nevertheless, it has been stated that fluorescent compounds are formed prior to brown compounds (Baisier and Labuza, 1992). In the final stage of the reaction, coloured intermediates and other reactive precursors (enaminol products, low-molecular-weight sugar analogues, unsaturated carbonyl products) condense and polymerise to form brown polymers, under acceleration by an amine catalyst. Some of their known properties are to be brown, high molecular weight, furan ring-containing and nitrogen-containing polymers; they may contain carbonyl, carboxyl, amine, amide, pyrrole, indole, azomethine, ester, anhydride, ether, methyl and/or hydroxyl groups (Ledl and Schleider, 1990, Ames, Apriyantono and Arnoldi, 1993, Tressl *et al.*, 1998). Studies on melanoidins formation have been summarized in different review articles (Feather, 1985; Namiki, 1988; Friedman, 1996; Rizzi, 1997). The isolation and identification of coloured Maillard products has so far been achieved only with model systems, mostly for low molecular weight (<500 Da) products. Hashiba (1982) concluded that browning was directly proportional to the reducing power of the sugar and to the amounts of glycine consumed, by comparing different sugars with one single amino acid. However, more recently, Rizzi (1997) stated that many coloured products appear to be (retro)aldolization/dehydration products of sugars which may or may not be attached to proteins or other sources of amino nitrogen. Also Hofmann (1999) using dosage/activity relationship combined with chemical/instrumental techniques and visual/sensory measurments, identified carbohydrate degradation products as browning precursors (deoxyosones, glyoxal, methylglyoxal, hydroxy-2-propanone, 3-hydroxy-2-butanone and glycoaldehyde), which is in agreement with the scheme of Tressl *et al.* (1995) (Figure 2.4) and demonstrated that their activity in producing browning substances changes during thermal treatment. So far, only partial structures of melanoidins have been elucidated. The actual chemical species responsible for it and its origin remain largely undefined. Further investigations are needed.

One of the most obvious negative consequences of the Maillard reaction in food is the loss of nutritive value of proteins involved, with a loss of quality and a possible decrease of food safety. In various studies (Ericksson, 1981; Namiki, 1988; Friedman, 1996) this loss was related with a decrease of digestibility, destruction and/or biological inactivation of amino acids, including essential amino acids like lysine and tryptophan, inhibition of proteolytic and glycolitic enzymes, and interaction with metal ions. Also, protein molecules can be crosslinked by Maillard reaction products (Chuyen, Utsunomiya and Kato, 1991, Pellegrino *et al.*, 1999). Moreover, the loss of nutritive value has also been associated with the formation of mutagenic compounds. Nagao *et al.* (1979) identified mutagenic compounds in instant and caffeine-free coffee. They consisted of dicarbonyl compounds, methylglyoxal, diacetyl and glyoxal, from which the methylglyoxal presented the highest mutagenic activity; however, no quantitative correlation with carcinogenic properties was found. Also in both fried and grilled meat and fish, mutagenic compounds were identified, mainly stemming from heterocyclic amines (Arvidsson *et al.*, 1998). However, flavones and flavonoids have been reported as inhibitors of the heterocyclic amine-type mutagens (Lee *et al.*, 1992) being defined as desmutagens. In the Maillard reaction, desmutagenic effects have also been reported (Yen *et al.*, 1992; Lee *et al.*, 1994). The reaction mechanism seems to have a major influence in the mutagenicity of the reaction products. For instance, ketose sugars showed a higher mutagenic activity than the corresponding aldose sugars (Brands *et al.*, 2000). With respect to food safety, the involvement of the Maillard reaction in the formation and elimination of mutagens is a matter that still needs to be elucidated. Up to date no reports correlated these compounds with human cancer.

Finally, the Maillard reaction has been shown to produce antioxidative components as well. One of the first observations was reported by Griffith and Johnson (1957), who demonstrated that the addition of 5 % glucose to sugar cookies produced a marked browning in the cookies and resulted in a greater stability to oxidative rancidity. Since then, reaction products from various amino compounds and sugars were studied with regard to antioxidative properties, able to protect food against lipid oxidation (Chuyen *et al.*, 1998).

Accordingly, many food quality aspects of the Maillard reaction, some desirable, others undesirable, should be taken into consideration in a food processing operation. The task of the food technologist is then to optimize the balance between the favourable and unfavourable effects of the reaction in a given process. It could be a question of minimising the nutritional losses while obtaining an optimal flavour production when roasting cereals; of maximising the antioxidant production while minimising flavour and colour production

in milk drying, etc. Understanding the Maillard reaction is therefore an added value not only for the traditional processes of roasting, baking and cooking but also for the development of new technologies, like microwave and high pressure processing (Ames, 1998; Shahidi, Ho and Chuyen, 1998).

2. Harmful Compounds: Polar compounds

In addition to food processing, either by frying, baking, roasting, or grilling, it is also important to know the influence of operations in product quality. Frying is a common process of food preparation that uses fat or oils as a means of heat transfer. The main purpose of frying is the development of properties in food, such as color and flavor in the external surface of cooked products. These qualities are developed through the combination of compounds and Maillard reactions of the absorbed oil. The main factors that control the change of color and flavor in certain foods are:

- The type of frying oil used;
- The age and thermal history of oils;
- The interfacial tension between oil and products;
- Temperature and time of frying;
- Size, moisture and food surface characteristics;
- Post-frying treatment.

Each of these factors, with any pre-treatment such as bleaching or partial drying, also influence the amount of oil which impregnates the food and in many fried foods, the oil may be about 45 % of the total mass of the product (Fellows, 2000).

The frying process includes three methods:

- By immersion (or intensive), where foods are cooked submerged in hot oil or fat;
- Frying surface, which involves cooking the food in e.g. a frying pan with a layer of oil or fat;
- Roast food in the oven.

Of these three methods, the immersion frying is by far the one which has been the most subjected to technological studies, since it is widely used in industrial production and requires constant monitoring. By immersion frying, fats or oils are exposed for long periods to a stress caused by heating and modification of food components (Bucarelli and Quaglia, 2001). This method of preparation is used to confer particular organoleptic characteristics (color, flavour, texture) to various animal or vegetable food products. Frying also works as a food preservation method, since it destroys micro-organisms, promotes the inactivation of enzymes and reduces the water activity on the food surface (Rossell, 2001; Quaglia and Bucarelli, 2001 and Fox, 2001). The sterilization of the surface is sufficient to ensure a good

shelf life, especially for frying products with subsequent freezing (Rossell, 2001). During immersion frying, food and oil are subject to physical and chemical changes, including the decomposition with a potential toxicological risk.

It is also important to distinguish between industrial frying of food for retail sale and frying in restaurants. In the latter case, the food is consumed immediately after frying and the cooker can give it the taste (s)he prefers, while when frying in the retail industry the food is cooled after frying, sometimes frozen and packaged, displayed in retail and stored in the consumer's kitchens and then re-heated and consumed. Any instability in the oil has time to develop taste for the period between the initial frying and consumption, emphasizing the need for high quality oils for industrial frying (Fellows, 2000). The time required to complete a fried food depends on:

- Type of food;
- Oil temperature
- Frying method (surface frying or intensive);
- Food thickness;
- Changes required in food quality.

The frying temperature is also determined by products' requirements. Foods which require a crust and a relatively high interior humidity are produced at higher frying temperatures. The rapid formation of crust is beneficial, since it insulates the moisture in the food and restricts the transfer of heat to the interior. Dryer foods are processed through frying over lower temperatures in order to cause deep and complete evaporation in food before the crust is formed. The texture of fryed products is produced by protein, fat and carbohydrate polymer changes. Changing the protein quality occurs as a result of the Maillard reaction with amino acids on the food surface. The loss of carbohydrates and minerals, not usually mentioned, should be preferably small (Fellows, 2000).

High temperatures cause a rapid deterioration of the oil and the formation of free fatty acids, which alter the oil viscosity, flavour and color and promote the formation of foam, increasing the frequency with which the oil must be replaced, with the consequent increase in costs.

The most important aspect of industrial frying is the oil, and the factors affecting the frying oil quality are:

- Its properties and composition;
- Transport, packaging and storage;

- The nature of the fried foods and their interaction with the frying oil;
- Equipment and frying process;
- The evaluantion of frying oil quality during its use (Rossell, 2001).

These factors affect the control of chemical changes that occur in connection with toxicity risks.

The main requirement on the quality of foods is their safety, which may be provided by analysis of risks and prevention through complete process monitoring, thus advising the adoption of HACCP systems (Bucarelli and Quaglia, 2001). Frying is a critical control point that is frequently the last step of the process or even the only treatment. Food safety requires that the preparation of food is sufficient to remove any pathogenic bacteria that may be present. This may be rapidly achieved through frying as it involves high temperatures. However, as the heat penetrates from the outside thus making the center the most cold part of the food, frying works as a surface sterilization procedure and this reflects the importance of measuring the temperature at the center of food as a precaution to ensure food safety and quality (Fox, 2001).

During food frying, the processes of oxidative and hydrolytic degradation gradually change the quality of the oil (Mestdagh *et al.*, 2007). For these reasons, understanding the degradation of frying oil is essential to produce high quality fried foods. In fact, the prolonged heating of oils to high temperatures used in frying causes a thermal and oxidative modification. The oxidation of oils is caused by the release of moisture from the food and also by the input of atmospheric oxygen in the oil, forming a wide range of volatile carbonyl, hydroxy-, keto- and epoxy- acids. This causes the appearance of taste and undesirable browning of oils.

Oil can be tested through the analysis of some parameters such as refractive index, color, total content of polar compounds, free fatty acids, rates of iodine and peroxide. Once tested, it is possible to establish a relationship between the oils' chemistry and the quality of food produced with them.

There are concerns about the amount of chemicals in terms of contamination, and some compounds of great concern are the polar compounds (Fox, 2001). The presence of these compounds can be used to measure the cumulative degradation of frying oil. Manral *et al.* (2007) and Bosko and Elmadfa (1999) consider that the measurement of the polar material is simply the most important and more reliable test to investigate the frying oil degradation. One of the weaknesses of this method is that some oil types, when perfectly fresh, have inherently high levels of polar compounds. These compounds are excellent messengers of

product quality in various operations and usually an increase in the fraction of polar compounds decreases the product quality (Bennett, 2001).

Stier (2001) states that the regulatory agencies in Spain, Portugal, France, Germany, Belgium, Switzerland, Italy and the Netherlands have established limits for the polar material in frying oil (Bennett, 2001). Each country has a law for the limit of acceptability of the quality of frying oils, and when the oil should be discarded (Table 2.2). Several countries specify a maximum for the frying temperature; however France is an exception for certain oils particularly stable, in which 200 °C are permitted. In Portugal, the maximum temperature of frying and the maximum percentage of polar compounds are admitted to be 180 ºC and 25 %, respectively (Fox, 2001).

Table 2.2: Regulations of the life-span duration of frying oils (adapted from Fox, 2001)

	Country															
	USA	Austtria	Belgium	Denmark	Finalnd	France	Germany	Greece	Ireland	Italy	Luxenburg	Netherlands	Portugal	Spain	Sweden	UK
Maximum frying temperature (ºC)	n.a.	180	180	n.a.	n.a.	180	n.a.	n.a.	n.a.	180	n.a.	n.a.	180	n.a.	n.a.	n.a.
Smoke point (min. %)	n.a.	170	n.a.	n.a.	n.a.	n.a.	170	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Free fatty acid (max. %)	n.a.	n.a.	2.5	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Acidity (max.)	n.a.	2.5	n.a.	n.a.	n.a.	n.a.	2	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Polar Compound (max. %)	n.a.	27	n.a.	n.a.	n.a.	25	24	n.a.	n.a.	25	n.a.	n.a.	25	n.a.	n.a.	n.a.
oxidized fatty acids (max. %)	n.a.	1	n.a.	n.a.	n.a.	n.a.	0.7	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
dimers and polymers (max. %)	n.a.	n.a.	25	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	16	n.a.	n.a.	n.a.	n.a.
Viscosity (max. mPa.s)	n.a.	n.a.	37	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.

n.a. – non-available

The formation rate of decomposition products depends on the type of food being fried, on the frying equipment and on the operation conditions (Manral *et al.*, 2007). The analysis performed by Manral *et al.* (2007) of the oil quality, in terms of its physical and chemical

parameters, during fish frying revealed that the quality of the oil begins to deteriorate with time. This study showed that the limit value of polar compounds (about 25 %) within the index of quality is reached after 10 hours of frying.

The polymerization of oil decomposition products, in the absence of oxygen, produces cyclic compounds and polymers of high molecular weight, which increase the viscosity of the oil. This reduces the area of heat transfer during frying and increases the amount of oil which impregnates the food (Fellows, 2000). Many of these compounds are polar and reduce water evaporation, with foaming. However, these polar compounds have beneficial effects on frying: they impart flavor to food, they contribute to the golden-brown color and they optimize the retention of fat.

The oil used in short periods, compared with the fresh oil, improves frying due to polar compounds that promote better contact between the oil, the water present in the food surface and the steam which is generated. This results in a better and uniform heat transfer and enhances the acquisition of flavor (Fellows, 2000).

According to Romero *et al.* (2006), the degradation and consequently the shelf life of frying oils are highly dependent on the type and quality of the frying oil, as well as on the type/composition of the fried food (Varela and Ruiz-Roso, 2001).

In the process of frying, oil is typically reused several times for frying portions of the same or different products. The issue of oil reuse is interesting and relates to the differences found in oils' shelf-life. This shelf-life is attained when the oil gets dark, thicker and some deposits emerge, which is sometimes accompanied by the production of an acid flavour.

3. Fried potatoes

The activity of *Preparation and storage of potatoes* (Portuguese Economic Activity Code – CAE – 15319) contributed in 2001 with 19 % of the total sales of CAE 153 (*Industrial preservation of fruits and vegetables*). From 1999 to 2001, the output of this activity had an increase of 12 %. However, the value of sales did not follow this increase and a decline of 11 % has been observed in 2001 compared to 2000. Fries, the main product of this activity, were the main responsible for this negative development (INE, 2003). More recently (2008), one of the main processors of this type of product in Portugal invested 4 M€ in its production plants, aiming at increasing the production capacity for chips. This plant alone produces 25 000 tons of chips per year, of which 50 % are consumed in Portugal and the remnant in Spain.

Potato (*Solanum tuberosum*) is a major agricultural crop and is consumed daily by millions of people in different ways.

The process of fried potatoes (chips) preparation is dynamic and includes several interdependent steps, to convert the potatoes into chips that meet consumer's expectations. Each step of the process is designed to perform a specific function, producing the desired characteristics in the final product or allowing a subsequent point of control to achieve its functions (Bennett, 2001).

The main raw materials used in processing chips are potatoes, frying oil, salt and flavorings. The storage conditions are critical for the quality of the chips produced and to control processing costs. The storage of salt and flavorings only requires a dry area with temperatures between 10 and 30 °C and free of contamination, while the storage of chips requires special tools and controls (Bennett, 2001).

In the industrial process of chips, potatoes are washed, peeled, sorted and cut into slices or sticks (Pedreschi *et al.*, 2007; Mai Tran, 2007). After that, they are fried in continuous hot oil deep fryers (at 170 °C), where they remain until their moisture content is reduced to about 2 % of the total weight. Finally, potatoes are salted, cooled and packaged (Pedreschi *et al.*, 2007).

Immersion frying has been defined as the immersion of food in heated edible fat at a temperature above the boiling point of water and can, therefore, be considered a dehydration process. These conditions are associated with high rates of heat transfer, fast processing, acquisition of brown colour, and development of texture, flavour and taste (Pedreschi *et al.*, 2005; Pedreschi *et al.*, 2007). The development of chips crust during frying influences the

heat and mass transfer phenomena (Krokida *et al.*, 2000), which are responsible for the development of desired chips sensory properties. As in the frying process of other foods, chips color is caused by the Maillard reaction that depends on the content of reducing sugars and amino acids or proteins at the surface of the food, together with frying temperature and time (Pedreschi *et al.*, 2005).

According to Bennett (2001), the main aspects that consumers evaluate when consuming chips are: appearance, texture, flavour and taste. It is known that the texture of the product depends on the potatoes quality and the technological parameters used during production (Pedreschi *et al.*, 2005).

Frying is also seen as a method that creates a flavor and unique texture, which improves the taste of food (Pedreschi *et al.*, 2007; Pedreschi *et al.*, 2005). The perceptions of consumers should be expressed in quantifiable characteristics that may assist in the evaluation of raw materials, the determination of process parameters, process monitoring and final product evaluation (Bennett, 2001).

3.1. Acrylamide formation in fried potatoes

Acrylamide is, as already mentioned, a suspected human carcinogen that has caused great concern because it forms during processing, either by frying or baking, of foods rich in carbohydrates such as potatoes (Grob *et al.*, 2003 and Mestdagh *et al.*, 2007).

Chips and other potato products contribute significantly to the acrylamide exposure (see Table 2.1) on the one hand due to high concentrations of this compound found in this food and on the other hand due to its high levels of consumption (Amrein, 2005). Acrylamide formation is related to the Maillard reaction, as already mentioned. Asparagine, a free amino acid, and reducing sugars are the main precursors (Mestdagh *et al.*, 2007). The potential of acrylamide formation is also related to the sugar content as glucose and fructose (Pedreschi *et al.*, 2004).

Frying is a process of heat transfer more efficient than roasting or baking due to high rates of heat transfer associated with hot frying oil. This feature of the frying process promotes the rapid formation of a crust on the food surface. However, since the predominant heat transfer mechanism in frying is conduction, the region near the food surface reaches much higher temperatures, promoting the formation of acrylamide (Figure 2.5). Indeed, this is why the acrylamide formation is mostly a superficial phenomenon.

During frying, the oil is used to increase the internal energy of the potatoes, until the surface reaches a temperature below the boiling temperature of water present in potatoes (103 - 104 ºC). After this point, the water evaporation releases large amounts of energy. When the oil temperature is sufficiently high (≥ 170 °C), the received energy is sufficient for the water evaporation and the temperature increase mutually, favoring the acrylamide formation (Palazoğlu and Gökmen, 2007).

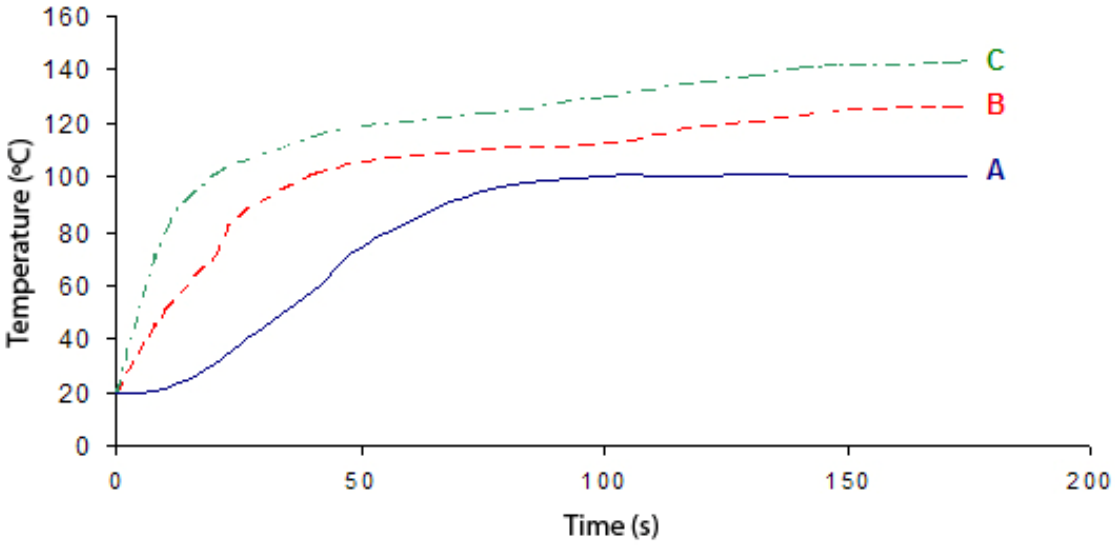

Figure 2.5: Simulation of temperature profiles at three locations over a slice of potato during frying to 170 ºC, (A) Inside the food, (B) Near the surface, (C) surface of the food (Adapted from Gökmen and Palazoğlu, 2007).

Acrylamide is produced by the thermal degradation of free asparagine with a reducing sugar. The following factors promote the acrylamide formation (Grob *et al.*, 2003):

1. A high asparagine content, which can be found in potatoes (about 100 times higher than in wheat flour);
2. A high reducing sugars content (fructose is about twice as active as the glucose)
3. Humidity below 30 %. The water activity affects the Maillard reaction, the reaction is faster at average water activity. However, the amount of water that interferes with the brown color acquisition depends on temperature (Ames, 1990). So it can be assumed that the amount of water affects acrylamide formation (Wicklund *et al.*, 2006);
4. Temperature above 100 ºC. High temperatures accelerate acrylamide formation.
5. A relatively inert matrix that prevents their elimination (Amrein, 2005), as starch.

Once the potatoes are rich in asparagine and products containing reducing sugars such as glucose and fructose, it is known that the Maillard reaction is the main responsible for acrylamide formation in fried potatoes (Pedreschi *et al.*, 2004).

The acrylamide concentration variation in foods is associated with different levels of acrylamide precursors in raw materials (Mestdagh *et al.*, 2007). In addition to food composition, other factors involved in acrylamide formation are the processing conditions (Pedreschi *et al.*, 2004).

3.1.1. Raw materials influence

In the work of Mestdagh *et al.* (2007) the pre-treatment of several raw materials was studied in order to minimize acrylamide formation. These techniques include the extraction of acrylamide precursors through immersion and bleaching in hot water or in acid solution. According to Fiselier *et al.* (2006), acrylamide concentration in French fries can be reduced from 2 to 3 times by reducing sugars and controlling the frying temperature. Ensuring a low sugar content in the potatoes means starting with a proper cultivation, followed by storage under conditions that prevent the release of reducing sugars and by a bleaching procedure for extraction and prevention of the presence of reducing sugars on potatoes' surface (Amrein, 2005). The main conditions that prevent the presence of reducing sugars on the surface of potatoes are:

- **Variety:** The amount of reducing sugars and free amino acids may differ significantly with the variety of potatoes. Several studies showed that concentrations of sugars and free asparagine may vary between varieties (Amrein *et al.*, 2004 and Amrein, 2005), which explains why are there variations in acrylamide concentrations in French fries from different origins (Amrein, 2005, Brunton *et al.*, 2007). The content of asparagine in potatoes depends, besides the variety, from other factors such as location, fertilization, storage and processing (Pedreschi *et al.*, 2004). According to Williams (2005), it is known that asparagine is more abundant in potatoes than reducing sugars. However, the concentrations of glucose and fructose change between varieties more than asparagine concentration. Mestdagh *et al.* (2007) state that the amount of reducing sugars is the main parameter to consider, since it is the limiting factor for the occurrence of Maillard reactions and browning. Indeed, when potatoes have a high concentration of reducing sugars during processing,

the amount of acrylamide formed is also high, and vice-versa (Fiselier *et al.*, 2006);

- **Fertilizer:** A fertilizer with nitrogen can influence potatoes composition. However, the influence of fertilization on acrylamide formation is still subject of debate;
- **Climate:** Temperature, sun incidence, and water availability affect the plants' metabolism and therefore their composition;
- **Storage:** Storage conditions are very important to potatoes' quality and has consequences in the processing results. Storage at temperatures below 8 °C is related to the accumulation of sugars. This can dramatically increase acrylamide formation. Furthermore, storage at temperatures near 15 °C can eliminate many, but not all, accumulated sugars. The amount of free amino acids may also vary during storage. According to Wicklund *et al.* (2006), color is the main indicator of chips' quality and can be improved by selecting the appropriate processing conditions.

3.1.2. Process influence

There are several studies seeking strategies to minimize acrylamide formation while maintaining the desired quality for the consumer. An integrated approach that combines the knowledge of how to reduce the acrylamide concentration with the corresponding specific aspects of quality such as color, texture and flavor is therefore required (Mestdagh *et al.*, 2007).

According to Amrein (2005), pre-treatments (bleaching and addition of additives such as citric acid and others) and the process itself (frying time and temperature; type of frying oil) have to be considered when evaluating the acrylamide formation potential. Strategies to reduce the levels of acrylamide should include optimization and a strict control of the frying process, citric acid addition and application of asparaginase to reduce asparagine content (Amrein *et al.*, 2004).

Bleaching or immersing potatoes in water allows to extract precursors (sugars and amino acids) of its surface before the chips preparation and Bleaching in hot water seems to be more effective in glucose and asparagine extraction than immersion in cold water (Amrein, 2005). Indeed, Grob *et al.* (2003) reported that hot water permeabilizes cell membranes, facilitating the extraction of such compounds.

Another development is the addition of acids (e.g. citric acid) to immersion solutions. At low pH values (for example pH 4 or pH 5), acrylamide formation is less pronounced (Amrein, 2005).

According Mestdagh *et al.* (2007), several substances such as organic acids, NaCl and Ca^{2+}, not only reduce the final acrylamide concentration but also decrease oil absorption during frying. This correlates perfectly with consumers' wish for healthy and low-fat products, aiming at avoiding obesity and heart disease. However, the use of additives may also stimulate other reactions, which can negatively influence the products' safety (Mestdagh *et al.*, 2007). Addition of salts such as NaCl and $CaCl_2$ before frying can increase the subsequent oil degradation rate of oil (Mehta and Swinburn, 2001 and Padilla, 2005). Results show that the compounds used and their concentrations must be carefully chosen so as not to create unwanted flavors or colors.

From a chemical point of view, a high temperature and/or prolonged heating are related to acrylamide formation and elimination. Grob *et al.* (2003) showed that frying temperature is the key factor in acrylamide formation and that this compound is mostly formed in the last two minutes of frying. The reduction of frying temperature from 180 °C to 170 °C allows significantly lower levels of acrylamide to be attained during chips production and allowed determining less critical final time and frying temperature as shown in Figure 2.6.

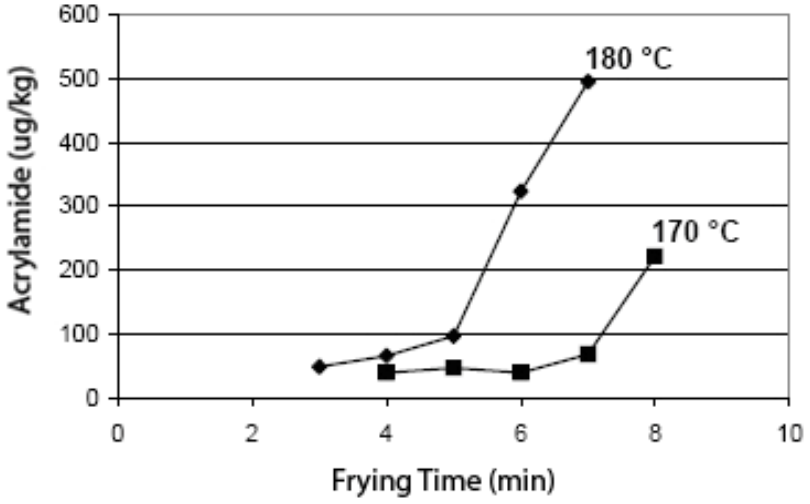

Figure 2.6: Formation of acrylamide in French fries prepared in oil with different initial temperatures (Adapted from Grob *et al.*, 2003).

According to Wicklund *et al.* (2006), the highest acrylamide concentration was not detected in French fries produced from raw materials with higher amount of asparagine. Instead, the two varieties with higher content of reducing sugars (before and after

bleaching), showed a greater potential for acrylamide formation when fried at 185 ± 1 °C. This indicates that reducing sugars concentration is more important than asparagine concentration when sudying acrylamide formation in fried potatoes.

In practice, if the raw materials are not appropriate, all efforts made to limit this compound formation in products with heat treatment will be unsuccessful. Both the selection of appropriate raw materials and the modification of techniques aiming at reducing acrylamide contents will be very important aspects in future research (Wicklund *et al.*, 2006).

3.2. Polar compounds formation during frying

According to Tiffany (2007), stating that oils and fats are dangerous to human health is an oversimplification. Fats provide energy, calories and essential fatty acids that can only be obtained through food, because the human body does not produce them. In fact, fats are not only essential to growth, health and skin absorption of nutrients, but are considered critical for the food industry as a way of frying.

Frying is a frequently used practice in the preparation and manufacturing of food. Today, fried foods consumption has increased with consumers' preference for modern, convenient foods, e.g. snacks and take-away.

The most poly-unsaturated oils are used in frying, being cottonseed and soybean oils the most common. Their poly-unsaturated structure makes them susceptible to degradation due to oxidation, hydrolysis or polymerization when exposed to air, water or heat (Bennett, 2001).

Oil degradation directly affects both quality and safety of fried foods. Although many of the changes are already known, it is difficult to predict the degradation rate due to the large number of factors involved. These factors are not only related to the environment, but also to the frying process (temperature, duration of heating, heating mode, frequency of oil replacement) or to the food itself (Bosko, 2003).

Conventional methods for determining the point at which the oils and fats discharge should take place are based on physical changes, subjectively assessed by the operator. This procedure often results in over-exploitation of the oil, leading to very high concentrations of polar compounds and polymers. In order to protect consumers from contaminated food, regulations or guidelines were established in several countries. Limits for polar compounds concentrations of ca. 25 % were established in official documents (see Table 2.2).

Furthermore, limits for polymer and free fatty acids concentrations have also been considered by several countries (Benedito *et al.*, 2007).

3.3. Polar compounds formation versus acrylamide formation during frying

Acrylamide forms when foods rich in carbohydrates are baked, roasted, toasted or grilled. Fried foods, such French fries, are likely to form acrylamide since they contain relatively large quantities of acrylamide precursors such as amino acids and reducing sugars. However, there may be other ways of acrylamide additional formation, starting with fat. It was suggested that triglycerols are partially hydrolyzed during frying, followed by dehydration of glycerol to acrolein. These three compounds can oxidize acrylic acid, which finally reacts with ammonia to form acrylamide (Mestdagh *et al.*, 2007).

Acrolein can be formed through pyrolysis of triglycerols without glycerol acting as an intermediate. In addition, monoglycerides break down into acrolein and free fatty acids at temperatures exceeding 150 ºC via an elimination reaction. This means that the products of hydrolysis of oils can act as precursors of acrylamide. The importance of this pathway for acrylamide formation in foods has not been completely clarified yet.

Apart from the fact that oil hydrolysis could produce glycerol as a precursor to acrylamide, mono and diglicerols generated may also alter the food's surface tension between water and the apolar oil. As a result, there will be an increase of the heat transfer coefficient and therefore more heat can be transferred from oil to food in a given period of time. According to Mestdagh *et al.* (2007), oxidation of oil may be an important issue affecting acrylamide formation due to its influence on the surface tension between the food and oil. Furthermore, polar compounds formed from oils oxidation can retain water in the frying oil for a long period of time, leading to increased heat transfer. In this context, the most oxidized oils will promote acrylamide formation and a relationship could be foreseen between acrylamide formation and an increase in the concentration of polar compounds.

3.4. Deposition of frying oil

It should be noted that, after frying, there is a problem related to oil discharge and this must be taken into account. Frying oils cause water and soil pollution problems. Even not being considered a hazardous waste when released into the public waste water system, it pollutes the water resources receptors and blocks the existing fat filters at wastewater treatment plants. In Canada, approximately 125 tons of waste cooking oil are produced annually, of which only about 3 tons are collected. These have been used for the manufacture of soap, oil and animal feed. These feeds are harmful to public health, since they introduce many toxic and even carcinogenic compounds into the food chain through animal meat fattened by such products. On the other hand it can be used for industrial purposes, e.g. to obtain biodiesel. Biodiesel is a fuel that can be obtained from used cooking oil, and can be used as fuel in a mixture with diesel (ratios of 5 to 30 % biodiesel to 95 to 70 % of diesel). This fuel has several advantages both at the energetic and at social, economic and environmental levels.

4. Materials and Methods

4.1. Acrylamide formation in French fries

4.1.1. French fries sampling

Potatoes (cv. *Asterix*, grown in Portugal, size: 40 – 70 mm, category II) were peeled, washed and after draining they were cut into sticks (9.0 mm × 9.0 mm × 60 mm). An electric deep fryer (Philips HD6162, 2200W, 50-60Hz) containing 2.000 mL of vegetable frying oil (Fula, Sovena, Portugal) was used to fry batches of 200 g of potato sticks (Auchan, Portugal) at different frying temperatures (170, 180, 190 and 200 ºC). Potato samples were removed in triplicate after 3, 6 and 9 minutes of frying. For temperatures of 170 ºC and 180 ºC the same oil was used for 10 successive batches, while for temperatures of 190 ºC and 200 °C, only 5 successive batches were performed with the same oil. The options for sampling times and number of batches with the same oil were taken after preliminary experiments where the both the appearance of the potatoes and the final amount of polar compounds in the oil were taken into account. Samples were stored in 100 ml sterile plastic container (VWR, Portugal).

4.1.2. Acrylamide extraction

The extraction of acrylamide from real samples was performed with water as the extractant, according to a combination of the optimum conditions cited in the literature (Wenzl *et al.*, 2003, along with included references).

Five grams of each food sample were ground in a Waring blender (Worten, Portugal), for 3 min and thoroughly homogenized with 5 mL of distilled water. Another 10 mL of water were added and the homogenates were left to stand in a thermostated water bath set at 70 ºC under agitation. After 30 min the homogenates were centrifuged (12000 rpm, 20 min, 4 ºC) to allow precipitation and filtered twice through Whatman No. 2 filter paper.

The filtrates were transferred to 10 mL volumetric flasks and diluted with distilled water until mark. Before analysis three portions (3 mL) of hexane were added to 1 mL of the sample solution to extract the remaining long chain fatty acids that could create problems in chromatographic analyses by generating peaks overlapping with the target analytes or blocking the polar column. The mixture was vigorously shaken and the upper

hexane layer was removed with a Pasteur pipette. 20 μL of the remnant liquid were taken with a Hamilton syringe directly from the lower aqueous layer and injected into the HPLC to be analyzed for acrylamide.

4.1.3. Acrylamide identification and quantification

Acrylamide was identified by HPLC. The system consisted of an isocratic pump (PU 880, Jasco, USA) at a flow rate of 0.6 ml/min (mobile phase: sulphuric acid 0.005 M), an UV detector (870-UV, Jasco, USA) at a wavelength of 200 nm, and a MetaCarb 87H column (300 × 7.8 mm, VARIAN, USA) housed in an oven at a controlled temperature of 30 ºC. The system is associated with an acquisition and data processing software (VARIAN STAR LC Workstation, USA).

A calibration curve with acrylamide standards at 0.1, 0.2, 0.3, 0.5, 1 and 2 $\mu g.ml^{-1}$ was built, as shown in Figure A.0.1 and Figure A.0.2 of the Appendix.

4.2. Polar compounds formation in French fries

The determination of polar compounds through chromatography has become an approved standard method (AOAC, 1995) and is recognized worldwide as the most reliable method for assessing the deterioration of oil during frying (Gil *et al.*, 2004). In this method the frying oils are separated in a chromatography silica gel column into polar and non-polar compounds. The polar compounds are determined indirectly by subtracting the amount of non-polar compounds to the total mass of the sample (AOAC, 1995).

4.2.1. Sampling

Samples of oil for determination of polar compounds were taken during frying of French fries (for determination of acrylamide), at four different temperatures of frying - 170, 180, 190 and 200 °C. For experiments at 170 ºC and 180 ºC the same oil was used for 10 successive frying cycles, while for experiments at 190 ºC and 200 °C, only 5 successive frying cycles were performed with the same oil. The options for sampling times and number of frying cylces with the same oil were taken after preliminary experiments where both the appearance of the potatoes and the final amount of polar compounds present in the oil were taken into account. In each cycle, oil samples were taken at 3 and 9 minutes of frying time.

The oil samples were collected and stored in sterile 50 mL plastic tubes (VWR, Portugal) and kept at room temperature prior to quantification of the compounds of interest.

4.2.2. Column preparation

A glass chromatography column (50 × 2.5 cm) with Teflon valve obtained from Vidrolab, S.A. (Marinha Grande, Portugal) was used and a piece of raw cotton was placed in the bottom of the column to prevent the silica gel from being washed out. 30 mL of eluent consisting of 87 % (v/v) of petroleum ether (boiling point: 40-60 °C) and 13 % (v/v) of diethyl ether, both from J.M. Vaz Pereira, Lda., Portugal were added to the column, removing the remnant air with a glass rod.

In a 100 ml beaker, 25 g of silica gel (Silica gel 60, particle size 0.063-0.200 mm, 70-230 mesh ASTM, obtained from Merck, Darmstadt, Germany) were mixed with 80 ml of the eluent. Air was removed with the help of a glass rod and the suspension was transferred to the column through a glass funnel. The same solvent was used to wash the beaker, the funnel and the walls of the column in order to achieve a quantitative transference of the silica gel to the column. The valve was subsequently open to drain the solvent until its level is about 10 cm above the silica gel. At this stage, the silica gel was compacted by soft vibrations of the column and the excess solvent was drained by closing the valve when its surface coincides with the surface of the silica gel.

Finally 4 g of purified sea sand (from Merck, Darmstadt, Germany) were added through the funnel and washed up again with solvent, letting the solvent's surface coincide with the surface of the sand.

4.2.3. Polar compounds determination

A sample of 2.5 ± 0.1 g was dissolved in 20 ml of the eluent mixture, slightly warmed up and cooled to room temperature. The volume was adjusted to a final value of 50 ml. From this solution 20 ml were drawn and gently placed at the top of the chromatography column. This solution was eluted by opening the column's valve until the liquid surface coincides again with the surface of the sand.

A 250 ml round-bottomed flask was previously dried with a nitrogen flow and weighted until constant weight; this flask was placed under the column to receive the fluid eluted from it.

Non-polar compounds were subsequently eluted with 150 ml of the eluent mixture, adjusting the flow so that the 150 ml take about 60 to 70 minutes to drain from the column.

The solvent was separated from the fraction of non-polar compounds using a rotary evaporator in a water bath at a temperature of 55 ºC, under vacuum. The fraction remaining in the flask, containing the non-polar compounds, further dried under a nitrogen flow until constant weight.

4.2.4. Determination of polar compounds concentration

The percentage of polar compounds (% polar compounds) present in the oil samples, which are retained in the chromatography column, was calculated from the following expression:

$$\%_{polar\ compounds} = 100 - \left(\frac{m_{\text{flask + resudue}} \quad m_{flask}}{\frac{V_{used}}{V_{total}} * m_{sample}} * 100 \right) \qquad \textbf{(Eq. 2.1)}$$

Where:

- $m_{flask\ +\ residue}$ - total flask mass after drying in the evaporator
- m_{flask} - mass of dry flask
- V_{used} - volume of sample used for polar compounds separation (20 ml)
- V_{total} - total volume of sample preparation (50 ml)

5. Results and Discussion

5.1. Acrylamide formation in French fries

The determination of acrylamide concentration formed during potatoes frying was performed using HPLC, and the corresponding retention time was of about 36 minutes. A calibration curve was previously established with standards of 0.1, 0.2, 0.3, 0.5, 1 and 2 $\mu g.ml^{-1}$, as shown in Figure A.0.1 and Figure A.0.2 (please refer to the Appendix).

This calibration curve was used to determine acrylamide concentrations 3 and 9 minutes after the beginning of frying under different temperatures - 170, 180, 190 and 200 °C, as represented in Figure 2.7 to Figure 2.10.

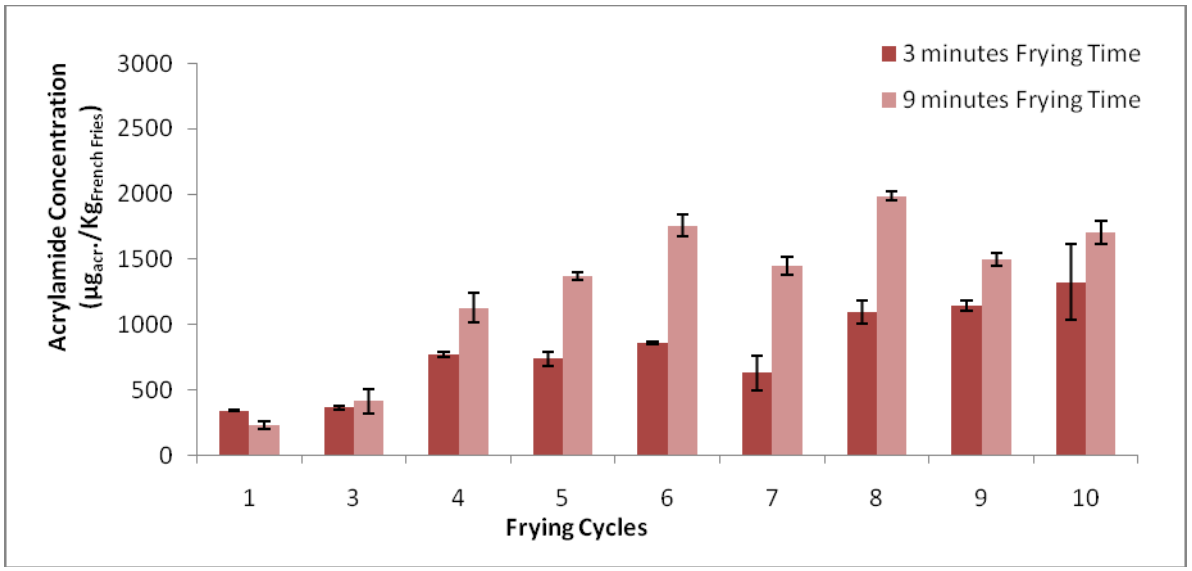

Figure 2.7: Acrylamide concentration in French fries at 170 °C for different frying cycles

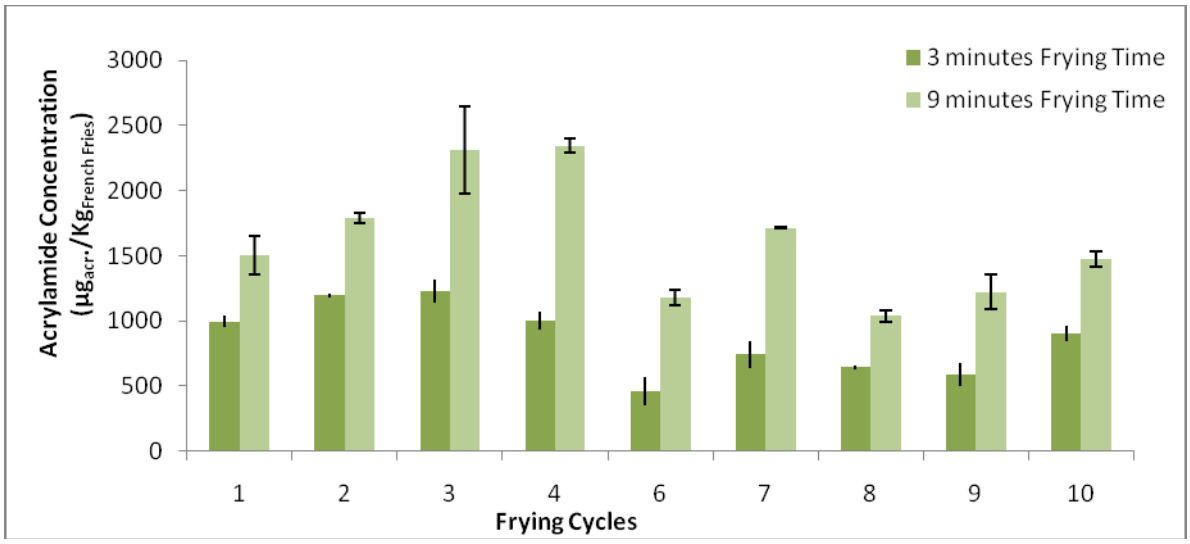

Figure 2.8: Acrylamide concentration in French fries at 180 °C for different frying cycles

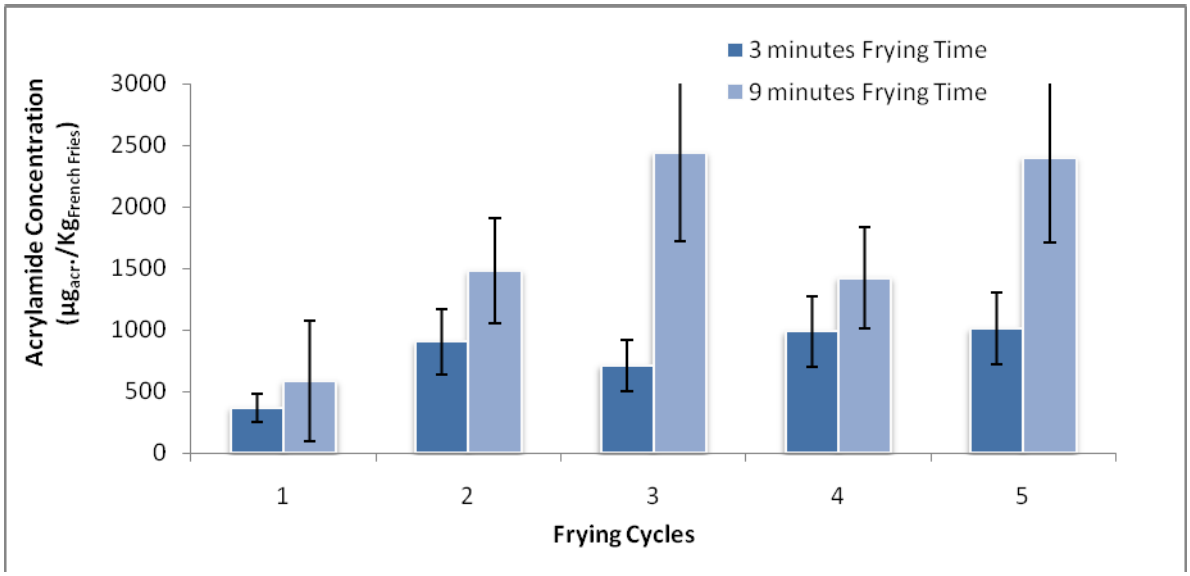

Figure 2.9: Acrylamide concentration in French fries at 190 °C for different frying cycles

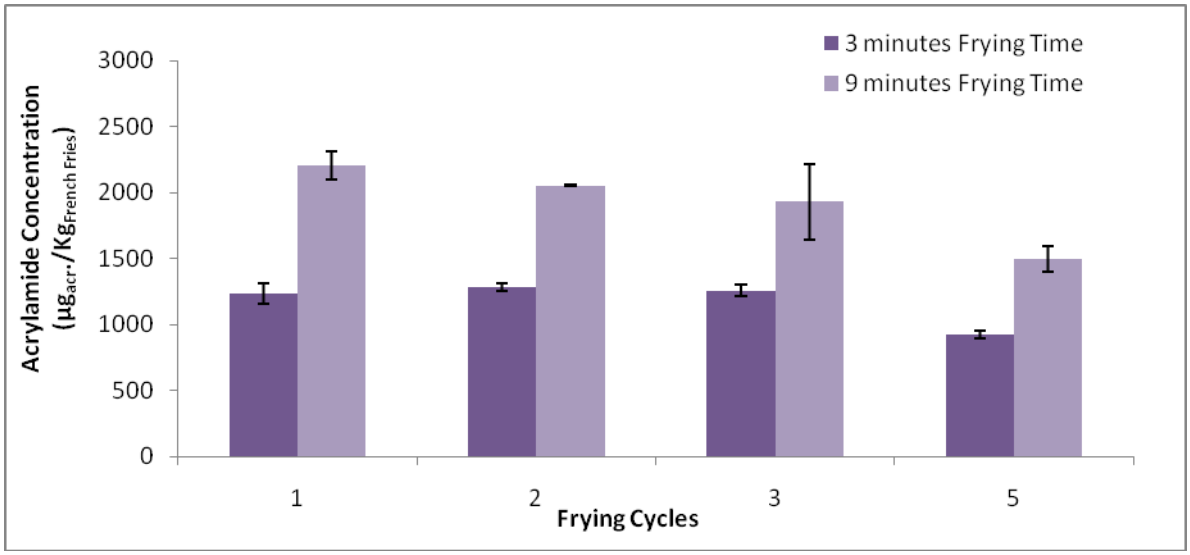

Figure 2.10: Acrylamide concentration in French fries at 200 °C for different frying cycles

These results show that acrylamide formation in potatoes during frying reaches concentrations exceeding 1000 $\mu g.kg^{-1}$ (which, according Amrein (2005), are high concentrations) at all temperatures tested. According to Grob *et al.* (2003), as already mentioned, potatoes composition together with processing at high temperatures promotes the formation of this carcinogenic compound. In the present work, as the lot and variety of potatoes were the same for the various tests performed, the differences in temperature of frying are very likely the cause of the different concentrations of acrylamide formed in the different experiments.

According to Figure 2.7, Figure 2.8, Figure 2.9 and Figure 2.10 the concentration of acrylamide in the French fries after 9 minutes of frying is always higher than the concentration after the first 3 minutes, and the range of acrylamide concentrations formed increases, in general, with increasing temperature. For example, for the frying temperatures of 170, 180, 190 and 200 °C, at the 3rd frying cycle, the acrylamide concentrations formed (i.e., the difference between acrylamide concentration after the first 3 and the 9 minutes of frying) are 49.40 ± 47.76 $\mu g_{acr}.kg_{French\ fries}^{-1}$, 1087.75 ± 172.71 $\mu g_{acr}.kg_{French\ fries}^{-1}$, 1730.43 ± 375.28 $\mu g_{acr}.kg_{French\ fries}^{-1}$ and 671.22 ± 145.49 $\mu g_{acr}.kg_{French\ fries}^{-1}$, respectively.

In addition to the increase of acrylamide concentration with frying temperature, an increase of acrylamide concentration during successive frying cycles occurs for each temperature. That is, using the same oil for successive frying cycles at a certain temperature gradually increases acrylamide concentration in the fryed products, until it reachs a peak. From this point onwards there are no statistically significant differences between subsequent values of acrylamide concentrations for any of the temperatures tested (p = 0.333 for a

frying temperature of 170 °C, $p = 0.232$ for a temperature of frying of 180 °C, $p = 0.155$ for a frying temperature of 190 °C and $p = 0.600$ for a frying temperature of 200 ºC).

Figure 2.11 shows that, for all temperatures tested, the amount of acrylamide formed increases through the course of frying, which results in increased values of the relative "variation of acrylamide formation" (VAF) represented in Figure 2.11 and calculated by:

$$"VAF" = \frac{[\text{acrylamide}]_{9\ \text{minutes}} - [\text{acrylamide}]_{3\ \text{minutes}}}{[\text{acrylamide}]_{3\ \text{minutes}}} \times 100 \qquad \textbf{(Eq. 2.2)}$$

Where:

- VAF - variation of acrylamide formation
- $[\text{acrylamide}]_{3\ \text{minutes}}$ - concentration of acrylamide after 3 minutes of frying
- $[\text{acrylamide}]_{9\ \text{minutes}}$ - concentration of acrylamide after 9 minutes of frying

Comparing the different curves in the initial frying, the variation in the formation of acrylamide (VAF) increases with the temperature of frying; this trend is kept until VAF reaches a maximum point at each temperature. Further, for the first frying, there is less variation in the concentration of acrylamide formed in the test performed at 170 °C and higher in the test at 200 ºC.

Also, as the temperature of frying increases, the maximum variation of acrylamide formation occurs at an earlier frying cycle. For a frying temperature of 170 °C, VAF peaks at the 7th frying cycle (130 %), while at 180 and 190 °C, such peak occurs at the 5th (160 %) and 3rd (243 %) frying cycle, respectively. These results show that, in addition to the fact that maximum acrylamide formation occurs at an earlier frying cycle, VAF is greater for higher temperatures. For samples fried at 200 °C, the maximum variation occurs at the first frying cycle, meaning that for this temperature acrylamide is forming at a very high rate; however, the value of the change is against the trend of the previous three temperatures. Once each frying cycle has a duration of 9 minutes, and the percentage of acrylamide formed is calculated from the difference of its concentration at 3 and 9 minutes, probably the peak of formation of acrylamide at this temperature would have been registered at a frying time even lower than 3 minutes.

Thus it is possible to confirm that when frying continuous fried foods, French fries in this case, acrylamide formation and concentration will be increasing. This trend is maintained up to a point where the formation rate of acrylamide is maximum, and this point

corresponds to a frying cycle where the potatoes acquire higher acrylamide concentrations. From this peak onwards, VAF is decreasing, and the concentration of the compound in the food tends to stabilize.

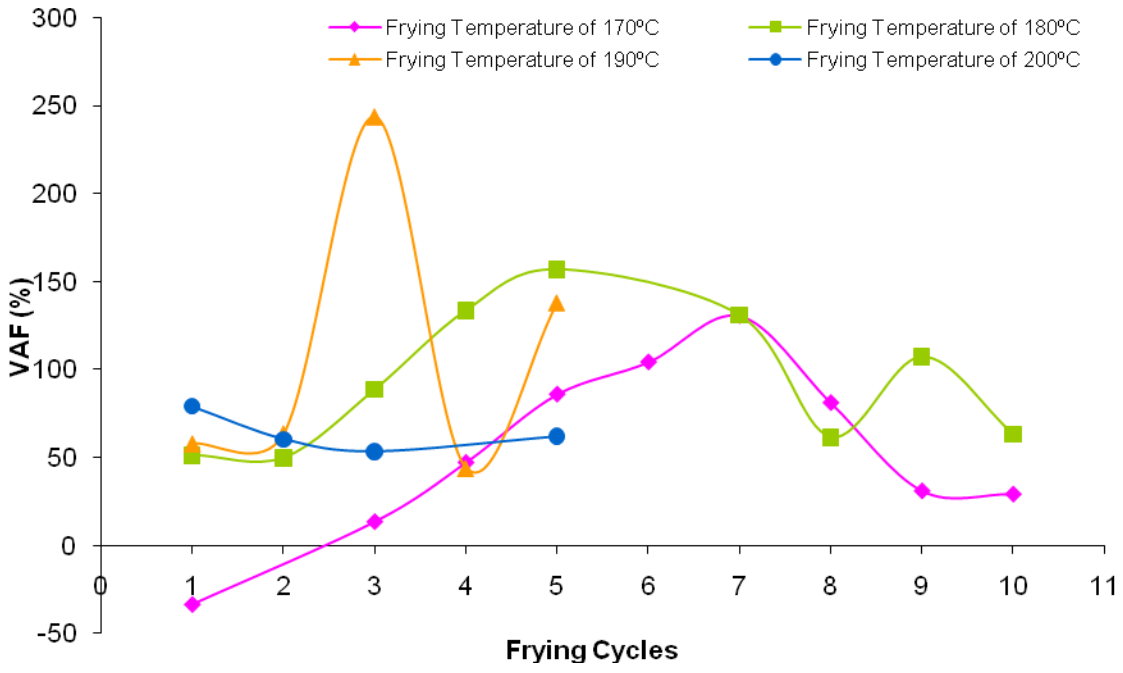

Figure 2.11: Relative change (in terms of VAF) of acrylamide concentration formed during frying at different temperatures as a function of the number of frying cycles

5.2. Formation of polar compounds in French fries

Oils and fats used to fry various products suffer various reactions during processing, including oxidation, hydrolysis and polymerization (potential for thermal degradation of frying oil) from which result substances such as polar compounds that are known to be related to the degradation of fats and oils and have carcinogenic effects. Thus, it is also important to control frying oils in terms of the presence and concentration of these compounds. Figure 2.12 presents the percentages (w/w) of polar compounds formed during the frying experiments.

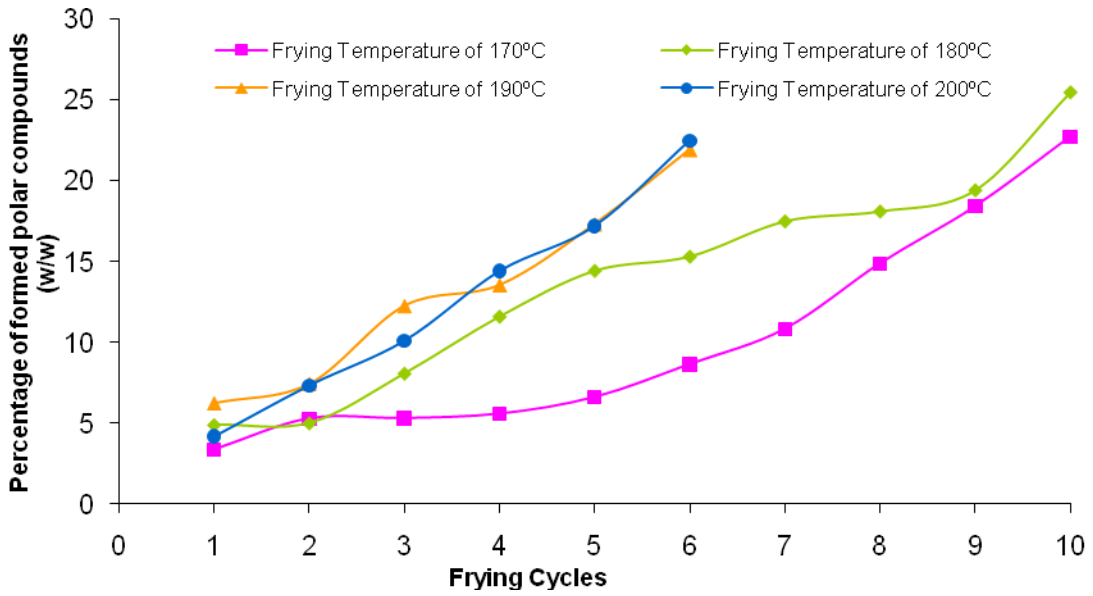

Figure 2.12: Percentage of polar compounds formed during the number of frying cycles at different temperatures

Figure 2.12 clearly shows that, as frying occurs at a certain temperature, the percentage of polar compounds is increasing steadily. This is why oil, used in short periods, improves frying when compared to fresh oil, due to polar compounds that are forming and promoting a better contact between the oil and both the water at the food surface and the steam which food releases at the same time, resulting in a more efficient and uniform heat transfer and acquisition of flavor (Fellows, 2000). Moreover, as the temperature of frying increases the percentage of polar compounds that is formed is greater, and the legal limit of polar compounds (25 %) is achieved for a lower number of frying cycles since the oil degradation is greater for higher temperatures. Thus, in order to meet the legal requirements presented in Table 2.2 (Chapter 2), a frying temperature of 180 ° C should be used, resulting in the use of oils for a longer period while still ensuring that safe amounts of polar compounds are present and providing a reduction in costs since the degradation of oil is slower.

This is in line with Rossell (2001), who stated that high temperatures cause a rapid deterioration of oils and the formation of free fatty acids, which alter the viscosity, color and flavor and promote foaming. For example, for the 3rd cycle of frying, the percentage of polar compounds in oil when heated to temperatures of 170, 180, 190 and 200 °C were 5, 12, 13 and 14 % respectively. According to Gertz (2006), the system will become more complex as the frying process proceeds once the composition of foods being processed as well as the physical and chemical characteristics of frying oils change continuously.

Furthermore, although there was a continuous increase in the percentage of polar compounds through the use of the same oil, it would be interesting to know the magnitude of the change (increase) of polar compounds concentration over several frying cycles using the same oil.

Figure 2.13 shows a clear reduction in the relative percentage of polar compounds that form during the frying, the rate of formation of polar compounds being higher in the first frying, decreasing from that point onwards towards a constant value. The relative "variation of polar compounds formation" (VPCF) is calculated by:

$$"\text{VPCF}" = \frac{[\text{polar compounds}]_{9\ \text{minutes}} - [\text{polar compounds}]_{3\ \text{minutes}}}{[\text{polar compounds}]_{3\ \text{minutes}}} \times 100 \qquad \textbf{(Eq. 2.3)}$$

Where:

- VPCF - variation of polar compounds formation
- [polar compounds] $_{3\ \text{minutes}}$ – percentage of polar compounds after 3 minutes of frying
- [polar compounds] $_{9\ \text{minutes}}$ - percentage of polar compounds after 9 minutes of frying

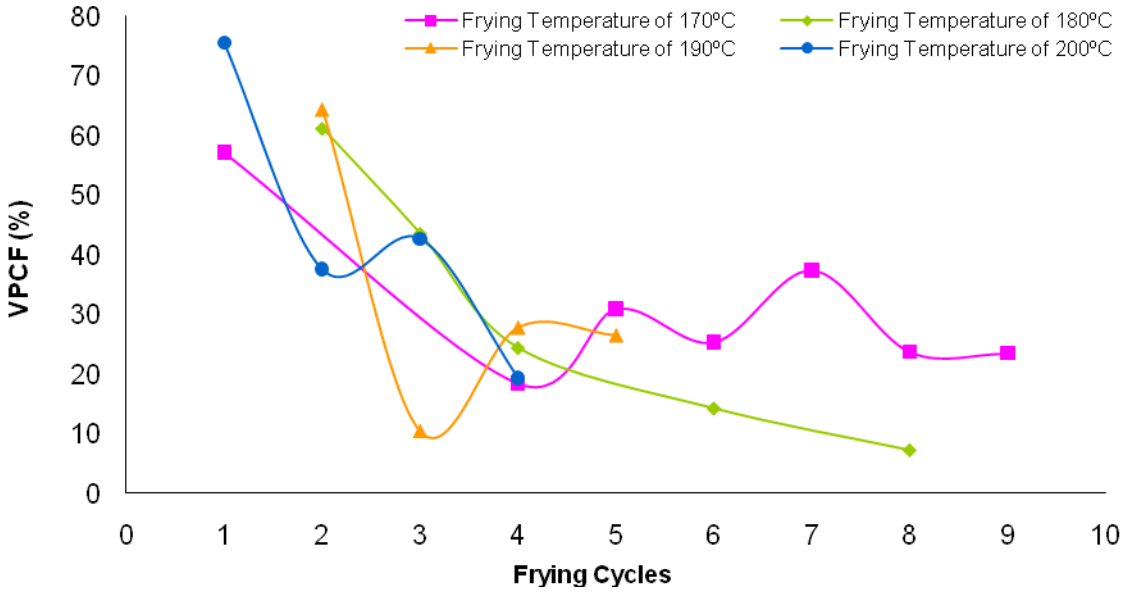

Figure 2.13: Relative change in the polar compounds formed as a function of the number of frying cycles at different temperatures

5.3. Relationship between acrylamide formation and polar compounds

The results presented in Figure 2.7, Figure 2.8, Figure 2.9, Figure 2.10 and Figure 2.11 indicate that the use of the same oil for repeated frying cycles of French fries, at a certain temperature, leads to products with successively higher acrylamide concentrations up to a maximum point, after which such concentration tends to remain constant. That is, on the one hand there is the increasing acrylamide concentration during frying at a certain temperature, and on the other hand it is known that oxidative and hydrolytic degradation processes occur leading to the formation of polar compounds and gradually change the oil in which foods are prepared. Given the two situations that occur during frying, this suggests that the conditions of polar compounds formation may also be related with the acrylamide formation in processed products.

This suggestion is in line with the work of Mestdagh *et al.* (2007), which certifies that the degradation processes of oil may cause or influence the acrylamide formation during frying. In order to confirm this assertion Figure 2.14 was built between the variation of polar compounds formation (VPCF), resulting from the degradation of frying oil, and the variation of acrylamide formation (VAF), over several frying cycles at the same temperature. This figure shows that it is possible to relate the acrylamide formation (calculated as VAF) and polar compounds formation (calculated as VPCF) for the four temperatures studied. For a frying temperature of 170 °C, the increase in VPCF is accompanied by an increase of VAF, and the rate of acrylamide formation is higher than that for polar compounds. In the case of frying temperatures of 180 and 190 °C (see Figure 2.15 and Figure 2.16), it appears that after a point where VAF reaches a maximum value, it decreases with the increase of VPCF. Although at some temperatures it is difficult to find a trend after this maximum has been attained, it may be possible to conclude that the most significant decrease has been registered for a temperature of 190 ºC. At those frying temperatures the rate of acrylamide formation decreases with the increasing rate of polar compounds formation.

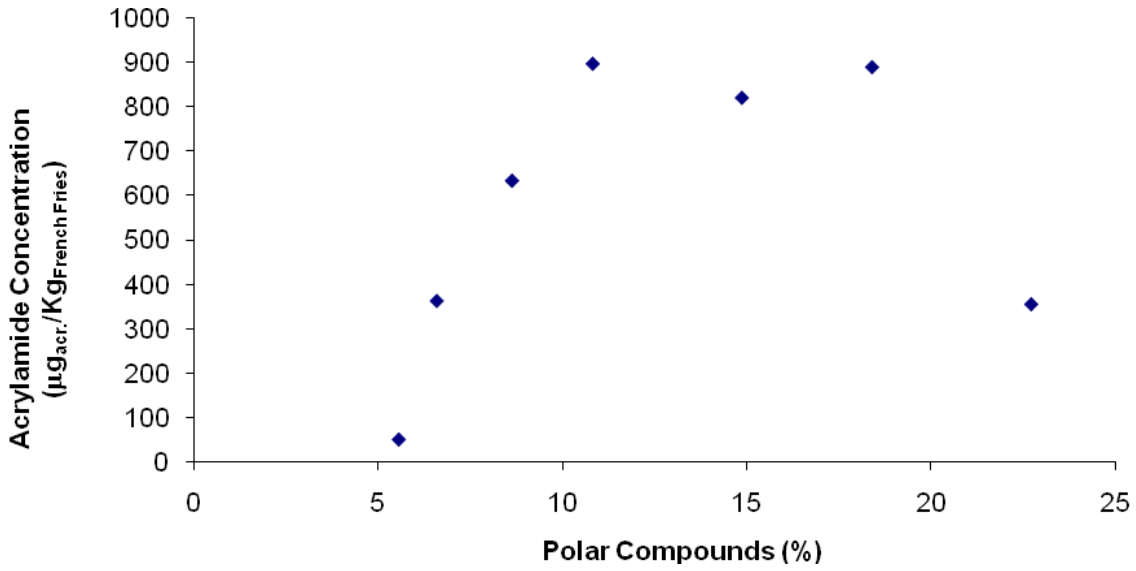

Figure 2.14: Representation of the relationship between acrylamide formed in French fries and the percentage of polar compounds produced at 170 ºC

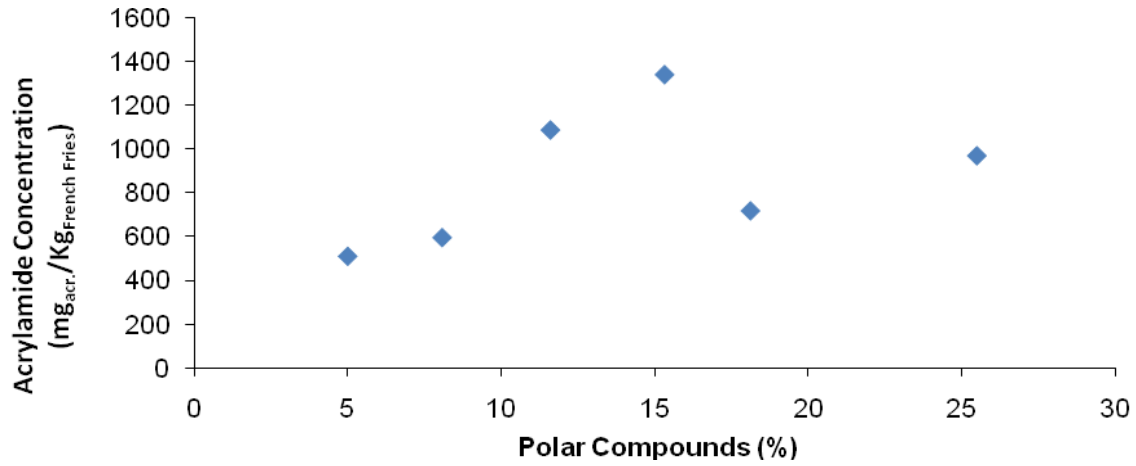

Figure 2.15: Representation of the relationship between acrylamide formed in French fries and the percentage of polar compounds produced at 180 ºC

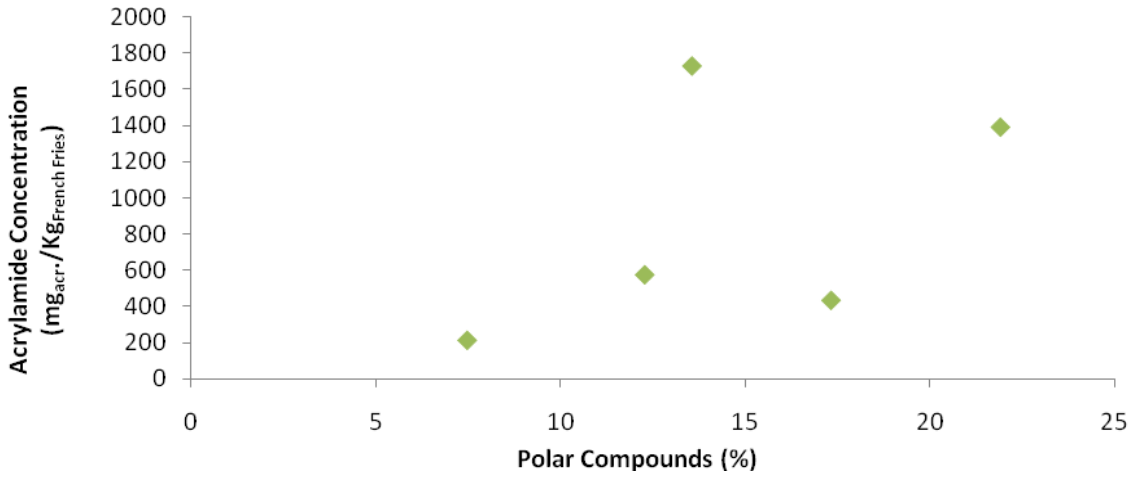

Figure 2.16: Representation of the relationship between acrylamide formed in French fries and the percentage of polar compounds produced at 190 ºC

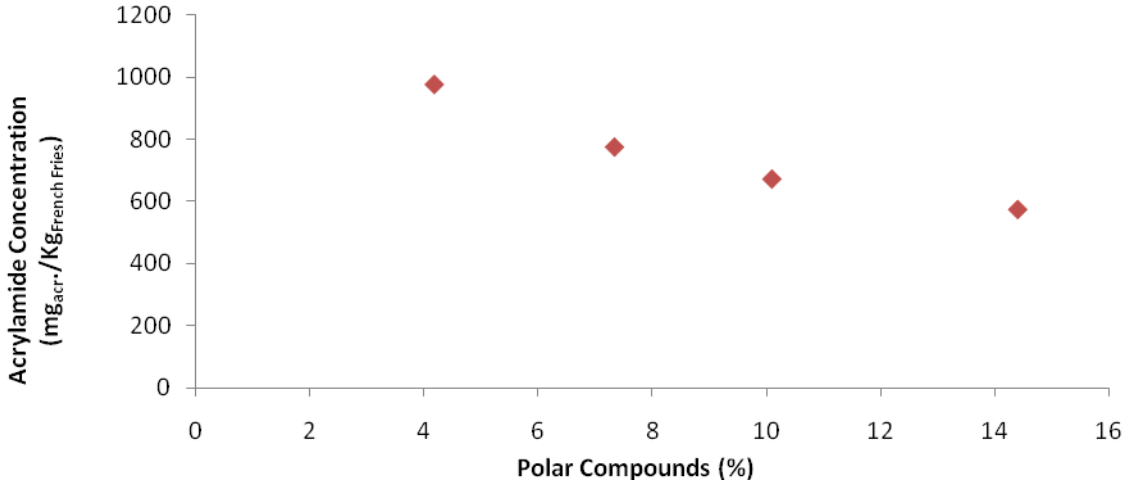

Figure 2.17: Representation of the relationship between the relative increase of acrylamide (VAF) formed in French fries and the relative change in the percentage of polar compounds (VPCF) produced at 200 ºC

For the frying temperature of 200 °C (Figure 2.17), VAF shows a much smaller decrease with increasing VPCF; the rate of formation of acrylamide is less dependent of the rate of formation of polar compounds.

The results obtained for a frying temperature of 170 °C may be justified according to Gertz (2004) and Gertz *et al.* (2003) by the formation of polar compounds (through oxidation of oils) which influence the surface tension between the food and oil, keeping the water in the frying oil for a long period of time culminating in increased heat transfer. Thus,

for higher rates of polar compounds formation there is also a greater variation in acrylamide formation (due to the presence of water). In the case of frying temperatures of 180 and 190 ºC this behavior is less evident; once the temperatures are higher, there may have been a decrease of water content in oil. Thus, according to Ahrné *et al.* (2007), high temperatures and low humidity result in a lower heat transfer rate and consequently a reduction of acrylamide formation.

6. Conclusions

During processing of some food stuffs a high number of chemical, enzymatic and non-enzyme reactions happens, which in addition to being responsible for changes in color and flavor of the final products may, according to the Maillard reaction, lead to the formation of both harmful compounds, including acrylamide, and compounds with interesting functional properties. In the case of oils and fats used in the frying process of various products, these reactions also result in polar compounds known to have carcinogenic effects.

This work focused on determining the conditions of acrylamide formation during frying of French fries. The mechanism of acrylamide formation is associated to the Maillard reaction, where free asparagine and reducing sugars are the main precursors (Mestdagh *et al.* (2007). Potatoes are rich in asparagine (Pedreschi *et al.*, 2004) and acrylamide concentration in foods is linked to the different levels of acrylamide precursors found in raw materials (Mestdagh *et al.*, (2007). For the potatoes, several studies showed that free sugars and asparagine concentrations can vary between varieties (Amrein *et al.*, 2004 and Amrein, 2005), influencing the concentration of acrylamide formed upon frying. In addition to food composition, processing conditions also exert a great influence on acrylamide formation (Pedreschi *et al.*, 2004).

The results obtained concerning polar compounds and acrylamide formation in French fries, show that during successive frying cycles there is a gradual increase of acrylamide concentration until the concentration is maximum (occurring at the 7^{th}, 5^{th} and 2^{nd} frying cycle for the frying temperatures of 170, 180 and 190 °C, respectively), stabilizing at that time; this occurred together with a progressive increase in the percentage of polar compounds with the number of frying cycles. Acrylamide and polar compounds concentration increases with the increase of frying temperatures, in parallel with the accelerated deterioration of oils by reactions of (auto) oxidation and polymerization (Gertz, 2006), and the formation of free fatty acids (Rossell, 2001). Although this occurs for each of the temperatures, and the global trend is the same, there are differences in the relationship between VPCF and VAF which is not the same for different temperatures under study. Results seem to point at the existence of a relationship between the kinetics of polar compounds and acrylamide formation.

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Chapter 3.

IDENTIFICATION, FORMATION AND DEGRADATION OF HARMFUL AND FUNCTIONAL COMPOUNDS IN BAKERY PRODUCTS

Abstract

During thermal processing, some foods are subjected to many reactions which, more than conferring colour and flavour to the final product, are linked to the formation of harmful compounds, such as acrylamide and polar compounds.

The aim of this part of the work was to assess if acrylamide and tocopherols are formed and/or destroyed during thermal processing and shelf-life of baked products.

During the shelf-life period, acrylamide and moisture content did not show statistically significant differences, however for tocopherols significant differences were found. Tocopherol concentration decreased 56 % during storage.

From the Pão-de-Ló samples tested, the one baked at 225 ºC showed the highest acrylamide concentration (1509.36 ± 250.44 $\mu g_{acr}.kg^{-1}$).

1. Bakery

The baking of bread is a process that includes three phases: crushing, fermentation and baking. Of all the cereals, wheat, rye, barley, oats, corn and rice, only the first two are important to get good structured bread (Coultate, 1989). However, it is the wheat that occupies a unique position among the cereals, due to the capacity of the flour to form, together with water, salt and yeast, the dough (Yúfera, 1998).

The first step in preparation for mixing is assembling and weighing the ingredients. Some ingredients require special preparation. The yeast, whether compressed or dry must be suspended in water according to manufacturer's instructions. The temperature of the water is important in controlling dough temperature. If mechanical refrigeration equipment is not available to chill the ingredient water, ice may be required. Most bakeries are equipped with mixing machines that circulate chilled water or refrigerants through coils between the walls of the mixing bowl. In that case ice would not be required. The purpose of mixing the dough is to distribute the yeast cells throughout the dough, to provide a uniform distribution of the substrate needed for yeast growth, and to form and develop the gluten. Gluten is formed when the two proteins of the flour, gliadin and glutenin come into contact with water. In gluten, glutenin and gliadin proteins contain a high proportion (over 35 %) of glutamic acid and relatively high amounts (12-14 %) of proline (Yúfera, 1998). The addition of chemical agents and antioxidants to both flour and dough can regulate the rheological characteristics of the dough, by intervening at the levels of maintenance and regulation of the intermolecular links established between those molecules (Yúfera, 1998). The time required to develop the gluten depends on the strength of the flour, and the speed of the mixing machine. Generally, the stronger the flour proteins the longer it takes to develope the gluten and the faster the machine, the shorter the mixing time. In small and medium size bakeries, the most used methods to mix doughs are the Straight Dough Method and the Sponge and Dough Method. Most of the large commercial wholesale bakeries use the Continuous Mixing Method that will be discussed in a later paragraph.

In the straight dough method of mixing all dough ingredients are mixed at one time, and prepared for a single fermentation process. Generally, the fermentation time for the straight doughs will vary from 2-1/2 hours to 3 hours. These doughs are also punched after about 80 percent of the fermentation time has elapsed and given an additional 20 percent fermentation before make-up.

In the Sponge and Dough Method, there are two mixing periods and two fermentation periods. Part of the formula ingredients are mixed and allowed to ferment for 4 to 6 hours. After the fermentation process is completed, the second part is called the Dough. Basically, the fermented sponge is thrown back into the mixer and the ingredients for the second part are added. The fermented sponge and all the second ingredients are mixed together to form the dough. After the gluten has been fully developed, the dough is dumped into a stainless steel dough trough and given a second fermentation time. As compared to the sponge, the fermentation time of the dough is very short (15 to 20 minutes).

The Continuous Mixing Method is very popular among large commercial bakeries. The bread produced by this method has very fine tight grain similar to the grain of cake. A baking liquid, brew or liquid sponge is prepared and allowed to ferment in stainless steel tanks under controlled temperature conditions for several hours. The fermented mixtures are cooled by the use of refrigerated coils between the walls of the tanks until ready to be used. This process eliminates setting individual sponges and doughs, and the need for dough troughs and large fermentation rooms. After going through the developer, what happens next varies with different bakeries. Some bakeries run the dough through conventional make-up equipment, rather than extruding the dough directly into the pan as was done a few years ago, because it is claimed that a better quality loaf results. Fermentation starts immediately after the yeast is put into the mixture. However, the fermentation period is considered to begin when the sponge or dough is dumped out of the mixer into the dough trough and rolled into the fermentation room which is maintained at a temperature of 27 ºC. and 75 % relative humidity. The chemical changes that occur continue until the yeast is killed by the heat of the oven. This is when the internal temperature reaches about 60 ºC. The fermentation period is considered to end when the dough goes to the divider to be divided into individual loaf pieces (loaves). Leavening the dough is one of the essential things that take place during fermentation. During fermentation, the yeasts ferment maltose and glucose and release CO_2 which leads to an increase in the mass volume. Carbon dioxide gas is produced and held by the gluten network. This causes the whole dough mass to expand which helps to condition the gluten, as was mentioned previously.

Alcoholic fermentation is the most desirable type of fermentation. To ensure that this type of fermentation predominates, the dough should come out of the mixer between 25 ºC. and 28 ºC and kept at 27 ºC in a fermentation room with 75 % relative humidity. Due to chemical changes that take place during fermentation , the sponge or dough temperature will increase to about 30 to 32 ºC, which is still in the range of alcoholic fermentation. In this

range, small amounts of acetic and lactic acids are produced which is beneficial. However, at higher temperatures a lot more of these acids is produced along with butyric acid which is very undesirable. This results in a poor quality product having a strong undesirable taste and flavor. The crust will have a pale straw color; the loaf will have poor symmetry of form and a very open grain. The crumb will have a yellowish color rather than a bright white color.

Fermentation time for sponges vary from 4 to 6 hours depending upon the strength of the proteins of the flour, temperature of the sponge, and the amount of yeast used. Basically the same procedure is used to determine fermentation time of the sponge as for straight doughs, with the exception that the sponge is not punched, but observation is made to determine when the sponge begins to recede slightly. This is normally known as the breaking point of the sponge. When this occurs, ca. 80 % of the total fermentation time has been achieved. The remaining 20 % fermentation time is calculated the same way as when determining the time after the punch for straight doughs. Dividing and scaling consists of cutting the dough (by hand or machinery) into loaf size pieces and weighing the pieces to ensure uniformity. Because of the average loss of about 12 % during make-up, proofing, baking and cooling, this loss must be taken into account. In the molder the dough passes through three distinct stages. Flattening is done in the head rollers of the molder. Secondly, the sheeting rollers sheet the dough into a flat piece of dough and the curling rollers and thread rollers twirl each piece of sheeted dough and give it a cylindrical shape. Subsequently, the drum or pressure plate rolls and seals the loaf into its final form. Conventional molders curl the dough in the same direction that it was sheeted. It is difficult to produce bread with a close uniform grain with this type of molder. For this reason some bakers twist two pieces of dough together or use cross grain molders. Cross grain molders curl the dough in the opposite direction from which it was sheeted. That is, the dough is caused to turn right or left after it has been sheeted and is then curled. This is called cross grain molding. Cross grain molding and twisting two pieces of dough together prior to putting them into the baking pan both produces loaves with a close smooth grain and texture.

Pan proofing is the process of rolling the panned and racked molded dough pieces quickly into the dough proofing cabinet which is well insulated and maintained at a temperature of 35 to 38 ºC and a relative humidity of 85 %. At the end of the pan proofing period, the loaves are loaded quickly, but carefully into the oven for baking (see Figure 3.1).


Figure 3.1: Cooking example of baking products

In large commercial bakeries the panned loaves are carried automatically on conveyor belts and loaded automatically into the oven. Oven temperatures vary from about 210 to 240 ºC. During the first minutes of the baking process the carbon dioxide gas within the dough expands. This expansion causes a very rapid rise of the dough known as ovenspring. Fermentation is more vigorous and more rapid at this stage than at any previous stage. When the inside temperature of the loaf reaches 60 ºC the yeast is killed and fermentation ceases. Alcohol produced during fermentation evaporates in the form of vapor. After the ovenspring the pliability of the dough gradually lessens and the dough becomes set and slowly changes to bread. Some of the moisture evaporates, the starch becomes gelatinized and more digestible, and the gluten and other proteins become coagulated. After the loaf sets, the intense heat dries out the part exposed to the air and causes a crust to form. The golden brown color of the crust is the result of chemical changes known as *browning* resulting from the Mailard reaction, in which amino acids are transformed into aldehydes. During this reaction there is a source of furfural, hydroxymethylfurfural, and other aldehydes (De Man, 1999). Within the loaf, the crumb near the crust is subjected to a temperature as high as 150 ºC. The temperature gradually decreases towards the center of the loaf at which point it reaches about 100 ºC. the same temperature at which water boils at sea level. At this temperature the gluten becomes stiff enough to give the loaf permanent form and retain its structure.

When the baking process is complete, the loaves are unloaded manually or automatically and either dumped on cooling racks or travel on enclosed air conditioned

conveyor belts to the cooling area. The loaves are allowed to cool for about 1 hour and is ready to be sliced and wrapped or bagged.

2. Tocopherol

In the last decade, the presence of compounds that have an inhibitory effect in carcinogenic cells has been thoroughly evaluated in foods and food ingredients. The nutritional prevention of cancer in human populations is based on mechanisms of action, toxicity and efficacy of certain chemical compounds, also called dietary agents, and micronutrients present in foods, which have been associated with the reduction of the risk of cancer (KHACHIK, 1991). These components include, among others, dietary carotenoids, retinoids, tocopherols and ascorbic acid.

The tocopherols and tocotrienols are a group of basic constituents of the human diet and are conventionally accepted to have beneficial health effects. There are four types of tocopherols and vitamin tocotrienols. All of them consist of a chromanol ring and a hydrophobic side chain, which is a phytil in tocopherols and a three double bonds isoprenil in tocotrienols (see Figure 3.2). The above referred compounds can be distinguished by the number and location of methyl groups in the chromanol ring (Ryynänen *et al.*, 2004).

Edible fats contain different tocopherols (δ-, β-, γ- and α-) that have different biological activities, allowing to precisely quantify the vitamin E amount through each of the homologues. In addition, these homologues' total content and distribution in oils vary with species and variety and this can be used to test for possible adulterations (Aucejo-Mauro *et al.*, 2003).

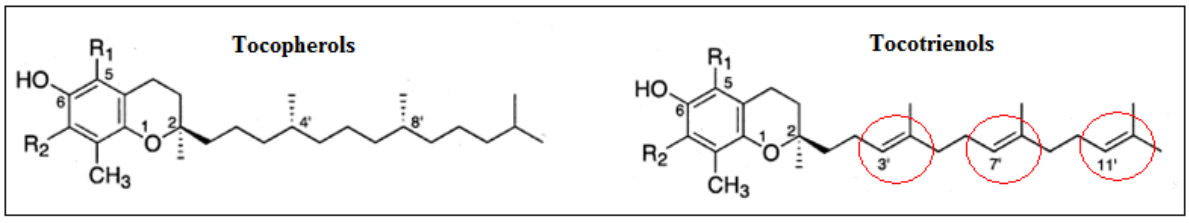

Figure 3.2: Molecular structures of tocotrienols and tocopherols (marked in the last double bond)

The tocopherols are natural antioxidants (Sanchez-Machado *et al.*, 2006), a natural form of vitamin E. Although acting primarily as an antioxidant (Pastor-Ferrer. *et al.*, 2000; Schneider, 2005, Lee *et al.*, 2003 and Sivakumar *et al.*, 2005), vitamin E may also be a pro-oxidant and can function as signaling molecule, regulator of gene expression, and possibly act on the prevention of cancer, heart disease and arteriosclerosis (Schneider, 2005 and

Sivakumar *et al.*, 2005) related to oxidative deterioration due to its antioxidant action. Moreover, this form of vitamin E participates in muscles and other tissues formation and functioning, and protects essential fatty acids.

The various forms of vitamin E reflect an antioxidant potential in food and therefore can improve its shelf life. Moreover, according to Nelis *et al.* (2000), these compounds provide information on possible effects of deterioration (oxidation) of food processing and storage.

All those forms of vitamin E possess antioxidant activity, but α-tocopherol is chemically and biologically more active, while the other forms have a lower activity: γ-tocopherol and δ-tocopherol have 10 % and 1 % of the α-tocopherol activity, respectively. The activity is based on the antioxidant action that slows or prevents the lipids oxidation. In general, when the activity of vitamin E in food is evaluated, the sum of the different tocopherols is calculated taking into account their relative activities.

Although α-tocopherol and γ-tocopherol are considered the most biologically active forms of vitamin E, recent studies suggest that tocotrienols are more efficient in the prevention of cardiovascular disease and cancer, making them also worthy of scientific research (Lee *et al.*, 2003).

According to Solomon *et al.* (1990), possible sources of tocopherols are: vegetable oils (corn, sunflower, soybean, etc.), olive oil, vegetable margarine, wheat germ, green-leaf vegetables (spinach, cabbage, etc.), cereals, oil seeds (walnuts, almonds, cashews, etc.), milk, eggs, liver or fish (Aoun *et al.*, 2005). Considering the vitamin E food sources, healthy people can obtain enough tocopherols from an healthy diet, preventing the symptoms of deficiency of vitamin E (Ryynaänen *et al.*, 2004).

The storage of raw materials and processed foods can cause a decrease in the vitamin E amount (Chun, 2002 e Fellows, 2000). The packaging methods and materials, time and storage temperature, food characteristics and its susceptibility to lipids oxidation, the availability of other natural or synthetic antioxidants in food, among other, affect the stability of vitamin E during food storage. The lipids oxidation is the main cause of secondary compounds and rancid development, and a number as other reactions that reduce the shelf life and nutritional value of food (Chun, 2002 and Fellows, 2000).

Vitamin E degradation rate depends on the availability of oxygen, temperature of storage, water activity, fat content and food composition (Chun, 2002). Both synthetic and natural antioxidants are used in foods rich in fat, to slow down lipid oxidation (minimizing the rancidity), delaying the formation of toxic oxidation products, and allowing the

maintenance of nutritional quality during shelf life (Madhavi *et al.*, 1996). The oxidation of lipids is a very important factor in heart disease, arteriosclerosis, cancer and the ageing process. In humans, a complex system of antioxidant defense normally protects the cell from the harmful effects of free radicals.

According to Eitenmiller and Lee (2004), low levels of α-tocopherols in oils are related to the rapid oxidation in frying products. According to the authors, canola oil with lower levels of tocopherols had a more rapid degradation rate. There was an inverse relationship between the total polar compounds and the loss of tocopherols. Oils with high rates of degradation of tocopherols have shown high rates of polar compounds formation.

3. Bakery products Raw Materials

The manufacture of bakery and pastry products is a good example of how different ingredients, quantities and methods of processing can be used. The main ingredients are: flour, fat, water and salt. Eggs, sugar, milk and cream, cocoa and its products, coffee, fruits (dried, fresh or crystallized), food additives and fermentation agents can also be added. Different types of products contain similar ingredients, but the proportion of these ingredients and the techniques used in each production make the product unique (Robinson *et al.*, 2001).

The functional properties of the ingredients used in the products manufacture are many and varied, and each ingredient can perform more than one function in a particular product (Cauvain and Young, 2006). According to these authors, the ingredients are used in order to adapt the final product's properties: stabilization and distribution of air (achieved through the use of e.g. fat and emulsifiers), nutritional quality of the meal (e.g. controlling fats in cookies and crackers), taste (e.g. using chocolate or cocoa powder), color (which may result from the addition of a given ingredient - e.g. cocoa - or through reactions occurring during cooking - e.g. Maillard reaction and browning) and increase of shelf life (e.g. using preservatives).

Each of these ingredients will be presented in terms of their composition and properties.

3.1. Wheat flour

Wheat, a non-perishable cereal with an oval fruit belonging to the family *Graminea* and genus *Triticum*, is easy to store and transport, has good nutritional properties and allows the production of a wide range of products of interest (Bushuk and Scratch, 1994). The wheat seed major application is the production of flour varieties. Grains are made of about 85 % of endosperm (which contains starch, protein, fiber and vitamins), 2 % embryo or germ (which contains fat, vitamins and minerals) and 13 % bark (which contains fibers, vitamins, minerals and proteins). The endosperm and therefore the flour, consists mainly of starch but contains also about 7 to 15 % of proteins.

These proteins (Faria *et al.*, 2004) can be divided into four groups: the water-soluble albumins (15 %), the globulins (7.5 %), the prolamins (gliadins - 32.5 %) and glutelins (glutenin - 45 %) (Street, 1991). The last two groups, that constitute the largest percentage of proteins of wheat, when combined with water and the supply of mechanical energy have

the property of forming a three-dimensional viscoelastic water insoluble network called *gluten*. The elastic membrane that forms the gluten has an important role in the structure, texture and therefore the quality of the final product (Bobbio and Bobbio, 1992).

3.1.1. Gluten

When the wheat flour and water are mixed together (Figure 3.3 (A) and (B)), a body formed by the gluten protein network associated with starch granules will be obtained (Figure 3.3 (C)). Gluten retains carbon dioxide produced during the fermentation process and causes the increase of product volume. Strong wheat flour has, in general, greater gas retention capacity, in poor flour, in turn, this feature is disabled (Guarienti, 1993).

The physical properties of gluten depend on both the wheat quality and the preservation of the flour obtained from it. Gluten toughness varies opposed to elasticity during dough production; while toughness diminishes over of time, elasticity increases, until they reach a point where both parameters decrease, rendering the dough more plastic (Almeida, 1997).

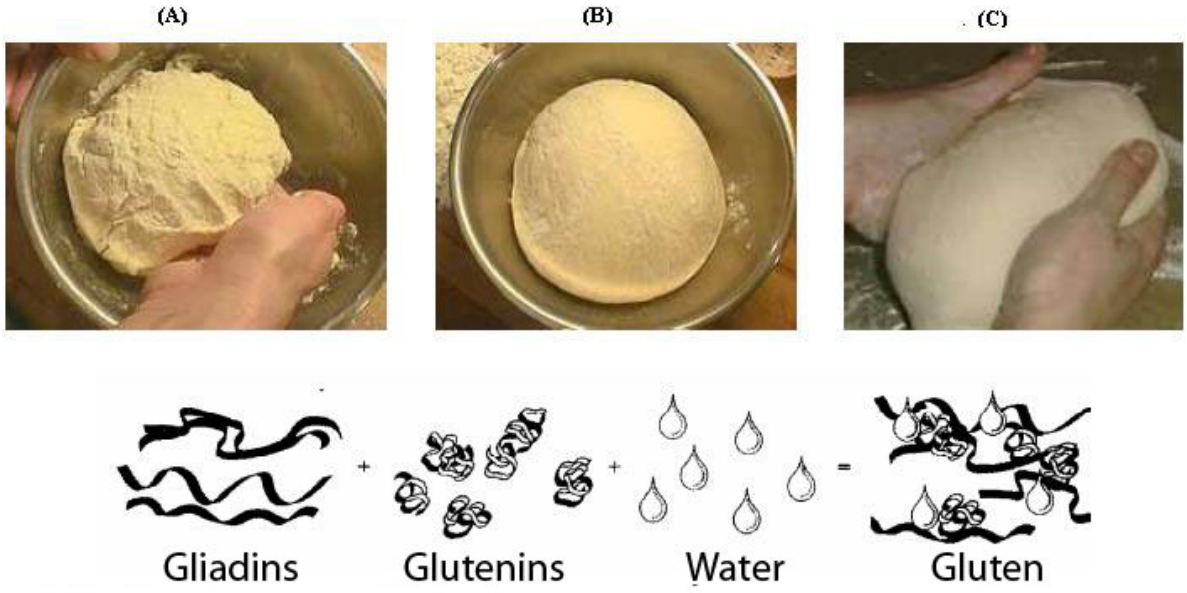

Figure 3.3: Gluten formation stages

3.1.2. Starch and other carbohydrates

Starch, which represents about 70 % of the flour, consists of branched (amylopectin) and linear (amylose) chains of glucose (Almeida, 1997). During baking of bakery products, amylase (found in flour) degrades these chains resulting in free sugars. According to Eynard *et al.* (1995), these components, even in small quantities, are relevant to the final product's texture (Almeida, 1997).

3.1.3. Lipids

The wheat flour contains approximately 2.2 – 2.9 % fat, which can be found in the starchy (0.8 – 0.9 %) and non-starchy (1.4 – 2.0 %) fraction of the flour. While the starchy lipids consist mainly of lisofosfolipids, non-starchy lipids belong to several classes (Hebeda and Zobel, 1996). The latter can be partially extracted with organic solvents and can be subdivided into an apolar fraction (triacylglycerols) and a polar fraction (phospholipids and glicolipids). The glicolipids and phospholipids are structural lipids and can be found in various membranes and organelles (Almeida, 1997).

Although lipids constitute only a small fraction of the total weight of the flour, their importance to the quality of the finished product is significant. The lipids play at least two different roles in the manufacture of the product: dough strengthening and tendering. It is known that part of the free lipids are linked firmly to the gluten (protein) when the flour is mixed in the dough (Hebeda and Zobel, 1996); this behaviour is certainly related with their roles in the process.

3.2. Eggs

The egg protein serves primarily as a means to incorporate air into the mixture, especially when egg yolk is separated from egg white and the latter is whipped and mixed at the end. Eggs are rich in fat soluble vitamins (see Table 3.1) and also provide liquid to the raw materials mix (Charley, 1987). The proteins of whole eggs or the egg white are used as "glueing" agents in order to thicken some products, providing structure and stiffening the mixture when the proteins coagulate (Hughes, 1995). When egg white is heated above a certain temperature it forms a strong gel due to the combination of hydrophobic interactions and dissulphite bridges. This imparts foods a "sponge-like" effect. While egg white proteins

are good gelling agents, egg yolk proteins are a rich emulsifiers source (Vaclavik *et al.*, 2003) due to the levels of lipoprotein (LDL). According to Hughes (1995), the addition of fats or sugars offsets the extra toughness of eggs in the formulation.

Dried eggs are often used in food preparations due to their microbiological safety and their reduced volume, compared with whole or liquid eggs. The use of dried eggs is generally associated with their convenience and long shelf. However, the quality of raw materials, processing and storage conditions, strongly influence the quality and safety of egg powder.

Table 3.1: Content of some fat-soluble vitamins in foods (Complete Book of vitamins and Minerals (2000). Publications International, Ltd.)

Foodstuff	Examples	Carotenes (µg)	Vitamins			
			A (µg)	D (µg)	E (mg)	K (mg)
Milk	Dairy milk	18	30	0.06	0.13	0.0003
Meat	Beef steak	-	-	0.02	-	0.008
Fish	Whitefish	-	7	1.1	0.4	-
Egg	Egg yolk	290	88	5.6	5.7	-
Vegetables	Spinach	4800	-	-	2.5	0.4
	Carrots	12000	-	-	0.47	0.015
	Tomatoes	590	-	-	0.81	0.006
Fruits	Orange	120	-	-	0.32	-
	Peach	400	-	-	0.97	-

3.3. Sugars

Sugars are the most important ingredients in the pastry products manufacture.

They have two main functions: (a) as yeast substrate during fermentation, and (b) to provide sweetness to each particular product (Kamel and Stauffer, 1993). Thus, sugars are added to foods as sweeteners, preservatives or suppliers of energy (Campbell *et al.*, 2002), while also increasing the volume of the product and contributing to the color of the crust as a result of the Maillard reaction. According to Pennington and Baker (1990), the contribution of sugar to the sweetness and flavor in products is well known. However, perhaps less known are the other functions, such as providing smoothness, moisture

retention, crust color formation, increasing shelf life, acting as fermentation substrate or simply as a preservative.

According to Charley (1987), during dough production sugar crystals are embedded in fat, while air is also introduced as it adheres to the crystals. For this purpose, the sugar must be in crystalline form to be more effective. Sugar also increases the temperature at which egg proteins coagulate during heating.

3.4. Baker's yeast

A wide range of yeasts are using when producing bread, fresh pasta, pizza, biscuits and other bakery products and pastries. Baker's yeast provide tastes and flavours which are not achievable when using chemical rising agents. However, in some foods, flavours from yeast fermentation may be undesirable. In industrial processes the use of baker's yeast is generally more expensive than using chemical agents, not only due to the higher cost of yeast, but also because yeast cells consume other materials (sugar) as a result of their activity and their action takes more time to be completed.

Although many microorganisms can ferment sugars generating carbon dioxide, *Saccharomyces cerevisiae* was elected for general use not only due to its fermentation abilities but also because of its GRAS status (Matz, 1992).

3.5. Fats

For food processing purposes fats are divided into yellow fats (butter or margarine), white fats (lard), vegetable fats and oils. Their chemical properties determine their state: either solid at room temperature or liquid (Booker *et al.*, 2004). This is an essential ingredient in many foods. Fats provide taste, texture and color to food products such as biscuits, cakes or pastries (Kamel and Stauffer, 1993).

3.6. Liquids

Liquids play important roles during dough production such as dissolving salts and sugars, dispersing fat and flour and hydrating flour protein and starch (Charley, 1987) thus contributing to gelatinize the starch and to establish the structure of food.

Many liquids may be used but the most popular are liquid water, fruit juices, milk or a mixture of water and milk solids. In particular, water is the component that triggers the fermentation process when mixed with yeast and flour; when used in food formulations it should be clear and free of chlorine.

3.7. Salt

Salt (sodium chloride) improves the plasticity of doughs and also contributes to increase their preservation by fluid retention, adjust the fermentation conditions and promote the generation of color and flavor (particularly at the later stages of baking, where it has an important function in crust formation).

3.8. Candied fruits

The addition of sugar to fruit is an ancient preservation method. Because it is used in a concentration above 50 %, it prevents microorganisms from degrading the fruits. Crystallization is achieved with a super-saturated sugar solution, which after heating covers the previously boiled fruit. The fruits are then subject to drying, acquiring a crystalline appearance. These fruits are used mainly in cakes decoration (Bennion *et al.*, 1997), such as the Portuguese “Bolo-Rei”.

The use of a wide range of raw materials in bakery products and pastries preparation, can create a problem in product quality if those materials are not carefully selected. This calls for adequate control procedures to ensure quality and safety of the final product.

4. Industrial Process

The conversion of all the ingredients listed in Table 3.2 in a final food structure, as shown in Figure 3.4, is made by a number of manufacturing operations (Tipple, 1975). According to Zghal and Scanlon (2001), the operations are directed so that the dough has appropriate mechanical properties to enable gas retention and consequently obtain a product with a well expanded and regular structure.

Table 3.2: Ingredients used in manufacturing of Portuguese traditional products: "Pão-de-ló", "Folar doce" and "Bolo-Rei"

Product	Ingredients
"Pão-de-ló"	Flour, eggs, sugar, baker's yeast and egg yolk
"Folar doce"	Flour, eggs, sugar, baker's yeast, margarine, salt, milk, cinnamon, fennel
"Bolo-Rei"	Wheat flour, eggs, sugar, baker's yeast, ground bread, egg yolk, butter, salt, lemon scraper, crystallized fruits or nuts, Port wine, "fava" beans

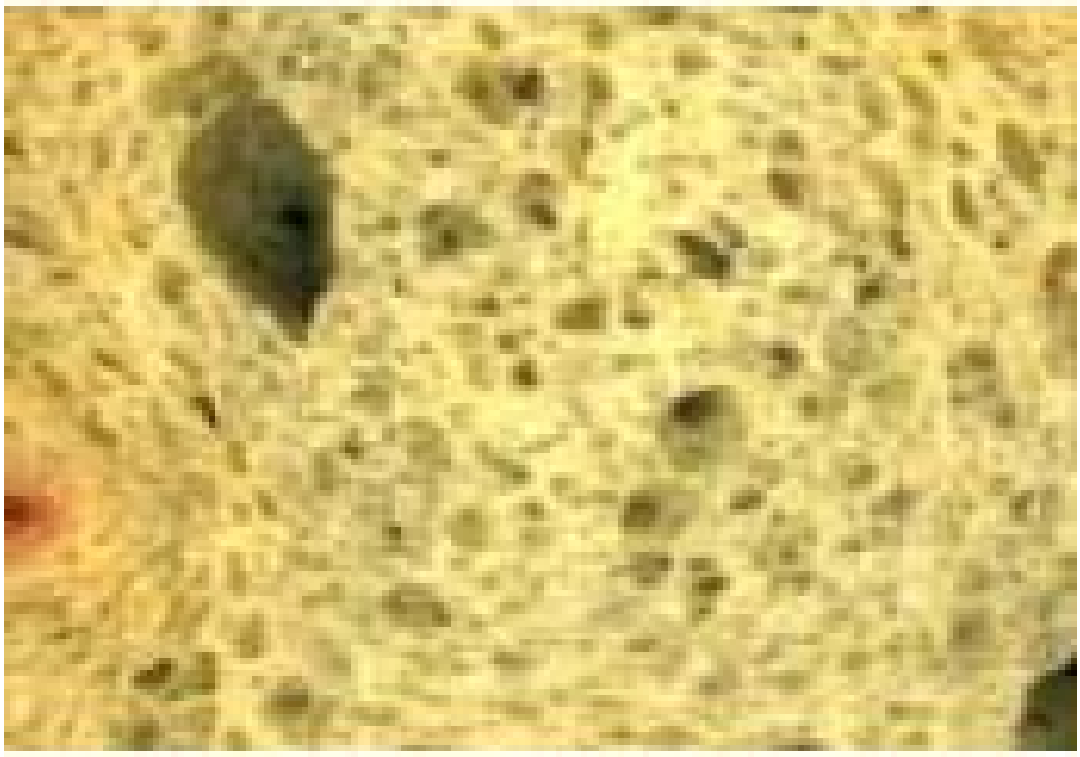

Figure 3.4: Digital image of a sample of bread prepared by the Chorleywood Bread Process (Zghal and Scanlon, 2001)

The production process involves three steps (Jiménez *et al.*, 2000):

1. Mixing and development of dough;
2. Dough fermentation;
3. Baking.

4.1. Mixing and development of the dough

The main purpose of mixing ingredients is to provide an uniform distribution of the components and the introduction of air (Charley, 1987). The creation of a porous and elastic structure in cakes is related to the movement that takes place while mixing the ingredients. According to Scanlon and Zghal (2001), the process of mixing is more than a homogenous dispersion of the ingredients: while the ingredients are mixed, air is occluded into the dough, starch gets hydrated and gluten proteins develop their action.

Fats and emulsifiers contribute to the stabilization of the incorporated air bubbles.

4.2. Fermentation

Sugars, such as glucose or fructose, are obtained from enzymes action on sucrose, maltose, lactose or other more complex carbohydrate molecules. Those sugars are consumed during fermentatin, as described in simple terms by equation 3.1:

$$C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2 \qquad \textbf{(Eq. 3.1)}$$

Carbon dioxide is formed which is responsible for the dough leavening (Figure 3.5), while ethanol helps in complex flavour formation in the product. A large proportion of these two compounds is lost during the stages of baking and cooling.

The enzymes that hydrolyze sucrose and maltose are present in yeast, but these organisms are unable to hydrolyze the starch directly to glucose. According to Matz (1992), amylases may be added to help with starch hydrolysis and there is also a small fraction of amylase activity naturally present in various flours. Sucrose is easily broken down by enzymes present on or in the yeasts cell wall, but the enzyme that digests maltose needs an activation period before becoming effective.

In parallel with alcoholic fermentation products several other substances are formed although in very small quantities. These are mainly aromatic substances that are not entirety

identified, but it is known that they consist of acids (like acetic acid), aldehydes and esters. Some of these compounds, which impart unwanted flavors, may react during baking and generate key components to the final product's flavour.

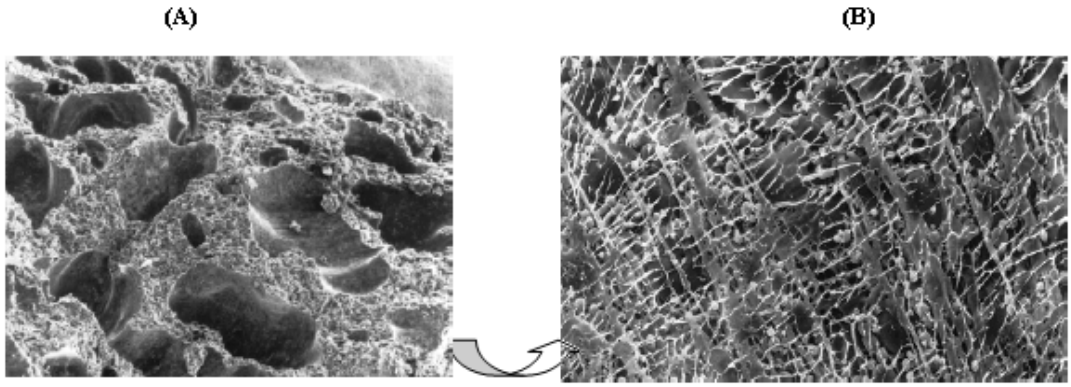

Figure 3.5: Structure of bakery and pastry products at the (A) start of fermentation and (B) after 2 hours of fermentation

4.3. Baking

According to Hui (1991), during baking, starch is transformed into a gelatinous material and proteins are denatured at a temperature of 60 to 80 ºC. Dough is therefore transformed into a crude, lightweight, porous and readily digestible product, (Jiménez *et al.*, 2000). In baking, according to Thorvaldsson and Kjjöldebrand (1998), the surface water content becomes lower than in the interior and this, combined with high temperatures, is one factor that imparts crust with its peculiar characteristics (Jiménez *et al.*, 2000).

The successive increase of dough temperature leads to the inactivation of yeast and enzymes, starch gelatinization and proteins modification (Esteller *et al.*, 2004). Finally, the crust is formed through chemical reactions of which the most important is the Maillard reaction. According to Calvel *et al.* (2001) these reactions are accompanied by the production of volatile compounds.

According to Hodge (1967), browning is the process by which colorless and sweet substances are transformed under the influence of heat into compounds that range from a yellow to a brown colour, creating a smooth and pleasant caramel, a taste of burnt, bitter and acid (Hui *et al.*, 2006). During browning, the brown color appears when the sugar reaches 436 K and this, together with the Maillard reaction, are the main causes of the bread crust color (Reinhart, 1998). In fact, according to Jiménez *et al.* (2000), sugars degradation requires more drastic conditions than the Maillard reaction, i.e., temperatures above 393 K,

pH under 3 or above 9 and low water activity. That is, when sugar is exposed to high temperatures for a long time, it suffers inside dehydration and forms compounds called melanoidins, which contribute to the brown color and final product appearance (Pennington and Baker, 1990).

The water content distribution and temperature are two important factors in the development of the sensory characteristics of this type of products (Jiménez *et al.*, 2000). Not all the compounds formed during browning were identified; however, the final products of these reactions are essentially unsaturated complex polymers (Hui *et al.*, 2006).

Jiménez *et al.* (2000) indicated that Maillard reaction, which is favored in foods with higher contents of protein and carbohydrates at a temperature of 323 K and pH values from 4 to 7, produces changes in color (melanoidins), flavour (aldehydes and ketones), functional properties and nutritional value (e.g. blocking or destruction of lysine).

Besides these changes, other factors influence bakery products' taste and flavour (Calvel *et al.*, 2001). These factors are:

- Flour protein and enzyme content;
- Dough composition;
- Oven type and operation temperature;
- The presence or absence of steam in the baking chamber;
- Baking duration.

All these factors have an effect on the crust, both in terms of its rigidity and the coloration degree, its dryness and its fragility.

The reactions described above, besides influencing favourably the final product's organoleptic characteristics, are also responsible for the formation of compounds identified as carcinogenic - acrylamide and 3 (2H)-furanones.

5. Formation of acrylamide in bakery products and traditional pastries

Model products made with original food industry recipes as well as the most similar conditions to those used in industrial production, are excellent tools to identify critical factors affecting acrylamide formation in bakery products and pastries, and have been widely applied (Amrein, 2005).

5.1. Influence of raw materials and formulation

In contrast with the potatoes (as seen in the previous chapter), the quantity of free asparagine is a critical factor in pastry and bakery, and is also correlated with the acrylamide level in the final product. The greater the amount of asparagine present, the greater the amount of acrylamide formed during processing of these foods (Ahrné *et al.*, 2007).

The flour is the main source of asparagine; however almonds, spices or honey can also contribute to increase the level of this aminoacid. Recently, the importance of selecting cereals with low levels of asparagine in order to reduce the final concentration of acrylamide formed during processing has been pointed out (Nunes, 2005).

The amount of asparagine in flour or dough depends on several factors:

- **Variety:** Rye flour usually contains higher levels of free asparagine than wheat flour. European flours have about 74 to 664 $mg.kg^{-1}$ of free asparagine .
- **Cultivation:** Fertilization, location, farming system and maturity may also influence the cereals' free amino acids and sugars concentration.
- **Flour type:** Improved extraction during milling, i.e. the inclusion of external parts of the seed, is related to the levels of free amino acids and sugars.
- **Fermentation:** Yeast and lactic acid bacteria consuming free amino acids during the dough fermentation may also influence their concentration.

The presence of free asparagine *per se* or its concentration alone do not have a direct relation with acrylamide concentration in bakery and pastry products. According to Amrein (2005) if reducing sugars are present (for example inverted sugar syrup can be replaced by a solution of sucrose), less acrylamide is formed.

5.2. Influence of the processing conditions

Long cooking and high temperatures increase the acrylamide levels in bakery products and pastries. In the work of Amrein (2005), after a long time and/or high baking temperature, there were higher acrylamide concentrations (often exceeding 1000 $\mu g.kg^{-1}$). Processing at lower temperatures can reduce acrylamide levels, but it may affect negatively the product sensory quality (Amrein, 2005).

In the past few years several efforts have been made to reduce acrylamide concentration in bakery products and pastries. As already mentioned, the main factors influencing acrylamide levels in these products are the contents of free asparagine and reducing sugars and the processing conditions.

In products where the brown color is not a parameter affecting the preference/choice of consumers, reducing sugars could be replaced by sucrose, a sugar that is associated with a decrease in the acrylamide levels. Moreover, the amount of free asparagine may be reduced through the use of flour with a lower degree of extraction and the rejection of products rich in free asparagine. Another possibility is the application of asparaginase to hydrolyze the amide groups of asparagine. This enzyme can be applied as pre-treatment of the flour or during the mixing process (Amrein, 2005).

The addition of tartaric or citric acid reduces the levels of acrylamide, but the application is limited due to the inhibition of "browning" and the acid taste imparted by those substances. However, large quantities are usually required and the impact on sensory and toxicological aspects is unclear.

6. Materials and Methods

6.1. Acrylamide extraction

The extraction of acrylamide from food samples was performed with water as the extractant, according to a combination of the optimum conditions cited in the literature (Wenzl *et al.*, 2003).

Five grams of each food sample were ground in a Waring blender (Worten, Portugal) for 3 min and thoroughly homogenized with 5 mL of distilled water. Another 10 mL of water were added and the homogenates were left to stand in a thermostated water bath set at 70 ºC under agitation. After 30 min the homogenates were centrifuged (12000 rpm, 20 min, 4 ºC) to allow precipitation and filtered twice through Whatman No. 2 filter paper.

The filtrates were transferred to 10 mL volumetric flasks and diluted with distilled water until volume completion. Afterwards, 1 mL of the sample solution were extracted 3 times with hexane (three portions of 3 mL) to remove remaining long chain fatty acids. These compounds may hinder the success of chromatographic analyses once peaks may overlap the target analytes or block the polar column. The mixture was shaken vigorously and the upper hexane layer was removed with a Pasteur pipette. 20 μL were taken with a Hamilton syringe directly from the lower aqueous layer and injected into the HPLC to be analyzed for acrylamide.

6.2. Acrylamide identification and quantification

The system consisted of an isocratic pump (PU 880, Jasco, USA) at a flow rate of 0.6 ml/min (mobile phase: sulphuric acid 0.005 M), an UV detector (870-UV, Jasco, USA) at a wavelength of 200 nm, and a VARIAN (USA) MetaCarb 87H column (300 × 7.8 mm) housed in an oven at a controlled temperature of 30 ºC. The system was associated with a software acquisition and data processing VARIAN STAR LC Workstation (USA).

A calibration curve with acrylamide standards at 0.1, 0.2, 0.3, 0.5, 1 and 2 $\mu g.ml^{-1}$ was built, as shown in Figure A.0.1 and Figure A.0.2 of the Appendix.

6.3. Tocopherols extraction, identification and quantification

The tocopherols quantification is usually made in oils (Cert *et al.*, 2000), and also in seeds (Manan, 1994), rice (Qureshi *et al.*, 2000) and cookies (Jamila *et al.*, 2004).

Tocopherols are extracted and saponificated before being quantified by HPLC. However, the saponification leads to losses of tocopherols during the process, even when cares such as the use of nitrogen and darkness are taken (Rupérez *et al.*, 1998), thus rendering this method not very effective. Many published studies have used direct analysis of tocopherols after diluting the oil in an organic solvent (Carpenter, 1979; Pocklington *et al.*, 1988; Tan *et al.*, 1989; Warner *et al.*, 1990; Gimeno *et al.*, 2000); in these methods sample preparation is just a collection, and no saponification step is involved.

A comparison between extraction with and without saponification has been performed by Manan (1994), who reported that the differences of the two methods in the case of seeds, are not significant. Therefore, in the present work the extraction of tocopherols was performed without saponification.

For the identification and quantification of tocopherols, separation may be achieved through various chromatographic techniques such as TLC, HPLC, both normal and reverse phase, among other chromatographic methods.

The most common method used for analysis of tocopherols is the direct analysis by HPLC using normal phase with detection by flurescence. Both fluorescence and UV detection are standardized by IUPAC and AOAC. There are also techniques for tocopherol detection in reversed phase HPLC columns. The analyses performed in this type of column have the advantages of a shorter time to reach equilibrium and a high reproducibility of the retention time, but it has the disadvantage of not differentiating between isomers β and γ. Normal phase allows a good separation of all isomers but the time of analysis is higher and the retention time is more variable. Analysis of tocopherols by GC usually involves saponification and separation by TLC (Cert *et al.*, 2000).

In the present work reverse phase HPLC was the method chosen to carry out the identification and quantification of tocopherols. The separation is based on the selectivity of the solvent, but can also be affected by column temperature and pH.

The extraction process followed was adapted from Gimeno *et al.* (2000), where methodologies for analysis in reversed-phase HPLC were published. In short, this method was based on an extraction with hexane, in which the chromatographic conditions were: eluent flow rate of 2 mL/min, temperature of 45 °C, mobile phase methanol:water 96:4

(v/v). The tocopherols were detected at a wavelength of 292 nm. In this study, we used a flow rate of 1 mL/min.

It is noteworthy that the extraction of tocopherols in bakery products is possible only if products containing tocopherols, such as oil or butter, are added, because bread in its traditional form shows no source of tocopherols. This means that quantifying these compounds in bread implies their incorporation in bread, or the use of sponge cake where they are known to be present in higher amounts.

6.3.1. Method for extraction of tocopherols

The extraction, identification and quantification of four types of tocopherols was sought (alpha-tocopherol, gamma-tocopherol and delta-tocopherol, all from Sigma Aldrich, while pure beta-tocopherol was not found on the market) and performed (except for beta-tocopherol) using the following reagents: n-hexane (Vaz Pereira, Lda., Portugal), methanol (Vaz Pereira, Lda., Portugal) and pure tocopherols.

The method used for extraction of tocopherols was as follows:

- Dissolve the sample (1 g in case of sponge cake or butter, or 1 mL in the case of oil) in 10 mL of n-hexane (put in a screw cap tube);
- Proceed with agitation for 30 min, using a magnet and a plate of agitation;
- Transfer to an Eppendorf already containing 600 μL of methanol 400 μL of the dissolved samlple;
- Subject the mixture to the vortex;
- Centrifuge for 5 min;
- After centrifuge, the samples are filtered through a filter with porosity of 0:45 μ m, using a syringe;
- Inject in HPLC.

The samples in glass tubes should be kept at temperatures below -20 ºC in dark environment, never more than a week.

6.3.2. Preparation of calibration curve

A calibration curve was built for each of the studied tocopherols based on five concentrations of the pure substance dissolved in n-hexane: 0.01 $mg.mL^{-1}$, 0.02 $mg.mL^{-1}$, 0.05 $mg.mL^{-1}$, 0.1 $mg.mL^{-1}$, 0.5 $mg.mL^{-1}$.

The corresponding curves are presented in the Appendix Figure A.0.3 and Figure A.0.4).

6.3.3. Tocopherols determination

Tocopherols determinations were performed by reversed-phase HPLC (Knauer, Germany), equipped with a Nucleosil ODS 5 column (Jones Chromatography, USA) of 150 mm of length and an internal diameter of 4.6 mm, coupled with either a fluorescence spectrophotometric detector (Knauer, Germany) or a UV detector (Knauer, Germany) at 292 nm. The eluent used was methanol:water (96:4, v/v) at an elution rate of 1 $mL.min^{-1}$. The injection was performed by means of an injection loop of 10 µL. The column temperature was set at 45 °C (achieved with an Oven 7971, Jones Chromatography, USA).

Elution profiles were shown in a computerized integrator Star Worksation (USA) (see Figure 3.6).

Unfortunately it has not been possible to obtain good results for γ-tocopherol determination; the peak was very weak (indicating a very low concentration of this compound) and it was not possible therefore to quantify it.

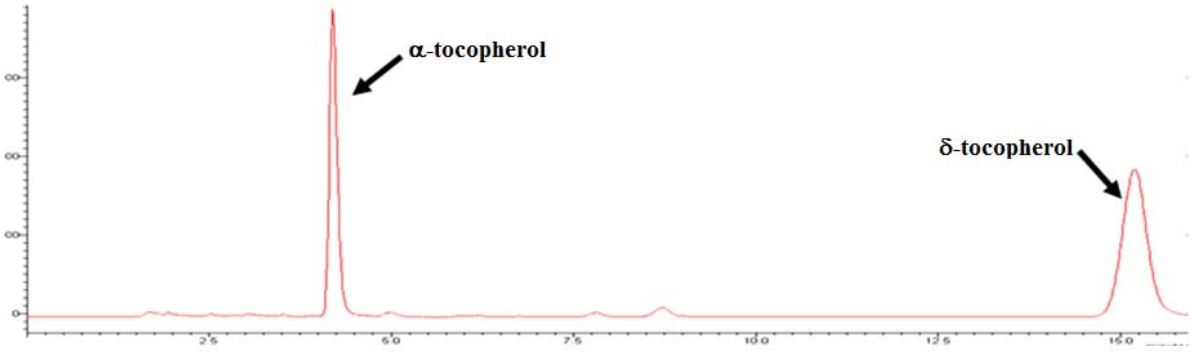

Figure 3.6: Example of HPLC chromatogram showing α- and δ-tocopherol peaks.

7. Results and Discussion

7.1. Degradation kinetics of α-tocopherol and δ-tocopherol in Pão-de-Ló

Pão-de-ló has in its formulation sugar, wheat flour, baker's yeast and eggs. In the present work this recipe was enriched with α- and δ-tocopherol standards to a final concentration of 10 $mg.g^{-1}$.The product was submitted to a baking process using oven temperatures ranging from 175 ºC to 225 ºC and processing times from 8 to 14 minutes. For each temperature samples were collected during the baking process to establish the degradation kinetics of both α- and δ-tocopherol.

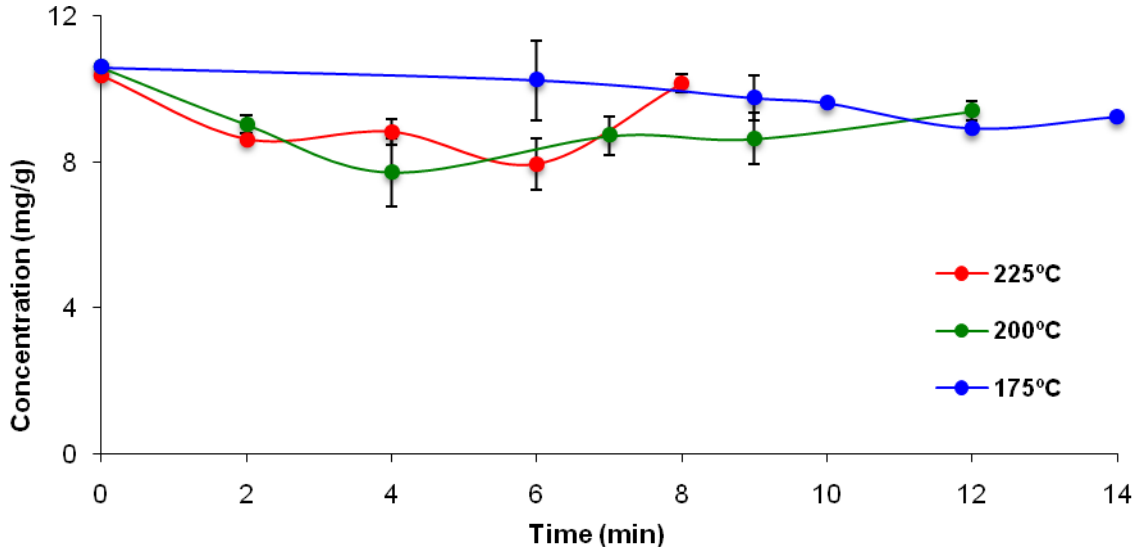

Figure 3.7: Degradation kinetics of α-tocopherol in Pão-de-Ló at different baking oven temperatures

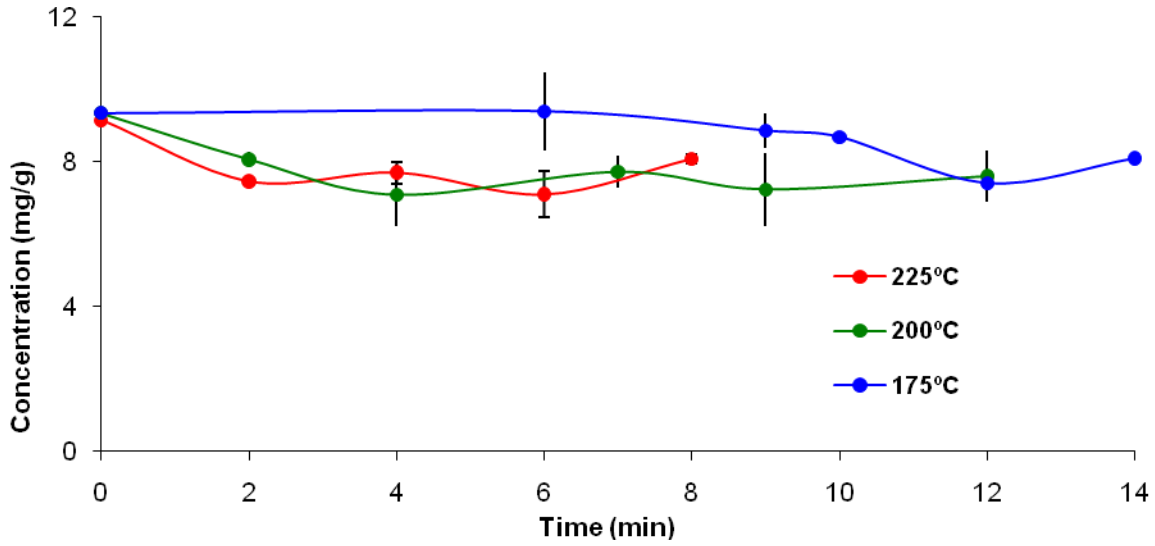

Figure 3.8: Degradation kinetics of δ-tocopherol in Pão-de-Ló at different baking oven temperatures

The results show that when using an oven temperature of 175 ºC tocopherols do not degrade during the baking process, although the baking time is clearly longer. The temperatures of 200 and 225 ºC have similar degradation kinetics, loosing in average 20 % of the concentration of both tocopherols quantified. This loss of vitamin E happens mostly in the first 3 minutes of baking.

7.2. Quantification of α-tocopherol and δ-tocopherol in flours from different cereals

Although supplementation with tocopherols is feasible, it is important to know at which levels are they present in the major raw-material.

The analyses of the different commercial flours reveal that they have, approximately, 4 times higher content in α-tocopherol than in δ-tocopherol (the exception being the rice flour, that presents similar quantities of both tocopherols – see Figure 3.9. Corn flour turned out to be a good base ingredient for enriched baking products due to the comparable (and high) concentrations of both tocopherols.

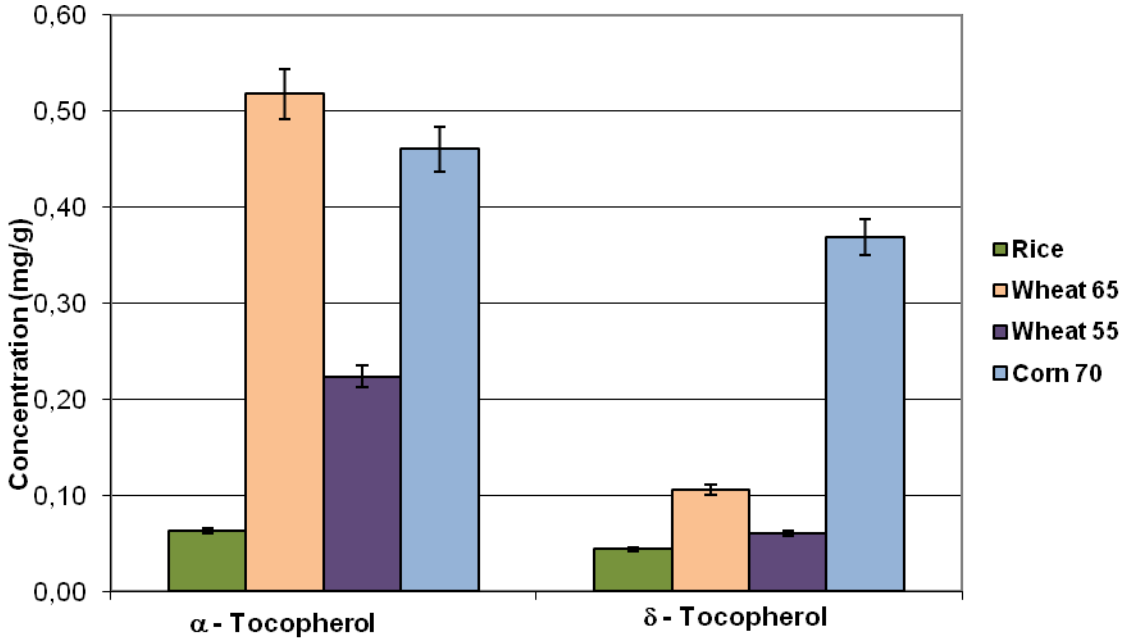

Figure 3.9: Quantification of α- and δ-tocopherol in several flours

7.3. Quantification of α- and δ-tocopherol in several baked products

In order to benchmark the amounts of tocopherols in several commercially available products, traditional corn bread, "bijou" bread, mold bread and rice cake were tested for the amount of tocopherols following the same experimental procedure used for Pão-de-Ló. The distribution of tocopherols in the different parts of the product was also evaluated.

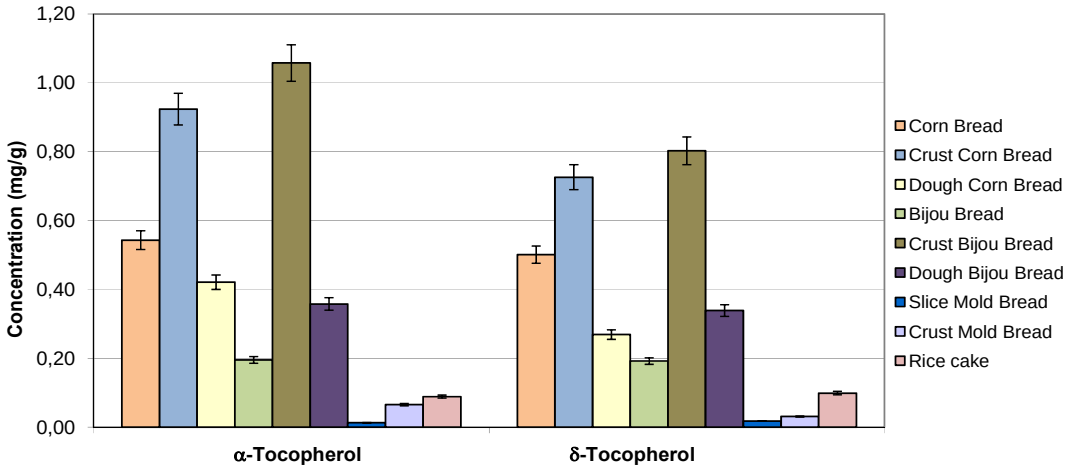

Figure 3.10: Quantification of α- and δ-tocopherol in different parts of several baked products

Figure 3.10 clearly shows that the content in α- and δ-tocopherols is approximately the same in the different tested products. Exception is for the crust of corn bread and the crust of "bijou" bread where the content of α-tocopherol is higher than that of δ-tocopherol.

The amounts of tocopherols are consistently higher in the crust of the products. Given that it has been demonstrated that baking temperature is not affecting tocopherols concentration at a great extent, these results may be explained by the concentration effect that the dough suffers at this location during the baking process.

7.4. Acrylamide in bakery products

Bakery products, such as French fries, are considered as acrylamide-rich products. The association of the chemical composition of these products, rich in sugars and amino acids, with the high processing temperatures to which they are subjected, creates an ideal setting for the formation of this carcinogenic compound.

These foods undergo a series of physical, chemical and/or microbiological changes throughout their shelf-life. The purpose of this part of the work was to examine whether there are also changes in the concentration of acrylamide during the shelf-life, since this is a dangerous compound with carcinogenic effects in humans.

For industrial "Pão-de-Ló", "Folar Doce" and "Bolo-Rei" acrylamide concentrations are shown in Figure 3.11. All products are in the same range of values, i.e. above 1000 $\mu g.kg_{product}^{-1}$ (according to Amrein (2005), this values are high concentrations). For "Pão-de-Ló", "Folar Doce" and "Bolo-Rei" the average concentrations of acrylamide were 1770.22 ± 704.19 $\mu g_{acr}.kg_{"Pão\text{-}de\text{-}Lo"}^{-1}$, 1472.40 ± 295.60 $\mu g_{acr}.kg_{"Folar\ Doce}^{-1}$ and 1198.96 ± 229.30 $\mu g_{acr}.kg_{"Bolo\text{-}Rei"}^{-1}$, respectively. For these three bakery products, no statistically significant change of concentration of acrylamide was found (p = 0.243). The high levels of concentration of acrylamide can be justified by the presence of several factors that promote acrylamide formation, such as a high content of sugars and asparagine, high processing temperature (Grob *et al.*, 2003) and an initial moisture content of about 30 %, which accelerates Maillard reaction and therefore acrylamide formation (Wicklund *et al.*, 2006) in bakery products. Throughout shelf life there was no statistically significant change of acrylamide concentration in these products (p = 0.267 for "Pão-de-Ló", p = 0.800 for "Folar Doce", p = 0.933 for the "Bolo-Rei").

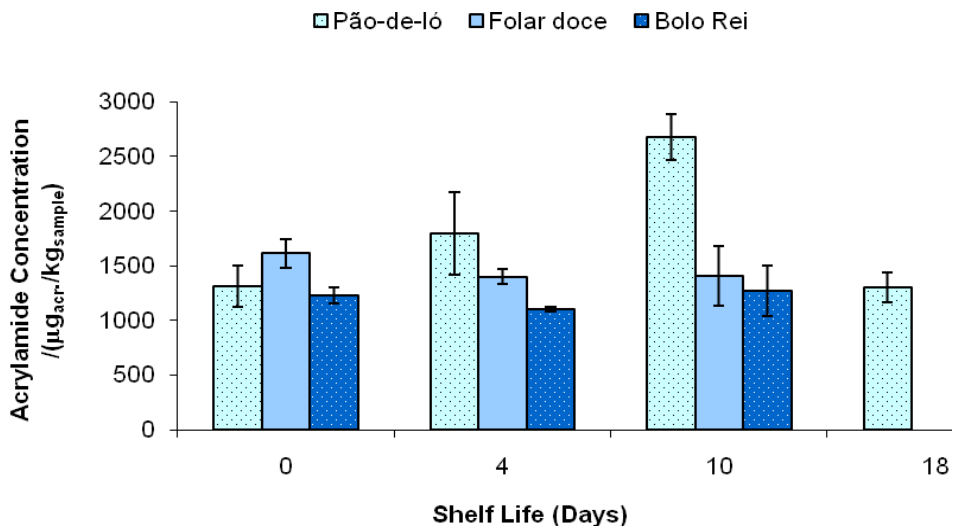

Figure 3.11: Acrylamide concentration through the shelf-life of bakery products

The next step was to evaluate this variation in the "Pão-de-Ló" when baked at three different temperatures (185, 200 and 225 ºC). Results are shown in Figure 3.12.

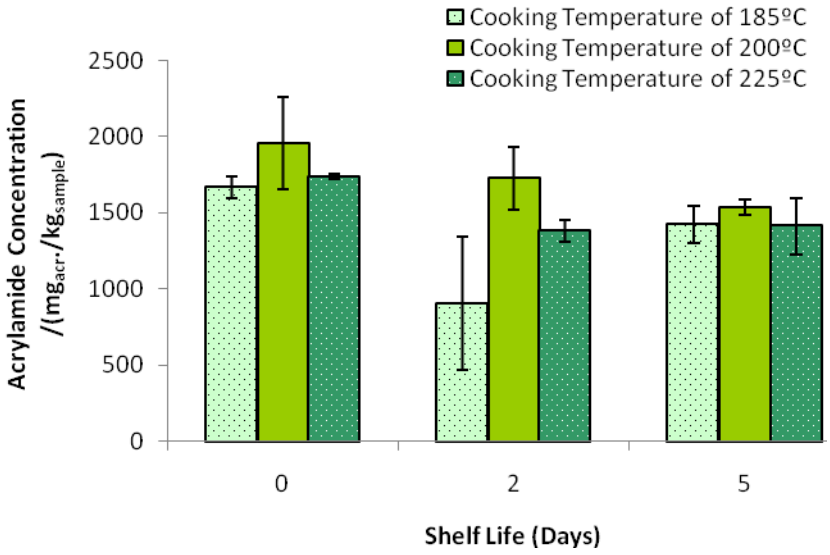

Figure 3.12: Variation of acrylamide concentration throughout shelf life of "Pão-de-Ló" baked at three different temperatures

"Pão-de-Ló" baked at temperatures of 185, 200 and 225 °C displayed acrylamide concentrations of 1538.78 ± 185.06 $\mu g_{acr}.kg_{"Pão\text{-}de\text{-}Lo"}^{-1}$, 1738.75 ± 380.43 $\mu g_{acr}.kg_{"Pão\text{-}de\text{-}Lo"}^{-1}$ and 1509.36 ± 250.44 $\mu g_{acr}.kg_{"Pão\text{-}de\text{-}Lo"}^{-1}$, respectively; no statistically significant difference could be found between these values (p = 0.243). The relatively high standard deviations obtained were the main obstacle to the observation of a trend, if there would be one. As the

raw materials used in the formulation of these products were the same, the factor that may have influenced the difference in concentrations (though statistically not significant, as noted before) is surely the temperature in agreement with Amrein (2005), which states that prolonged cooking and high temperatures increase the level of acrylamide in bakery products. The fact that the average concentration of acrylamide in the "Pão-de-Ló" up to a temperature of 225 ºC is less than that which was cooking temperature of 200 °C, can be justified according Ahrné *et al.* (2007), who claimed that at high temperatures and low humidity, the concentration of acrylamide decreased. However, these authors studied temperatures well above acceptable values (about 260 ºC), which would render the product unacceptable for consumption, due to its final sensorial characteristics. Throughout the shelf-life of "Pão-de-Ló" produced at the three studied temperatures (185, 200 and 225 °C), no statistically significant changes were observed in acrylamide concentration (p = 0.400 at 185 ºC, p = 0.800 at 200 °C and p = 0.333 at 225 ºC).

The results obtained here point at the conclusion that shelf-life is not influencing the formation/degradation of acrylamide in bakery products and that this is primarily attributable to the composition and processing conditions of the products.

8. Conclusions

The results suggest that though oven-baking has a tendency to decrease the concentration of tocopherols, this loss is generally not significant. For all tested temperatures, the loss of vitamin E happens in the first 3 minutes of baking and cannot be considered relevant.

Concerning the analyses of the different commercial flours tested it was found that they have, approximately, 4 times higher contents in α-tocopherol as compared to δ-tocopherol. In the case of rice flour, the contents in both α-tocopherol and δ-tocopherol are extremely low.

Different contents of α- and δ-tocopherols were also found in different traditional bakery products and in different parts of those products: higher α-tocopherol concentrations were registered in corn bread, in the crust of corn bread and in the crust of "bijou" bread than in corn bread dough and "bijou" bread dough.

When evaluating the evolution of acrylamide concentration during shelf-life of industrial "Pão-de-Ló", "Folar Doce" and "Bolo Rei", it was concluded that there is no significant changes: 1770.22 ± 704.19 $\mu g_{acr}.kg^{-1}$, 1472.40 ± 295.60 $\mu g_{acr}.kg^{-1}$ and 1198.96 ± 229.30 $\mu g_{acr}.kg^{-1}$ for industrial "Pão-de-Ló", "Folar Doce" and "Bolo Rei". The same conclusion was drawn for samples of "Pão-de-Ló" baked at 185, 200 and 225 °C, registering acrylamide concentrations of 1538.78 ± 185.06 $\mu g_{acr}.kg^{-1}$, 1738.75 ± 380.43 $\mu g_{acr}.kg^{-1}$ and 1509.36 ± 250.44 $\mu g_{acr}.kg^{-1}$, respectively.

Future work should consider monitoring levels of precursors of acrylamide in bakery products and pastries during their processing, in order to correlate acrylamide formation with changes in those levels. This might provide an *a priori* indication of the potential of acrylamide formation in a given product (assuming constant processing conditions). On the other hand, knowing that the efficiency of acrylamide formation depends on the type of sugar used, it would be interesting to develop products with different sweeteners/sugars and test their acrylamide formation potential.

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Chapter 4.

PANIGEST® - BAKERY MANAGEMENT SOFTWARE

Abstract

Food safety has become an important food quality attribute. Both food industry and authorities need to be able to trace back and to authenticate food products and raw materials used for food production to comply with legislation and to meet the food safety and food quality requirements.

PaniGest® is a user-friendly computer package designed to manage traceability and help in the quality control and production improvement. This application was developed in Visual Basic language over an SQL database and its main features are: to register quality control parameters of raw materials, in-course products and final products; to manage reception, production and expedition orders; to analyse production costs, productivity, raw materials and products' consumptions; to trace products during the food chain. It runs on a personal computer over Windows Vista or Windows 7 operating system. The program also uses common Internet Browsers to make information available to users.

1. Introduction

In the recent years, new policies regarding food safety and food safety management were adopted by Governmental Authorities and Food Industry as a consequence of several food incidents and scandals. These incidents caused serious loss of confidence of the consumers that started to demand for high quality food, food integrity, safety guarantees and transparency. To answer these consumers' requirements, quality assurance has become a cornerstone of food safety policy in the food industry that started to implement integrated quality and food safety management systems. These systems include all steps in food production chain namely the supply of raw materials, food manufacturing, packaging, transportation and logistics, research and development, maintenance of production equipment and training and education of staff. Increasingly, food quality is associated with a proactive policy and the creation of requirements to maintain a safe food supply (Folstar, 2001).

Global food safety policies were adopted by Governments and a new series of regulations were created and adopted all over the World, with particular incidence in EC (European Community). One of the concepts introduced by these new legal documents was traceability. EC/178/2002 defines traceability as the ability to trace and follow food, feed, and ingredients through all stages of production, processing and distribution. This regulation is applied to all food industry. The Regulation contains general provisions for traceability which cover all food and feed, all food and feed business operators, without prejudice to existing legislation on specific sectors such as beef, fish, GMOs, among others. The requirement for traceability is limited to ensuring that companies are, at least, able to identify the immediate supplier of the product in question and the immediate subsequent recipient, with the exception of retailers to final consumers (one step back/one step forward). Traceability is the ability to track back a product and its history through the whole, or part, of a production chain from harvest through transport, storage, processing, distribution and sales or internally throughout the production stages. Traceability is a generic issue, as its fundamentals are independent of the type of product, production and control system it serves (Kim *et al.*, 1995).

Traceability and Food Safety Management systems can work properly based on pen and paper versions but they are time and resource consuming which makes them difficult to implement in small and medium companies where the resources are scarce.

Moreover, the International Standardization Organization (ISO) also started to work on the adaptation of the Quality Management Systems standard to the food industry ISO 22000, which is was published in 2005. ISO 22000:2005 was designed to cover all the processes along the food chain that deal directly or indirectly with the end product being consumed. Furthermore, it specifies the requirements for food safety management system by incorporating all the elements of Good Manufacturing Practices (GMP) and Hazard Analysis Critical Control Points (HACCP) together with a comprehensive management system. This standard is made up of eight core elements, detailed below.

The use of new technologies of information and communication to support and facilitate the practical implementation of these complex systems is very recent and, until now, it can be found only in larger food production units. The development of adequate computer packages to reduce the paperwork involved in the management system can be extremely helpful for SME's.

In the baking industry, the traceability process is very complex due to the diversity of raw materials used and the large number of different products that a single batch can generate. Moreover, at the end of production process, there are several finishing raw materials used in the product that, usually, are not controlled or even traced back to the supplier. Therefore, the development of a computer application, using a user-friendly interface, specially designed for small and medium food companies, was pertinent. PaniGest® was created to respond to these needs integrating some legal requirements such as traceability with quality control, production management, raw material and finished products stock control, consumptions, production costs and HACCP systems.

2. Materials and Methods

2.1. PaniGest® Database Development

Planning the database structure is one of the most important tasks when developing a computer application once all the information required will be stored in and related by the database. Depending on the model used, the information can be related in different ways. Early models included the hierarchical model (where files are related in a parent/child manner, with each child file having at most one parent file), and the network model (where files are related as owners and members, being that each member file can have more than one owner). Relational databases offer many advantages over unstructured forms of data storage (Codd, 1970 and Harrington, 2002).

Through the use of indexable variables and other optimizing devices, speed gains for searches may be considerable. Moreover, redundancy and therefore storage space are minimized. Also, data is handled by a single computer which is easily backed up and/or mirrored by a second computer (Bradley *et al.*, 2004). Another important advantage of relational databases is the ability to rapidly summarize data with a small number of commands, usually using the structured query language (SQL). With a few of these commands, one rapidly obtains a comprehensive overview of a data set, something that could, otherwise, take many hours programming. But, perhaps the most important advantage of relational databases is that they impose a consistent data format (Bradley *et al.*, 2004).

The major issue in database development is to find a database structure that is broad enough to be applicable in several industries and, at the same time, adequate to consistently structure data. The relational database model was a huge step forward, because in order to relate any two files or records, they simply need to have a common field, which makes the model extremely flexible. The relational database model, being a table-based structure, naturally groups data conditions according to type or product. For example, there might be tables containing lists of products, raw-materials, users, formulations and quality control data. One or more additional tables would store production data, and a final table would serve as a hub linking production data to product and raw-material data.

By means of cross references, the complete traceability of raw-materials to final clients as well as users actions associated with any production could be derived from the database, without reference to external sources.

The production stages and the records needed during the production process under analysis (baking process) had to be studied in detail in order to develop the database that records all the relevant information to achieve total traceability of the product and, simultaneously, manage paperwork. Figure 4.1 presents the production flowchart that was used to develop the database.

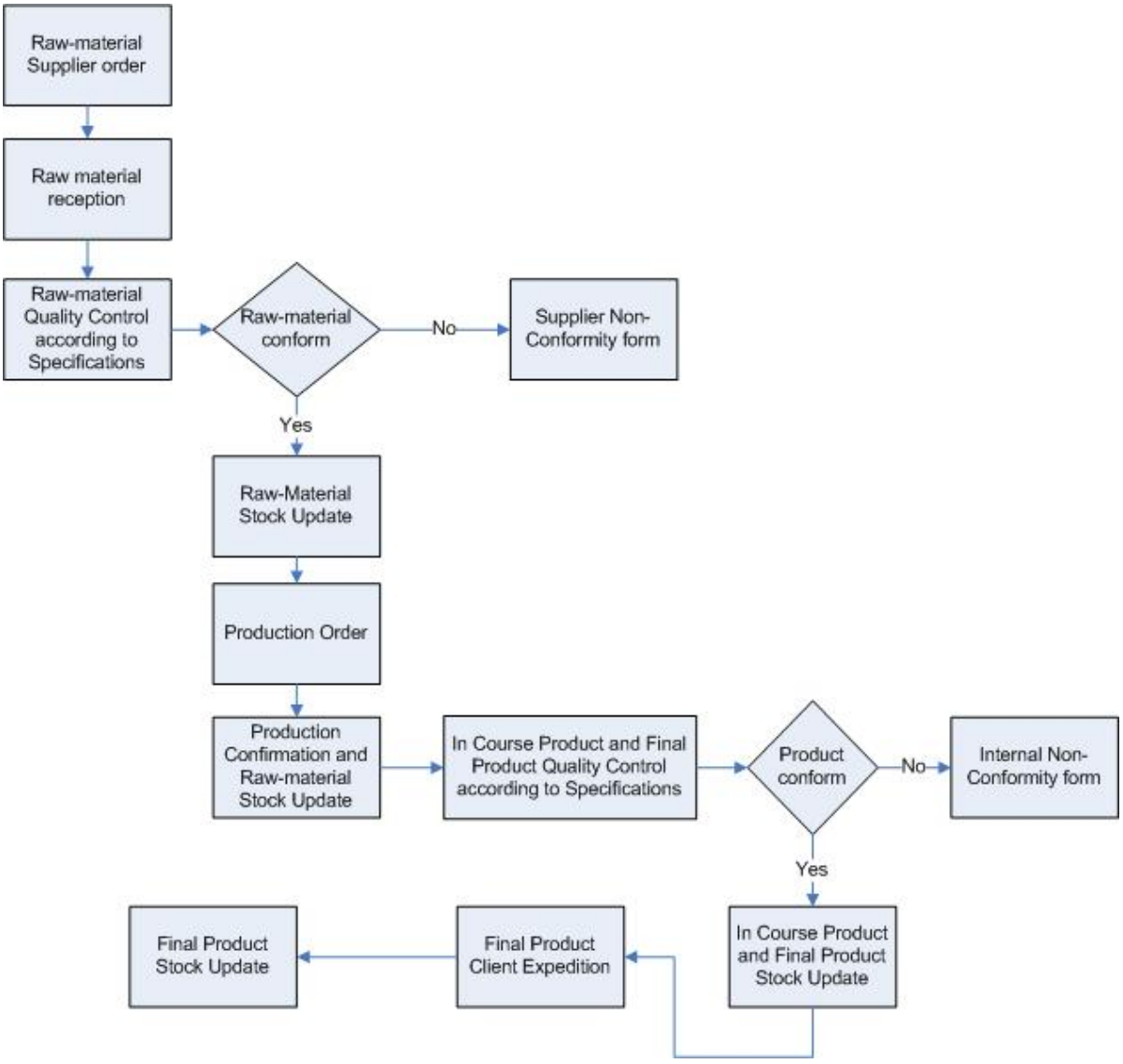

Figure 4.1: Baking Process flowchart

The conclusions of the production flowchart analysis were that the traditional baking industry produces a wide range of products (bread and cakes), ready to be sold, based on the same batch of ingredients. This process becomes even more complex when more than one of those batches is made during the day. Moreover, the difficulty increases during expedition of final products to the client because different ready to sell products originating

either from the same batch or from different batches are very often delivered to the same client and, in many cases, the client is the final consumer.

The developed database is a relational database and Figure 4.2 exemplifies some of the multiple relationships established between tables.

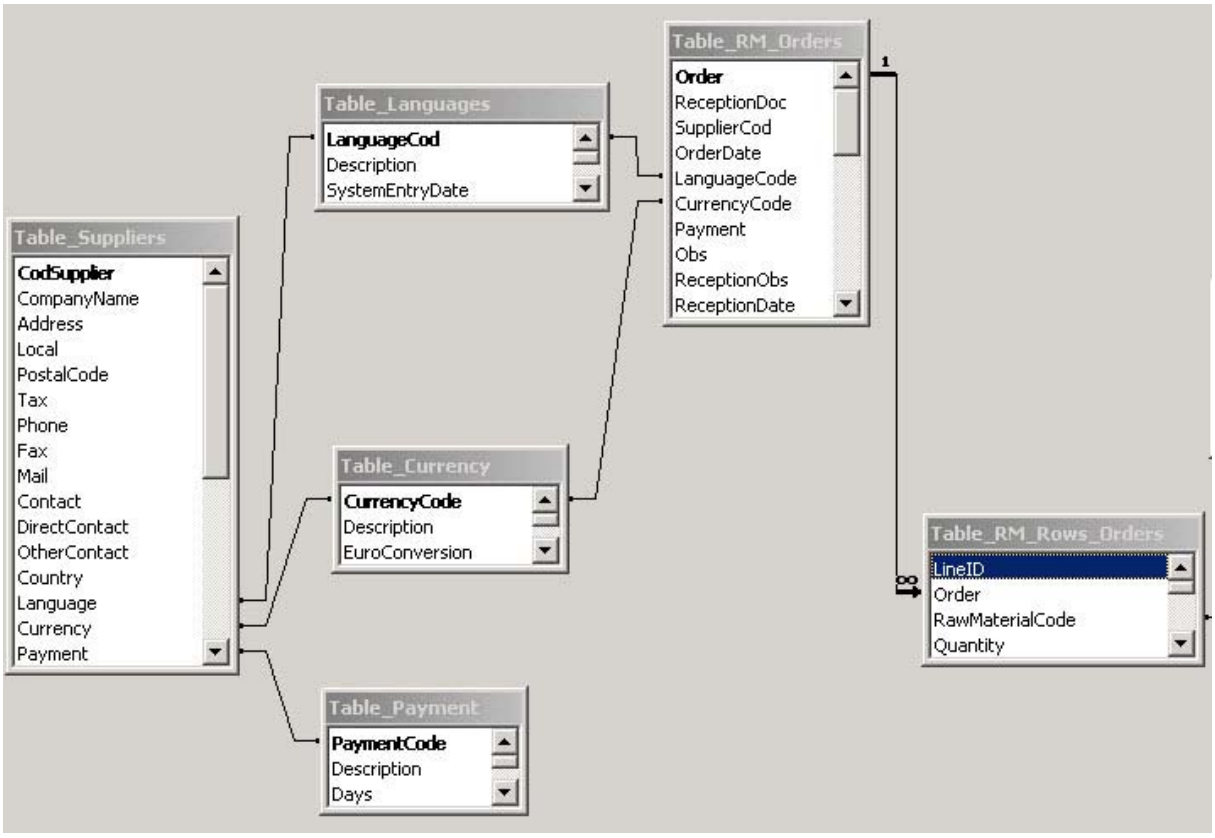

Figure 4.2: Database relationships.

For example, the field "Order" in "Table_RM_Orders" table is related with the field "Order" in "Table_RM_Rows_Orders" table. In this particular example, the first table should hold suppliers order data and the second table records the multiple lines of the supplier's order. The type of relationship is a one-to-many relationship because a purchase order could have multiple lines of different products. Multiple relations such as that described above are developed and implemented in this database.

Initially, the database was developed in Microsoft Access and then was updated to SQL Server. The Access version of the application was maintained in order to reach clients that are unable to install in their units an SQL Server. There are several advantages when using a SQL Server database instead of a Microsoft Access database.

For example, some of the codes written in SQL language, used to query the database, can be integrated directly into the SQL Server database using Store Procedures thus making the application lighter. Also, if the Client suggests some program changes, it is much easier

to update the Database without making another program executable (file extension .exe). This makes the file lighter and if there is the need of change concerning queries to database it is simpler and there is no need of re-designing the program code. Another important advantage is data security, integrity and user access control, which are considerably better in a SQL Server Database.

2.2. Interface Development

PaniGest® is intended to be used by several departments within the Organization thus is extremely important that the interface is user-friendly.

Those computer programs packages were developed using Visual Basic 6.0™ by Microsoft® under a Microsoft® Windows® operating system (e.g. Windows Vista or Windows 7). A comprehensive, rapid application development environment, Visual Basic helps developers to quickly create and deploy client/server applications, and to program easily for the Internet using familiar Visual Basic programming tools and techniques.

The application also uses commonly known ActiveX controls and some Dynamic Link Library (DLL) from Microsoft. A DLL is a library for applications within the Windows operating system as well as for programming and contains one or more functions that are compiled, linked, and stored separately from the applications using them (Takeuchi, 2002). However to support some of the present application's functionalities several DLLs had to be developed.

2.3. Error Handling

One of the major concerns in software development is the error treatment and handling. PaniGest® error handling concerned two major types of errors: the common and usual user errors (e.g. typing letters in instead of numbers) and the database operations errors. For the database operations errors the transaction methodology was used.

Being so, in any database operation, such as inserts, updates and deletes, the program executes all the code and then verifies if an error has occurred; if there are no errors, the transaction is committed, otherwise it is rolled back. The main advantage of this methodology is that it assures data integrity, avoiding the insert, update or delete of partial data. This is commonly known when the SQL instruction is made over two or more different database tables. For example, in Figure 4.2, the field "Order" in

"Table_RM_Orders" table is related with the field "Order" at "Table_RM_Rows_Orders" table. As referred above, the first table holds suppliers order data and the second table records the multiple lines of the supplier's order. Using the transaction technology when changing a raw material order, for example, there is no risk at all of updating the table that holds supplier data and not updating the table that holds the multiple product order lines. It either updates everything or it does not update anything at all.

This type of code is written as in the following example where cpcOT is the variable that stores the link to the database. If an error occurs while executing the SQL instruction, the program jumps to the "Erro:" function and stops and rolls backs the SQL instruction that was already executed. This is demonstrated in the following error handling code example:

```
On error goto ErrorHandler
    cpcOT.BeginTrans
    SQL Instruction
ErrorHandler:
    If Err = 0 Then
            cpcOT.CommitTrans
            Err.Clear
    Else
            cpcOT.RollbackTrans
            Err.Clear
            Exit Sub
    End If
```

Another important measure to control errors is the implementation of an error sub-routine in any user action. If an error occurs a message to the user is generated. In background, the application records the occurred error in the database table for future analysis and correction. This is of extreme importance because it allows the authors to implement corrective actions in future updates of the application. Moreover, when a connection to the Internet exists, error messages can be sent to the author (by e-mail) for immediate analysis. In some cases it is possible to implement corrective actions through Internet connection.

2.4. Outputs

Considering the application of this computer package to Food Safety Management Systems it was fundamental to create easy-to-read output documents or pre-defined reports. Seagate Crystal Reports was used to develop all the output documents. To access the database, Seagate Crystal Reports uses an Open Database Connectivity (ODBC) connection. ODBC is a widely accepted application programming interface (API) for database access. It is based on the Call-Level Interface (CLI) specifications from X/Open and ISO/IEC for database APIs and uses Structured Query Language (SQL) as its database access language. Data Sources ODBC is used to access data from a variety of database management systems.

Another important feature is the browser access to the information which makes it possible to query important information such as product specification, using a simple computer with Internet browser installed.

3. Results and Discussion

3.1. PaniGest®

PaniGest® main features are: to register quality control parameters of raw materials, in-course and final products; to manage reception, production and expedition orders; to analyze production costs, productivity, raw materials and product's consumptions; to define and print labels containing lot information and other relevant data; to implement traceability back and forward in the food chain (from raw materials to the final client).

In order to maintain total traceability (back and forward) different types of internal lots were implemented (autonumbers): the internal lot for raw-material; the lot of finished products and the daily production reference. The use of these auto-numbers (internal lots) will be explained further in the text. When the application starts, it opens its main form where the user can access multiple options and menus. Several of those options and menus correspond to table management (example: VAT codes). These data, introduced firstly in the program, are indispensable for the subsequent actions. Other options and menus, explored further ahead, are the operation and control options and menus, such as the raw-material section, production section, quality control section and final product section.

A sample code of PaniGest® is presented in Appendix.

3.1.1. The main form

The requirements for software vary according to the goals of its users. In the food industry the level of computer skill and computer access varies widely among users, and this is usually related to their responsibilities in the whole process. These responsibilities are even more important when a safety and quality management system is implemented and maintained within the organization. For all users it would be more effective if the computer program interface could be changed according to the user's level of computer proficiency and authorization level.

For novice users, complicated input and output options are confusing and often result in users missing the objective of the program, and also may result in errors in operation. Dismissing unnecessary options and simplifying the interface, therefore, would be of great benefit to this type of users. Using graphics, such as charts, figures, and diagrams to represent the results of analysis is often more effective than a numerical description. Also

different responsibilities related to filling down registries or validate them; release orders, recall procedures and so on are established within an organization with a defined hierarchy related to the existing quality management or HACCP systems. Being so, the software was developed using a friendly interface to all users and with user-level authentications (Figure 4.3).

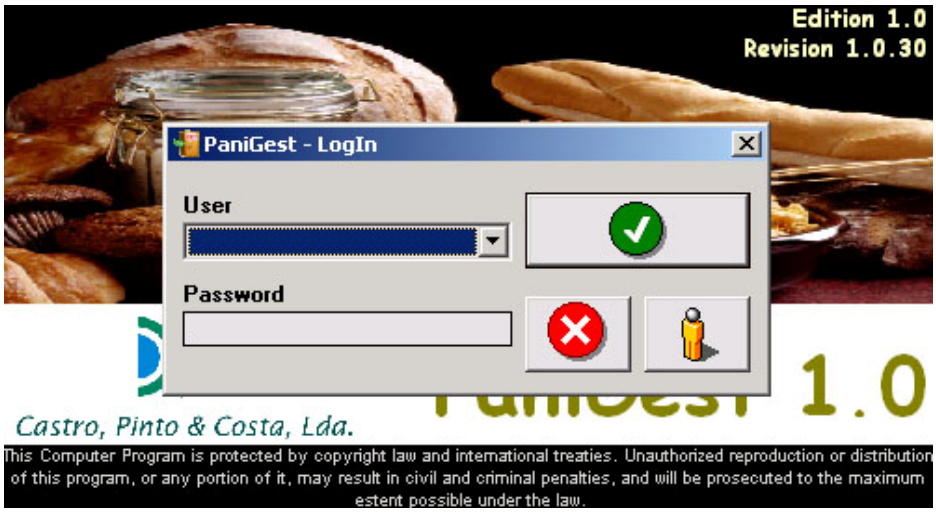

Figure 4.3: User log-in

It also allows multiple users to access the program simultaneously. However, a safety measure was implemented during development: it is impossible to use the same user name simultaneously in different workstations. After entering the user name and password, the software executes a Store Procedure with a database query to upload the user permissions and validate the user log in. A DLL was developed to automatically import user and computer name from the Windows Operating System. This DLL is of extreme importance not only because it limits access to the users but also because it can be used as a "signature". With this feature, it is possible to audit all the users operations and to determine responsibilities.

After the user log-in, the main form (Figure 4.4) contains a lateral menu with the different sections of the computer package: Raw-materials; Production; Quality Control; Finished Product; Clients Definitions; Tables; Utilities; Lists and Software Configuration. This lateral menu was made using an OCX control called vbaccelarator Explorer Bar Control available at the vbaccelarator web site for download. It can also open a "Status Form" showing the actual status of supplier's orders, production orders, raw-materials expiration dates and current stocks. All the options available in the lateral menu also exist at the upper menu of the main form.

Inside each main menu sections there are sub-menus opening inside the main form (MDI form). This type of construction allows the left menu to be always available. All sub-forms of the program open inside the main form.

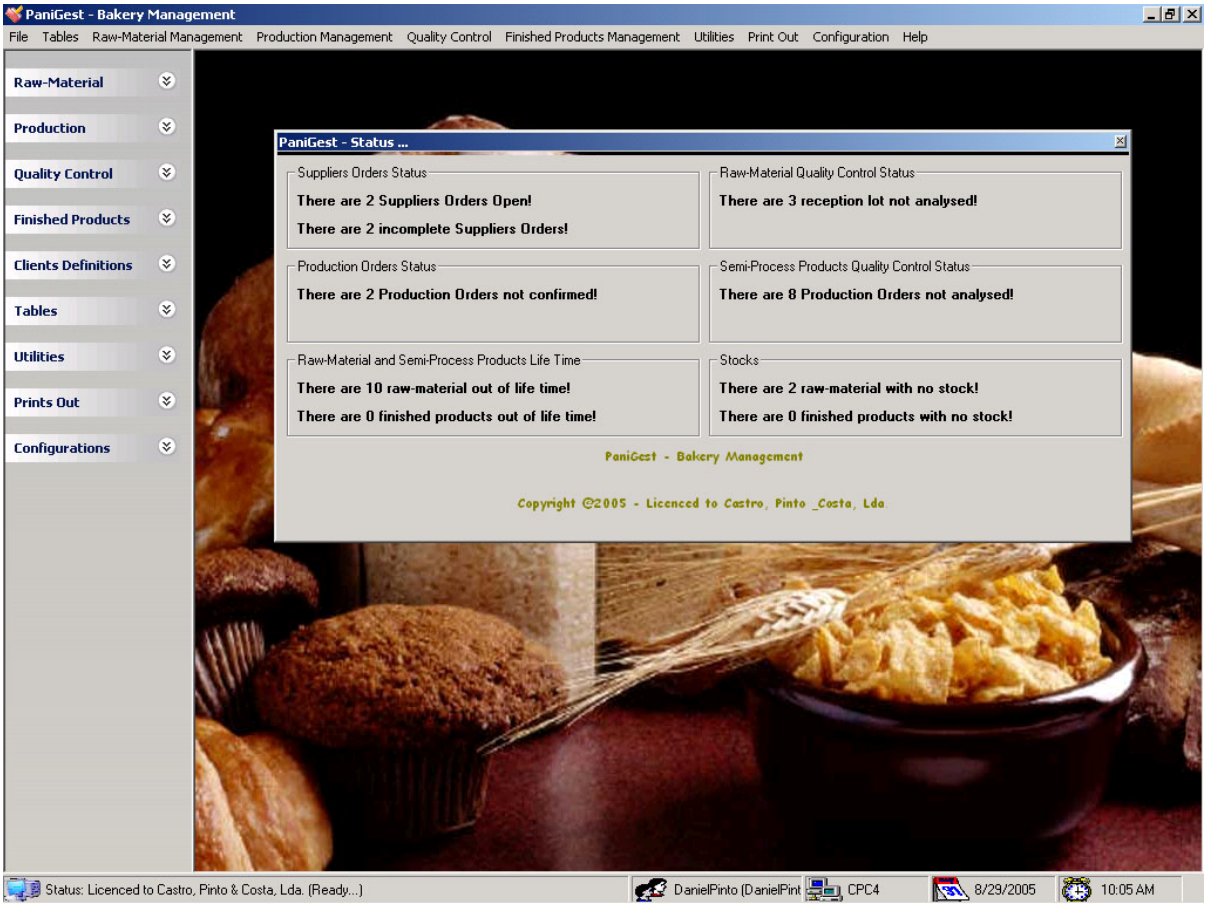

Figure 4.4: Main form

3.1.2. Section "Raw-materials"

The raw-materials section is the startup section of this program as in a real industry production context. It is used to execute supplier's orders, reception of raw-materials and raw-materials stock control. Specifications of raw-materials are extremely relevant to HACCP systems so the application is prepared to fill in raw-materials forms.

In this form all the relevant data, namely microbiological and organoleptic data, are recorded as well as acceptation/rejection criteria. Moreover, suppliers' information, costs, average costs, minimum and maximum stocks are also defined under this section. When inserting a new raw material, the user can activate the option that forces that raw-material to

pass by a quality control check, on reception. Further ahead, the quality control section will be explored and explained.

Perhaps the most important feature of this section is the raw-material reception (Figure 4.5). This is made automatically by the attribution of a sequential number, the reception number (RN), by supplier lot which is the first internal lot (auto-number). To easily guarantee the raw-materials traceability it is advisable to attribute a new number (auto-number) for each supplier lot even if it corresponds to the same delivery (order form). Furthermore, this sequential number will be used to maintain traceability and stocks.

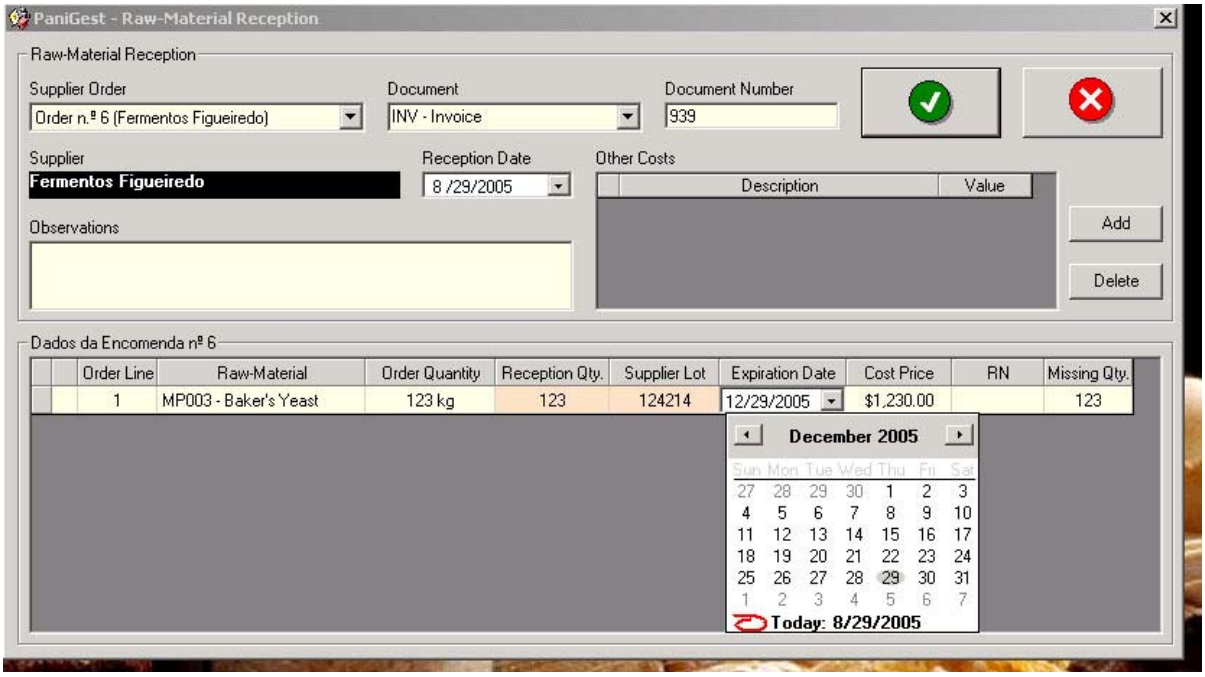

Figure 4.5: Example of a raw-material reception

3.1.3. Section "Production"

It is in this section of the program that data of on-course and finished products can be introduced. In food safety systems it is important to define internal specifications for the products in order to control the production. The program has forms for intermediate and final products, including the insertion of product's formulations as well as different final products derived from the same formulation (example: 1 kg bread, 0.25 kg bread). Similarly to the raw-material section, it is possible to define whether a product goes or not through a quality control stage before expedition.

Production orders (Figure 4.6) automatically indicate the raw-materials type, quantity and suggest the raw-material internal lot to be used as well as define the lot number (auto-

number). The suggestion of the internal lot is made by a very complex algorithm that calculates raw-materials distribution according to FIFO (First in – First out) and expiration dates. It also considers quality control blocked raw-material lots. If there are not enough raw-materials an error message will be generated with the information of the missing quantity of the raw-material in question.

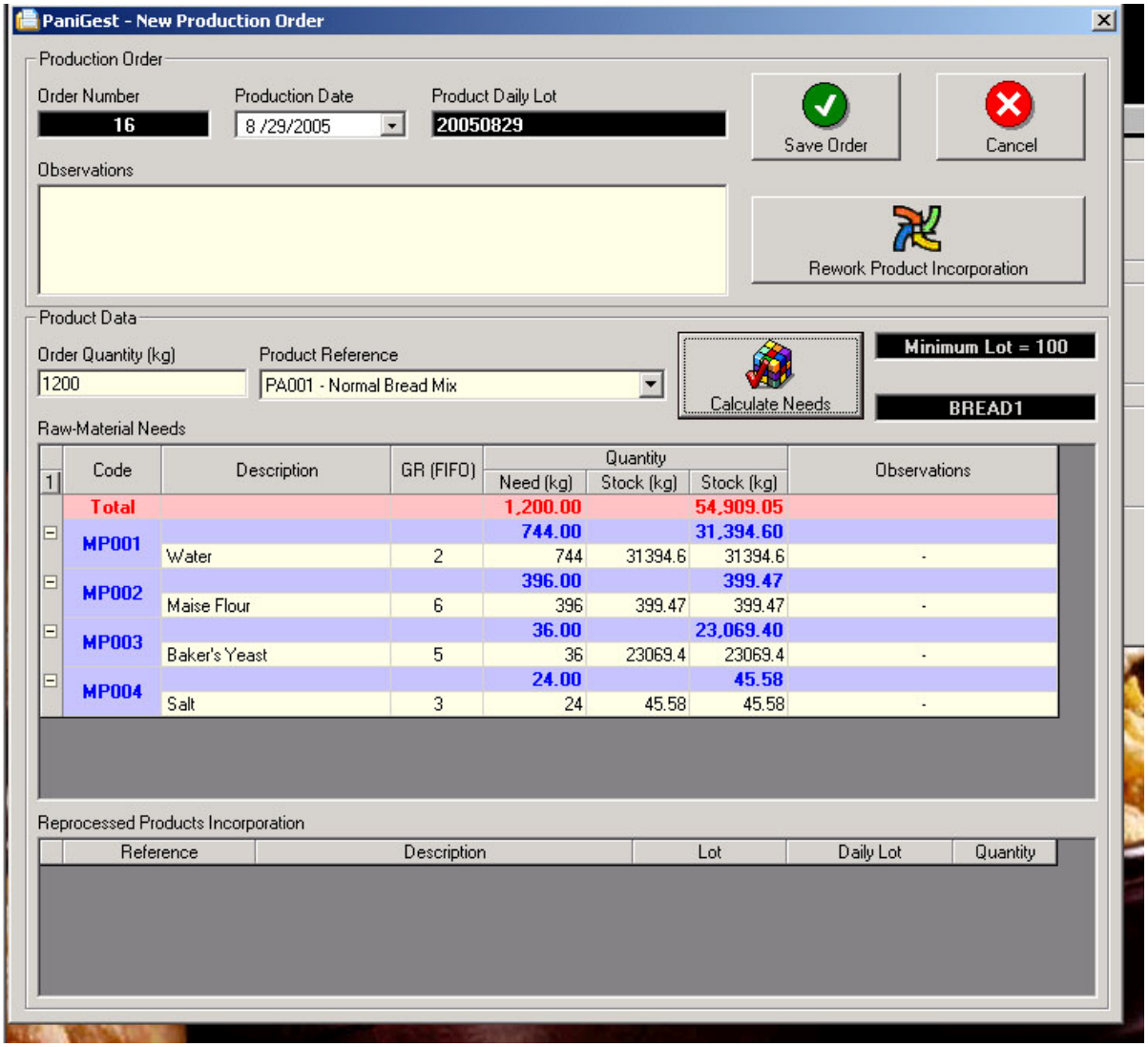

Figure 4.6: Example of a production order

The production order is a printable output used by the production section as a registry (Figure 4.7).

CASTRO PINTO & COSTA
QUALIDADE & INOVAÇÃO

Production Order

Number: 16

Production Order 25-11-2009

Reference: PA001 Description:

Lot: 200901125 Preview Quantity: 1.200,00 kg

Daily Lot: BREAD1 User: DanielPinto

Order Observations: -

Raw-Material

Code	Description	Quantity (kg)	GR	GR Used	Real Quantity (kg)
MP001	Water	744,00	2		
MP002	Maise Flour	396,00	6		
MP003	Baker's Yeast	36,00	5		
MP004	Salt	24,00	3		

Reprocessed Products

Reference	Description	Quantity (kg)	Lot - Daily Lot

Figure 4.7: Pre-defined report output for Production Order

The production operator should write down in the referenced paper form the exact quantity of the used rawmaterials and the correct internal lot when there are changes to the original order. The lot number of a final product consists of two different parts identifying the production date (code 1) and the product code (code 2), respectively.

If more then one batch of a given product is produced in the same day the lots will be identified sequentially (20090225 BREAD1, 20090225 BREAD2, for example). The lot codification format is defined by each company where this program is installed and implemented.

PaniGest® is also prepared to incorporate and trace back re-worked products, which is an extremely difficult task in the food industry (e.g. leftovers from the previous day) in a production order. This option allows tracing back reworked products that were used in a specific production order and, consequently, identify any problems related with the use of

leftovers in a fresh product. After confirmation of the exact raw-material consumption the production manager closes the production order. It is only atthis stage that raw-material stock movement is finally saved and the product becomes available to the quality control (if this option is activated at the configuration section). In this section, it is also possible to run production simulations in order to preview raw-material consumption thus allowing managing supplier's orders. All simulations that are made and saved may be printed out.

3.1.4. Section "Quality Control"

All the data and documents that a Quality Control Department involve are implemented and can be generated by the program. The main goal of this section is to document quality control operations namely acceptation/rejection decisions, release procedures, printing technical analysis bulletins, among others (Figure 4.8). The quality control is made according to the specifications that were inserted simultaneously with the introduction of a new raw-material or product. A security measure was implemented: the stock movement and updates is made only when the quality control result is conform. If there is a non-conformance, a non-conformance form (internal or to the supplier) can be generated and printed. As referenced before, the program has an algorithm to block a raw-material lot by an authorized user. If this happens, the specific raw-material becomes unavailable for movement and consequently for production.

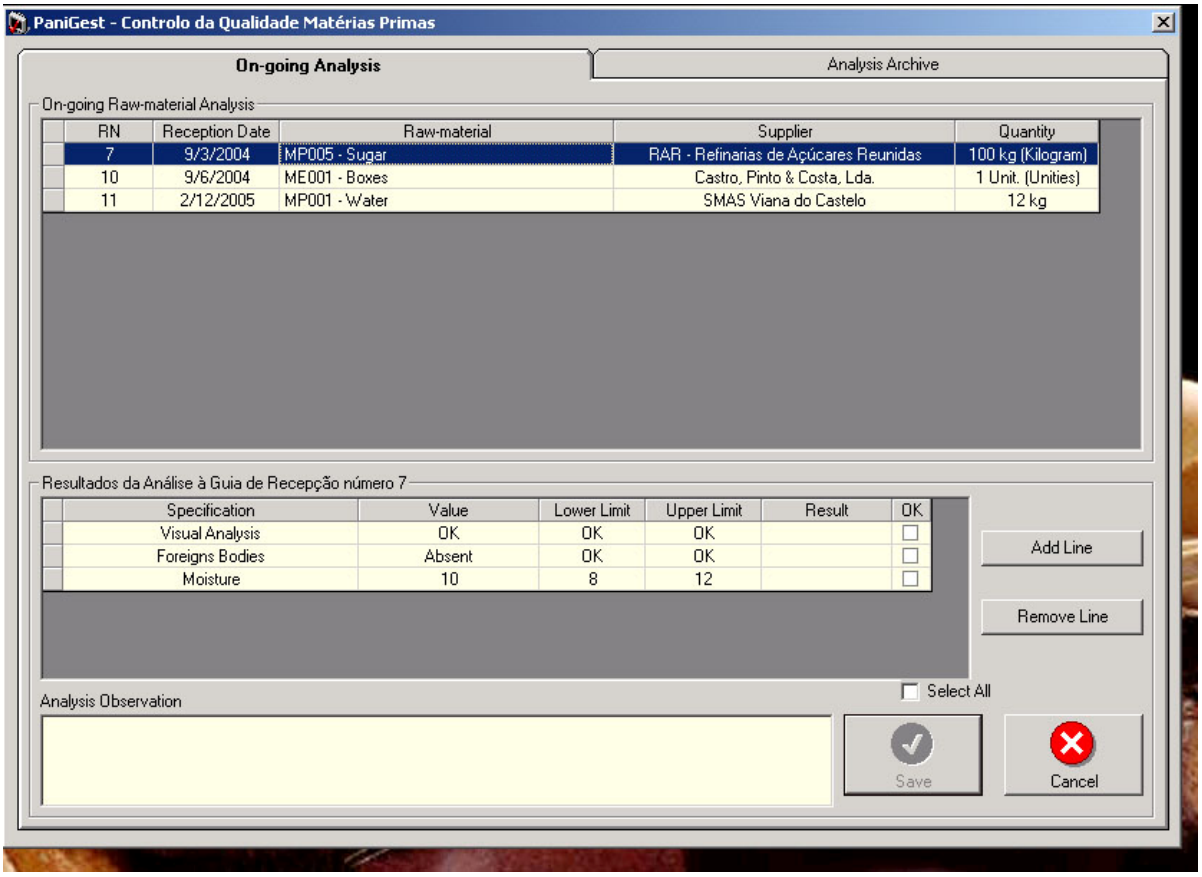

Figure 4.8: Raw-Material Quality Control Form

Traceability is, perhaps, the most important feature of the application and runs as a sub-section of quality control.

Two types of traceability queries can be made: traceability to final products (Figure 4.9) and traceability of rawmaterials.

In the first case, the user inputs the final lot of the product (code 1 or code 1 plus code 2) and all the information of traceability is displayed in a tree form (Figure 4.9). The displayed information (for code 1 input) is distributed by the following sections: the daily production references; the product and production data; specifications and quality control results; the raw-materials (and lots) that were used in the production; the quantity of each raw-material; the specifications and quality control results; the finishing (if applicable) raw-materials; in-course products; the re-worked products that were used and those who have been generated within this production. It is also possible to trace if the product was released and sold and if so who was (were) the client(s) it was sold to. If the user inputs a raw-material lot the finished products lots where that particular raw-material was used are displayed.

Recall procedures have to be made concerning two major objectives: to minimize the economical losses and to guarantee safety. Being so, the more specific the records are, the fewer products need to be recalled. However, this has to be done as quickly as possible. Using PaniGest®, it is just a matter of seconds to find out where the product is and to segregate it or to recall it.

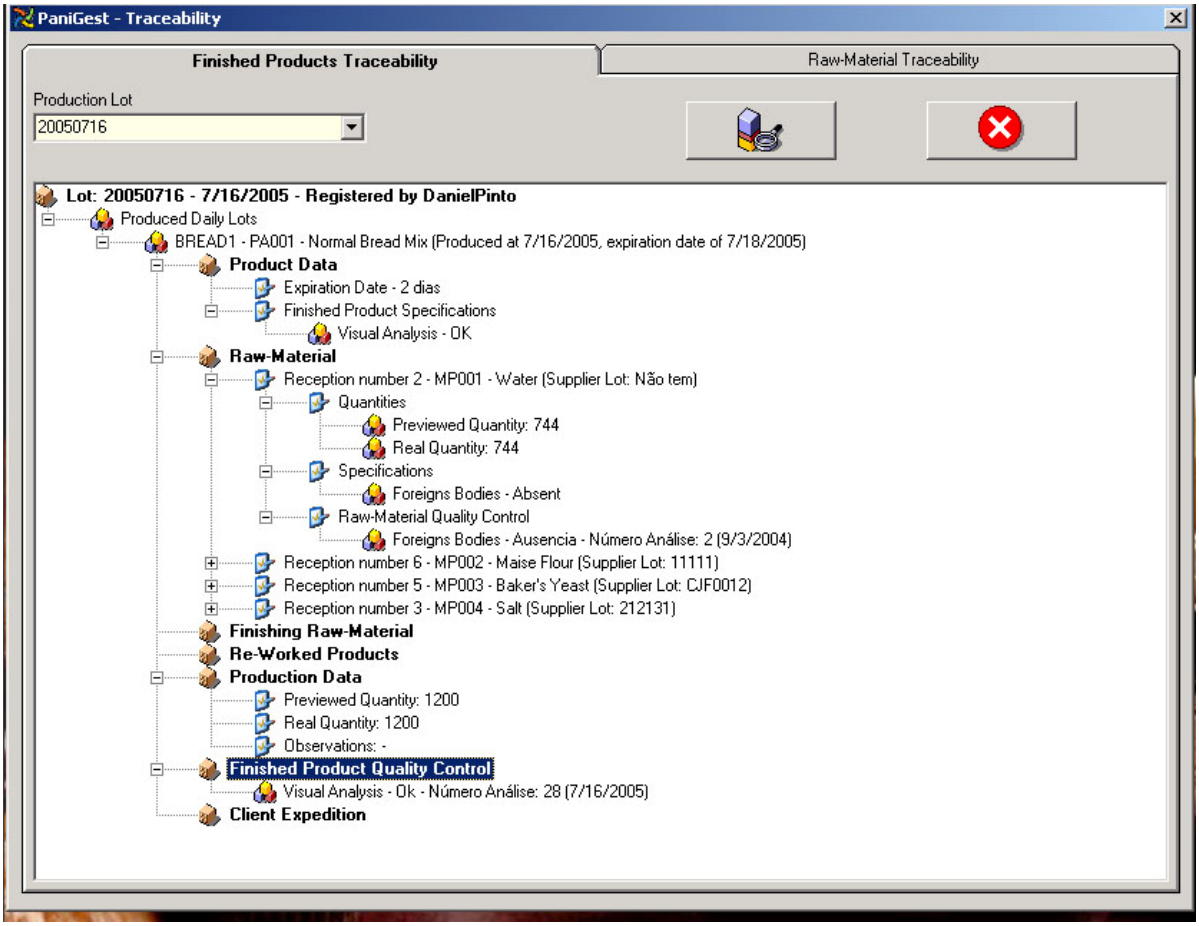

Figure 4.9: Traceability form example

3.1.5. Section "Finished Product"

Finally, the final product must be finished and delivered to the client. In this section there is the possibility of finishing products (for example when a product is packaged). It is also possible to query the final product stocks, to make expeditions and to query product stocks movements. According to EU legislation traceability must be possible also to the final client. In order to achieve this requirement, with this program expedition can be made either to single clients or to pre-defined routes (a very common feature in baking industry). A pre-defined route consists of a group of clients, usually in the same geographical location,

with standard quantity and products orders. If this is the case, the user inserts the route at the client definition section and at the expedition moment, the program, once again, suggests the lots of products to be supplied (using FIFO) and the user just has to confirm the lots and the quantity.

3.1.6. Other Sections

Under Tables section, useful information can be stored which will be used by other sections. Some examples are payment conditions; type of documents; VAT taxes; languages; types of currency and measuring units. The Utilities section is used to activate, inactivate or re-activate clients, suppliers, raw-materials and products.

4. Conclusions

The developed computer package, PaniGest®, is currently under testing in several Portuguese baking industries. So far, the feedback has been good, especially because it reduces the amount of paperwork and allows to clearly identify responsibilities. Also, the application allows implementing traceability in the food (baking) industry without a large number of technical and human resources and it facilitates recall procedures with economical and safety benefits. The generated outputs are also important records in case of an Audit.

PaniGest® is very user friendly, especially because it runs under a common Microsoft Windows environment and uses common computer user controls. Another important advantage is the use of a web browser to easily query information by users who do not need the program installed on their personal computer. It is designed to meet the needs of the food industry, specifically baking industries, merging production management, food safety management and systems with regulatory requirements.

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Chapter 5.

LabGest® - Laboratory Management Software

Abstract

Laboratory accreditation according to NP EN ISO/IEC 17025 gained importance in the last decade being a fundamental requirement to work with major companies.

This standard imposes several requirements to the labs namely in terms of internal and external quality control, traceability, results validation, among others. Paperwork needed to comply with this standard is huge and it becomes a major obstacle for small laboratories with limited resources..

LabGest® is a user-friendly computer package designed to manage the laboratory work integrating the major quality control tasks. This application was developed in Visual Basic language over an SQL database and its main features are: to manage culture medium from reception of components to production, including quality control and traceability to the assays in which the media is used; to manage sample reception including planning of analytical control plans; to trace technicians and equipments used in the assays; to calculate results using pre-defined formulas and to print the results into a Test Report.

It runs on a personal computer over Windows Vista or Windows 7 operating system. The program also uses common Internet Browsers to make information available to users.

1. Introduction

Companies have to continuously deliver high-quality products and/or services if they want to be successful in the marketplace in the long term. Quality improvement has become a key national and international business strategy. Most companies are using quality systems as a method of assuring the consistent conformity of products or services to a defined set of standards or customer expectations.

Paralel to food safety and food safety management systems developement, Laboratory quality management systems are almost mandatory and are being demanded by industry and Governmental Authorities. These systems include all steps in sample preparation and processing chain namely the sample conditions, supply of culture medium raw materials, sample processing, analytical results, maintenance of laboratory equipment and training and education of staff.

Laboratories play an important role in the quality systems of the companies. The ISO/IEC 17025 can be used as a standard to develop and establish a quality system in the laboratory and for assessment by their clients or third parties. The standard is also being used as a criterion for laboratory accreditation.

The first edition (1999) of the International Standard "General Requirements for the Competence of Testing and Calibration Laboratories" was produced as a result of extensive experience in the implementation of ISO/IEC Guide 25 and EN 45001, both of which it replaced. It contains all the requirements that testing and calibration laboratories have to meet if they wish to demonstrate that they operate a management system, are technically competent and are able to generate technically valid results.

Management requirements of the first edition referred to ISO 9001:1994 and ISO 9002:1994. These standards have been superseded by ISO 9001:2000, which made an alignment of ISO/IEC 17025 necessary. In the second edition of NP EN ISO/IEC 17025, released in 2005, clauses were amended or added only when considered necessary in the light of ISO 9001:2000. Testing and calibration laboratories that comply with this International Standard will therefore also operate in accordance with ISO 9001(including its last edition of 2008). Accreditation bodies that recognize the competence of testing and calibration laboratories use this International Standard as the basis for their accreditation.

ISO/IEC 17025 is divided into five chapters, two annexes and one bibliography section:

- Chapter 1: Scope - The standard covers the technical activities of a laboratory and the management and organizational aspects to perform the technical activities in a competent way.
- Chapter 2: Normative References.
- Chapter 3: Terms and Definitions.
- Chapter 4: Management Requirements - Most of the requirements are similar to those specified in the ISO Standard 9001:2008.
- Chapter 5: Technical Requirements - Most of the requirements come from the ISO Guide 25.
- Annex A: Cross References to ISO 9001.
- Annex B: Guidelines for Establishing Applications for Specific Fields.
- Bibliography.

Most important are chapters 4 and 5 on management and technical requirements.

Traceability and Laboratory Quality Management systems can work properly based on pen and paper versions but they are time and resource consuming which makes them difficult to implement in small and medium laboratories where the resources are scarce.

The use of new technologies of information and communication to support and facilitate the practical implementation of these complex systems is very recent and, until now, it can be found only in larger companies. The development of adequate computer packages to reduce the paperwork involved in the management system can be extremely helpful for NP EN ISO/IEC 17025 accreditted laboratories. Therefore, the development of a computer application, using a user-friendly interface, specially designed for laboratories, was pertinent. LabGest® was created to respond to these needs integrating requirements such as traceability with quality control, culture medium production management, raw material and finished culture medium stock control and consumptions.

Resuming, implementation of NP EN ISO/IEC 17025 provides a system for continuous improvement of daily laboratory practices. Direct benefits include faster identification and resolution of issues, improved customer satisfaction, meeting of quality requirements of specialized customers, and an overall increase in laboratory business.

2. Materials and Methods

2.1. SQL - Structured Query Language

SQL is the industry standard language for relational databases. The standard adopted in 1986 to ANSI. The main relational databases accept some form of SQL, trying to be in ANSI. Some terminology used in the Database is:

- Table - Basic structure of data storage;
- Line - Combination of the values of columns in a table. Same as record;
- Column - represents a type of data in a table. It is described by a name of column;
- Attribute - an area which contains a data type. It is the intersection of a row with a column. The even field;
- Primary Key - It is the exclusive column that identifies each row of a table;
- Foreign key - A column or set of columns for a primary key of another table. From the foreign key can relate tables (join).

2.2. SQL Server Management Studio

SQL Server Management Studio is an integrated environment for accessing, configuring, managing, administering, and developing all components of SQL Server. SQL Server Management Studio combines a broad group of graphical tools with a number of rich script editors to provide access to SQL Server to developers and administrators of all skill levels.

SQL Server Management Studio (Figure 5.1) combines the features of Enterprise Manager, Query Analyzer, and Analysis Manager, included in previous releases of SQL Server, into a single environment. In addition, SQL Server Management Studio works with all components of SQL Server such as Reporting Services, Integration Services, and SQL Server Compact 3.5 SP1. Developers get a familiar experience, and database administrators get a single comprehensive utility that combines easy-to-use graphical tools with rich scripting capabilities.

SQL Server Management Studio includes the following general features:

- Supports most administrative tasks for SQL Server;

- A single, integrated environment for SQL Server Database Engine management and authoring;
- New management dialogs for managing objects in the SQL Server Database Engine, Analysis Services, Reporting Services, Notification Services, and SQL Server Compact 3.5 SP1, that allows you to execute your actions immediately, send them to a Code Editor, or script them for later execution;
- Non-modal and resizable dialogs allow access to multiple tools while a dialog is open;
- A common scheduling dialog that allows you to perform action of the management dialogs at a later time;
- Exporting and importing SQL Server Management Studio server registration from one Management Studio environment to another;
- Save or print XML Showplan or Deadlock files generated by SQL Server Profiler, review them later, or send them to administrators for analysis;
- A new error and informational message box that presents much more information, allows you to send Microsoft a comment about the messages, allows you to copy messages to the clipboard, and allows you to easily e-mail the messages to your support team;
- An integrated Web browser for quick browsing of MSDN or online help;
- Integration of Help from online communities;
- A tutorial on SQL Server Management Studio to help you take advantage of the many new features and become more productive right away. To take the tutorial, go to Tutorials;
- A new activity monitor with filtering and automatic refresh;
- Integrated Database Mail interfaces.

A database in SQL Server is made up of a collection of tables. These tables contain data and other objects, such as views, indexes, stored procedures, user-defined functions, and triggers that are defined to support activities performed with the data. The data stored in a database is typically related to a particular subject or process, such as inventory information for a manufacturing warehouse.

The LabGest® database was developed in SQL Server 2008 using SQL Server Management Studio 2008.

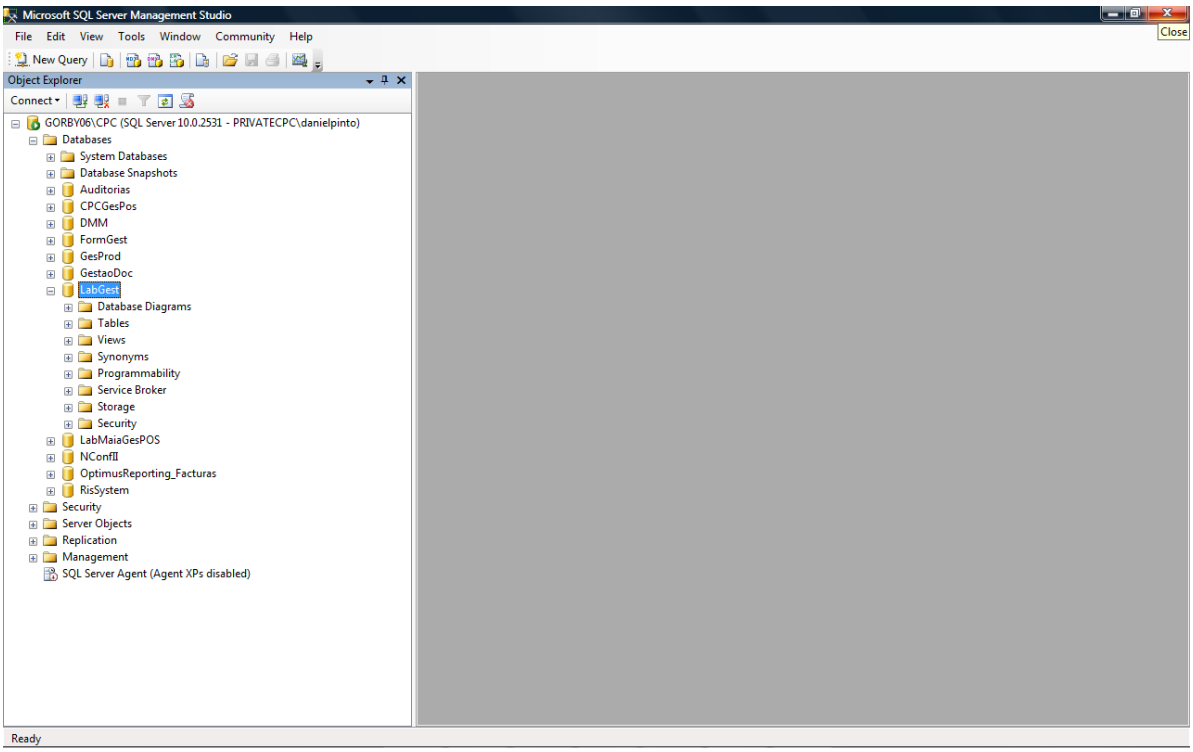


Figure 5.1: Overview of SQL Server Management Studio 2008

2.2.1. Triggers

Microsoft® SQL Server™ 2000 triggers are a special class of stored procedure defined to execute automatically when an UPDATE, INSERT, or DELETE statement is issued against a table or view. Triggers are powerful tools that sites can use to enforce their business rules automatically when data is modified. Triggers can extend the integrity checking logic of SQL Server constraints, defaults, and rules, although constraints and defaults should be used instead whenever they provide all the needed functionality.

Tables can have multiple triggers. The CREATE TRIGGER statement can be defined with the FOR UPDATE, FOR INSERT, or FOR DELETE clauses to target a trigger to a specific class of data modification actions. When FOR UPDATE is specified, the IF UPDATE (column_name) clause can be used to target a trigger to updates affecting a particular column.

Triggers can automate the processing for a company. In an inventory system, update triggers can detect when a stock level reaches a reorder point and generate an order to the supplier automatically. In a database recording the processes in a factory, triggers can e-mail or page operators when a process exceeds defined safety limits.

Triggers contain Transact-SQL statements, much the same as stored procedures. Triggers, like stored procedures, return the result set generated by any SELECT statements in the trigger. Including SELECT statements in triggers, except statements that only fill parameters, is not recommended. This is because users do not expect to see any result sets returned by an UPDATE, INSERT, or DELETE statement.

Below is an example of a trigger that gives automatically the final certificate code in the LabGest® database:

```
USE [LabGest]
GO
/****** Object:  Trigger [dbo].[InsertCodigoFinal]    Script
Date: 08/13/2009 11:30:22 ******/
SET ANSI_NULLS ON
GO
SET QUOTED_IDENTIFIER ON
GO
ALTER TRIGGER [dbo].[InsertCodigoFinal]
   ON  [dbo].[Tabela_Boletins]
   AFTER INSERT
AS
     declare @Numero Int
     declare @NumeroBoletim Int
     declare @datarecepcao NVARCHAR (4)
     declare @ano NVARCHAR (4)
     declare @tipo NVARCHAR
     declare @codigofinal NVARCHAR (50)
     declare @idtipo int
     declare @estado int

BEGIN
     Select @numero = idnumero from INSERTED
     Select @numeroboletim = idboletim from INSERTED
     Select @ano = ano from INSERTED
     Select @tipo = tipoboletim from INSERTED
     Select @idtipo = idtipoanalise from INSERTED
     Select @estado = estado from INSERTED

if @estado <> 6
     set @codigofinal = @tipo + ' ' +
CONVERT(NVARCHAR,@numero) + ' - ' + @ano
if @estado =6
     set @codigofinal = @tipo + ' - Prevista'
if @estado <> 6
     update Tabela_TiposAnalises set ultimonumero = @numero
where idtipoanalise = @idtipo

     update tabela_Boletins set codigofinal = @codigofinal ,
numeroboletim = @numero where idboletim = @numeroboletim
```

```
END
```

2.2.2. Stored Procedures

When an application is created using Microsoft® SQL Server™ 2000, the Transact-SQL programming language is the primary programming interface between the applications and the SQL Server database. When Transact-SQL programs are used, two methods are available for storing and executing the programs. Either one can store the programs locally and create applications that send the commands to SQL Server and process the results, or store the programs as stored procedures in SQL Server and create applications that execute the stored procedures and process the results.

Stored procedures in SQL Server are similar to procedures in other programming languages in that they can:

- Accept input parameters and return multiple values in the form of output parameters to the calling procedure or batch;
- Contain programming statements that perform operations in the database, including calling other procedures;
- Return a status value to a calling procedure or batch to indicate success or failure (and the reason for failure);

The Transact-SQL EXECUTE statement can be used to run a stored procedure. The benefits of using stored procedures in SQL Server rather than Transact-SQL programs stored locally on client computers are:

- They allow modular programming. Once the procedure is created it maybe used repeatidly whenever it is necessary. Even if the program source code (e.g visual basic) is replaced by another interface programming code the procedure remains valid because it is programmed into the database
- They allow faster execution. If the operation requires a large amount of Transact-SQL code or is performed repetitively, stored procedures can be faster than batches of Transact-SQL code. They are parsed and optimized when they are first executed, and a compiled version of the stored procedure remains in memory cache for later use. This means the stored procedure does not need to be reparsed and reoptimized with each use resulting in much faster execution times.

- They can reduce network traffic. An operation requiring hundreds of lines of Transact-SQL code can be performed through a single statement that executes the code in a procedure, rather than by sending hundreds of lines of code over the network.
- They can be used as a security mechanism. Users can be granted permission to execute a stored procedure even if they do not have permission to execute the procedure's statements directly.

A SQL Server stored procedure is created with the Transact-SQL CREATE PROCEDURE statement and can be modified with the ALTER PROCEDURE statement. The stored procedure definition contains two primary components: the specification of the procedure name and its parameters, and the body of the procedure, which contains Transact-SQL statements that perform the procedure's operations. As explained at LabGest® Database Development section stored procedures were used to execute partial code in order to improve database and program performance.

Also, in SQL Server, many administrative and informational activities can be performed by using system stored procedures. The system stored procedures are grouped into the categories shown in the following table.

Table 5.1: System stored procedures categories

Category	Description
Active Directory Stored Procedures	Used to register instances of SQL Server and SQL Server databases in Microsoft Windows 2000 Active Directory.
Catalog Stored Procedures	Used to implement ODBC data dictionary functions and isolate ODBC applications from changes to underlying system tables.
Change Data Capture Stored Procedures	Used to enable, disable, or report on change data capture objects.
Cursor Stored Procedures	Used to implements cursor variable functionality.
Database Engine Stored Procedures	Used for general maintenance of the SQL Server Database Engine.

Table 5.2: System stored procedures categories (continued)

Category	Description
Database Mail and SQL Mail Stored Procedures	Used to perform e-mail operations from within an instance of SQL Server.
Database Maintenance Plan Stored Procedures	Used to set up core maintenance tasks that are required to manage database performance.
Distributed Queries Stored Procedures	Used to implement and manage distributed queries.
Full-Text Search Stored Procedures	Used to implement and query full-text indexes.
Log Shipping Stored Procedures	Used to configure, modify, and monitor log shipping configurations.
Automation Stored Procedures	Used to enable standard Automation objects for use within a standard Transact-SQL batch.
Policy-Based Management Stored Procedures	Used for Policy-Based Management.
Replication Stored Procedures	Used to manage replication.
Security Stored Procedures	Used to manage security.
SQL Server Profiler Stored Procedures	Used by SQL Server Profiler to monitor performance and activity.
SQL Server Agent Stored Procedures	Used by SQL Server Agent to manage scheduled and event-driven activities.
XML Stored Procedures	Used for XML text management.
General Extended Stored Procedures	Used to provide an interface from an instance of SQL Server to external programs for various maintenance activities

To execute a stored procedure, use the Transact-SQL EXECUTE statement. Alternatively, a stored procedure can be executed without using the EXECUTE keyword if the stored procedure is the first statement in the batch.

System stored procedures begin with the characters sp_. They are physically stored in the Resource database, but logically appear in the sys schema of every system- and user-defined database in the instance of SQL Server. System stored procedures can be executed from any database.

2.3. LabGest® Database Development

As refered in the last section, there are several advantages when using a SQL Server database instead of a Microsoft Access database. So, LabGest® database was developed directly in SQL Server. SQL Server has many embedded statistical functions and can handle databases containing a large volume of data. The SQL Server 2008 can service requests from applications running on both, the same or a remote computer, while it can be efficiently interfaced with Visual Basic applications (Kostas Papadakis, Eftichios Koutroulis and Kostas Kalaitzakis, 2005).

As refereed, one of the advantages of using SQL Server is that part of the programming code could be stored into the dababase server reducing the size of the executable application. Being so, several "stored procedures" were developed and run whenever the user commands it at the interface. An example of a stored procedure, used to select current analysis, is presented in following code lines:

```
USE [LabGest]
GO
/****** Object:  StoredProcedure [dbo].[GET_ParametrosBoletim]    Script Date:
07/23/2009 19:33:28 ******/
SET ANSI_NULLS ON
GO
SET QUOTED_IDENTIFIER ON
GO

ALTER PROCEDURE [dbo].[GET_ParametrosBoletim]
        @idboletim int
AS
BEGIN
        SET NOCOUNT ON;
SELECT     TOP (100) PERCENT dbo.Tabela_Boletins_Parametros.IDBoletim,
dbo.Tabela_Boletins_Parametros.Analista,
dbo.Tabela_Boletins_Parametros.EstadoIncuba, dbo.Tabela_Parametros.Descricao,
dbo.Tabela_Parametros.MetodoReferencia,
```

```
dbo.Tabela_Parametros.ValorReferencia, dbo.Tabela_Parametros.UnidadesReferencia,
dbo.Tabela_Parametros.IDTipoParametro,
dbo.Tabela_Parametros.FormulaFinal, dbo.Tabela_Boletins_Parametros.IDParametro,
dbo.Tabela_Boletins_Parametros.IDParametroOriginal,
dbo.Tabela_Boletins_Parametros.Resultado, dbo.Tabela_Boletins_Parametros.Expoente,
dbo.Tabela_Unidades.Abre,
dbo.Tabela_Unidades.IDUnidade, dbo.Tabela_Parametros.IDTipoParametro AS Expr1,
dbo.Tabela_Familias_Parametros.MetodoReferencia AS MR,
dbo.Tabela_Familias_Parametros.ValorReferencia AS VR,
dbo.Tabela_Familias_Parametros.UnidadesReferencia AS UR,
dbo.Tabela_Boletins.FamiliaParametros, dbo.Tabela_TiposParametros.LocalReport
FROM        dbo.Tabela_Parametros INNER JOIN
dbo.Tabela_Boletins_Parametros ON dbo.Tabela_Parametros.IDParametro =
dbo.Tabela_Boletins_Parametros.IDParametro INNER JOIN
dbo.Tabela_Unidades ON dbo.Tabela_Parametros.UnidadesReferencia =
dbo.Tabela_Unidades.IDUnidade INNER JOIN
dbo.Tabela_Boletins ON dbo.Tabela_Boletins_Parametros.IDBoletim =
dbo.Tabela_Boletins.IDBoletim INNER JOIN
dbo.Tabela_TiposParametros ON dbo.Tabela_Parametros.IDTipoParametro =
dbo.Tabela_TiposParametros.IDTipoParametro LEFT OUTER JOIN
dbo.Tabela_Familias_Parametros ON dbo.Tabela_Parametros.IDParametro =
dbo.Tabela_Familias_Parametros.IDParametro AND
dbo.Tabela_Boletins.FamiliaParametros = dbo.Tabela_Familias_Parametros.IDFamilia
where dbo.Tabela_Boletins_Parametros.idboletim = @idboletim ORDER BY
dbo.Tabela_Parametros.IDTIpoParametro, dbo.Tabela_Parametros.Descricao
END
```

2.4. Interface Development

The interface was developed using Visual Basic 6.0™ by Microsoft® under a Microsoft® Windows® operating system (e.g. Windows Vista or Windows 7).

The application also uses commonly known ActiveX controls and some Dynamic Link Library (DLL) from Microsoft. A DLL is a library for applications within the Windows operating system as well as for programming and contains one or more functions that are compiled, linked, and stored separately from the applications using them (Takeuchi, 2002). However to support some of the present application's functionalities several DLLs had to be developed.

2.5. Error Handling

The error handling methodology used is identical to the one used in PaniGest® and was explained in the Chapter 4, section 2.3.

2.6. Outputs

Pre-defined reports were created using Seagate Crystal Reports as previously explained in Chapter 4, section 2.4.

3. Results and Discussion

3.1. LabGest®

LabGest® is a software completely based on the most recent technologies. It is highly customizable and easily adapted to the working practices of each laboratory. This system was developed according to the requirements of the standard NP EN ISO/IEC 17025. A sample code of LabGest® is available in Appendix.

LabGest® main features are:

- Configuration of techniques, products (culture media or solutions) and parameters;
- Print of technical sheet for sample collection with correct barcode identification;
- Integration with other billing systems;
- Web customer zone for results and reports over Internet;
- Terminal access from any computer of the laboratory with no installation and no licences
- Register analysis and control parameters;
- Manage reception of reagents, production orders of culture medium and checmical soluctions;
- Define and print labels containing lot information and other relevant data;
- Implement traceability back and forward in the laboratory;
- Define, notify responsibles and register quality control plans;
- Issue test reports;
- Manage incubation temperatures and times.

When the application starts, it opens its main form where the user can access multiple options and menus. Several of those options and menus correspond to table management (example: parameters). These data, introduced firstly in the program, are indispensable for the subsequent actions. Other options and menus, explored further ahead, are the operation and control options and menus, such as the analysis section, production section and quality control section.

3.1.1. The main form

The requirements for software vary according to the goals of its users. According to NP EN ISO/IEC 17025, one of the most important requirements is to manage human resources competences, and this is usually related to their responsibilities in the whole laboratory management system. For all users it would be more effective if the computer program interface could be changed according to the user's level of computer proficiency and authorization level.

For novice users, complicated input and output options are confusing and often result in users missing the objective of the program, and also may result in errors in operation. Dismissing unnecessary options and simplifying the interface, therefore, would be of great benefit to this type of users. Using graphics, such as charts, figures, and diagrams to represent the results of analysis is often more effective than a numerical description. Also different responsibilities related to filling down registries or validate them have to be managed. Being so, the software was developed using a friendly interface to all users and with user-level authentications (Figure 5.2).

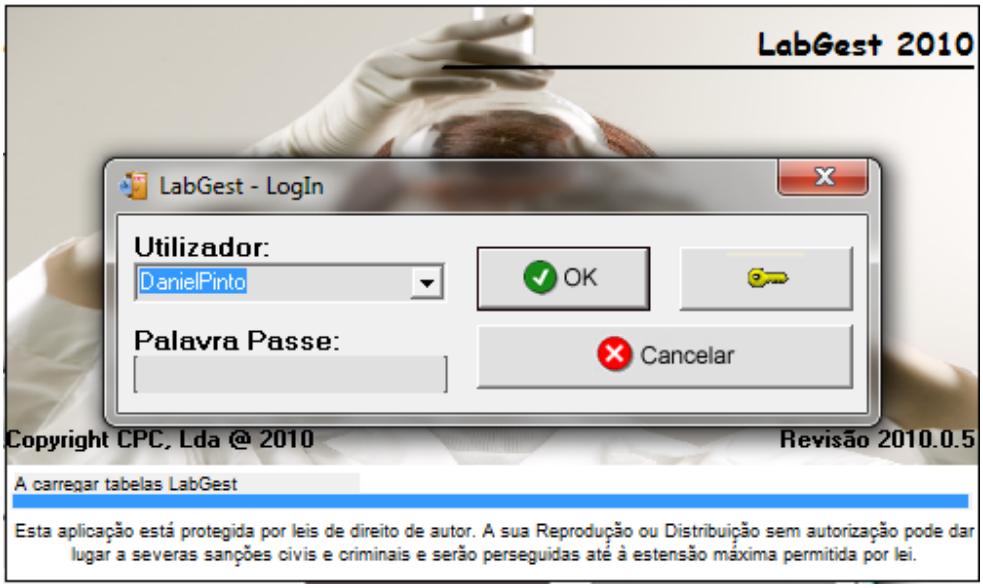


Figure 5.2: User log-in

It also allows multiple users to access the program simultaneously. However, a safety measure was implemented during development: it is impossible to use the same user name simultaneously in different workstations. After entering the user name and password, the software executes a Store Procedure with a database query to upload the user permissions

and validate the user log in. A DLL was developed by the authors to automatically import user and computer name from the Windows Operating System. This DLL is of extreme importance not only because it limits access to the users but also because it can be used as a "signature". With this feature, it is possible to audit all the users operations and to determine responsibilities.

After the user log-in, the main form (Figure 5.3) contains a lateral menu with the different sections of the computer package: Analysis Management; Laboratory Management; Traceability; Quality Control; Solutions and CM; Lists; LabGest® Tables and Configurations. This lateral menu was made using an OCX control called vbaccelarator Explorer Bar Control available at the vbaccelarator web site for download. It can also open a "Status Form" showing the actual status of pending, ongoind and finished analysis. All the options available in the lateral menu also exist at the upper menu of the main form. The first group of options (Analysis Management) is a dynamic group wich is created by reading the database analysis types table. Each time a new analysis type is inserted, LabGest® asks user to restart and refresh (recreate) the Analysis Management Menu Group. (Figure 5.4 to Figure 5.7). At the same time, a folder to hold certificates analysis is created at the Administrator defided LabGest® main path (Figure 5.8)

Inside each main menu sections there are sub-menus opening inside the main form (MDI form). This type of construction allows the left menu to be always available. All sub-forms of the program open inside the main form.

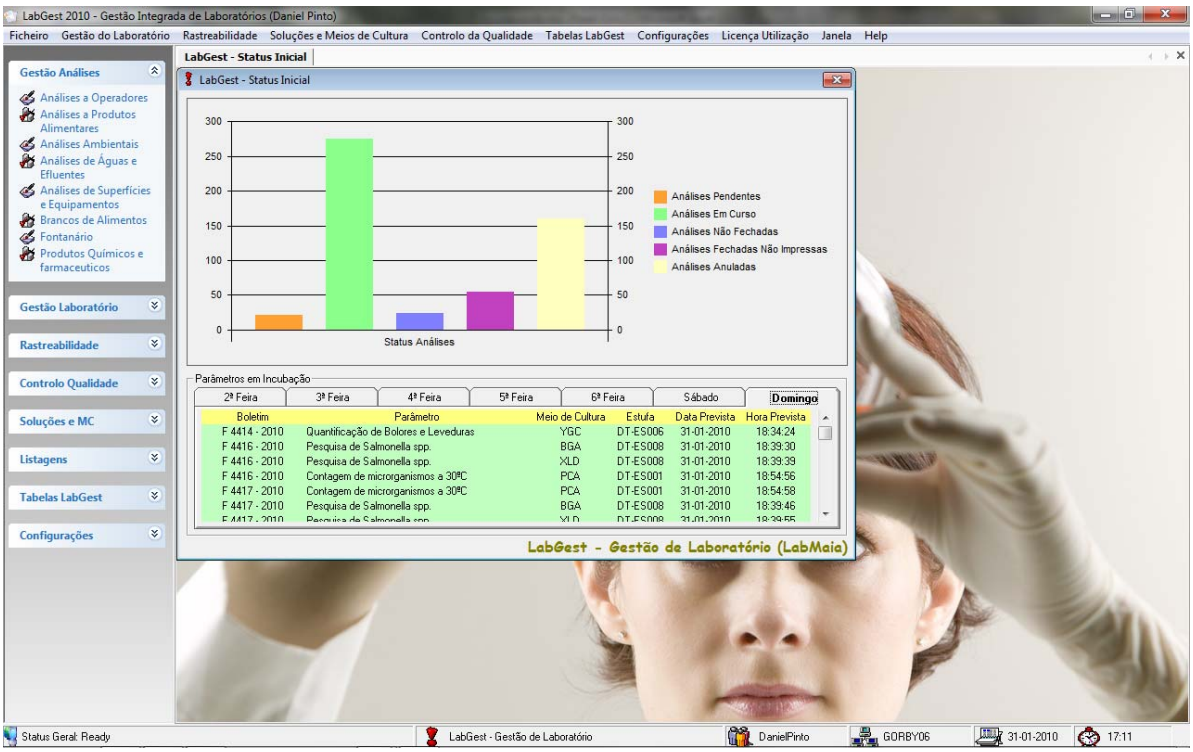


Figure 5.3: LabGest® Main form with initial status

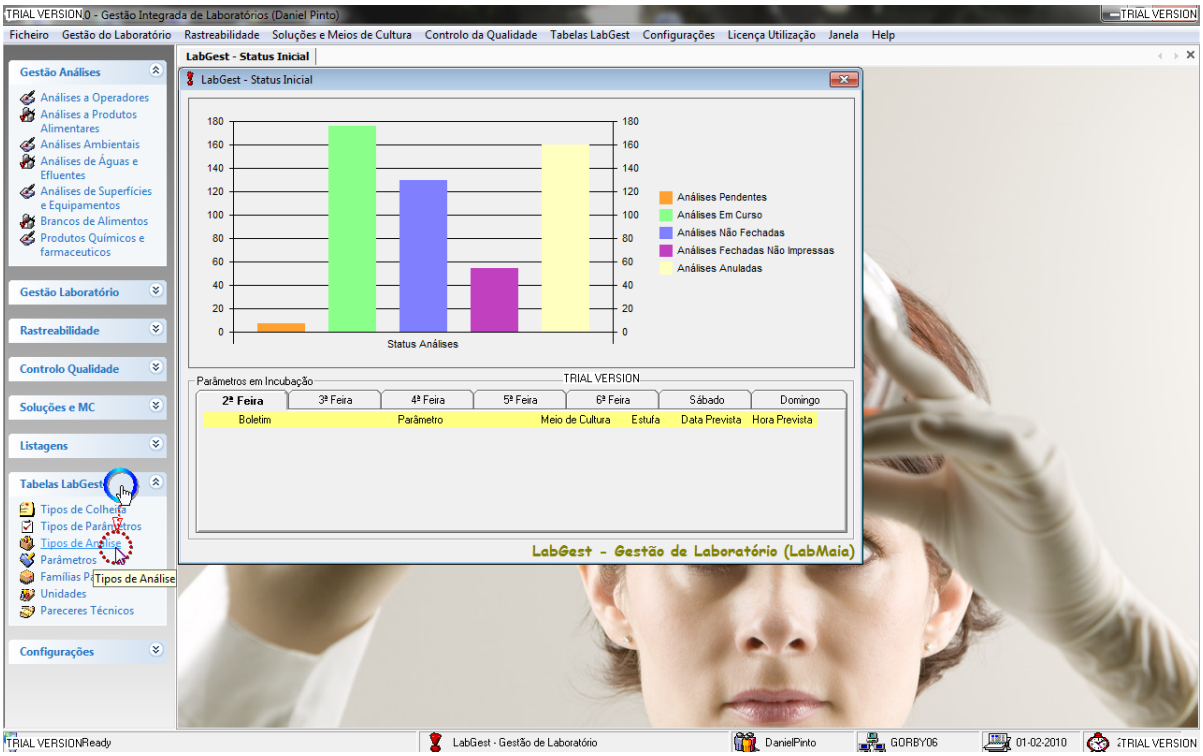


Figure 5.4: Analysis Type Management and creation of Analysis Management Options Group Step 1 of 4: choosing the "Analysis Type" option

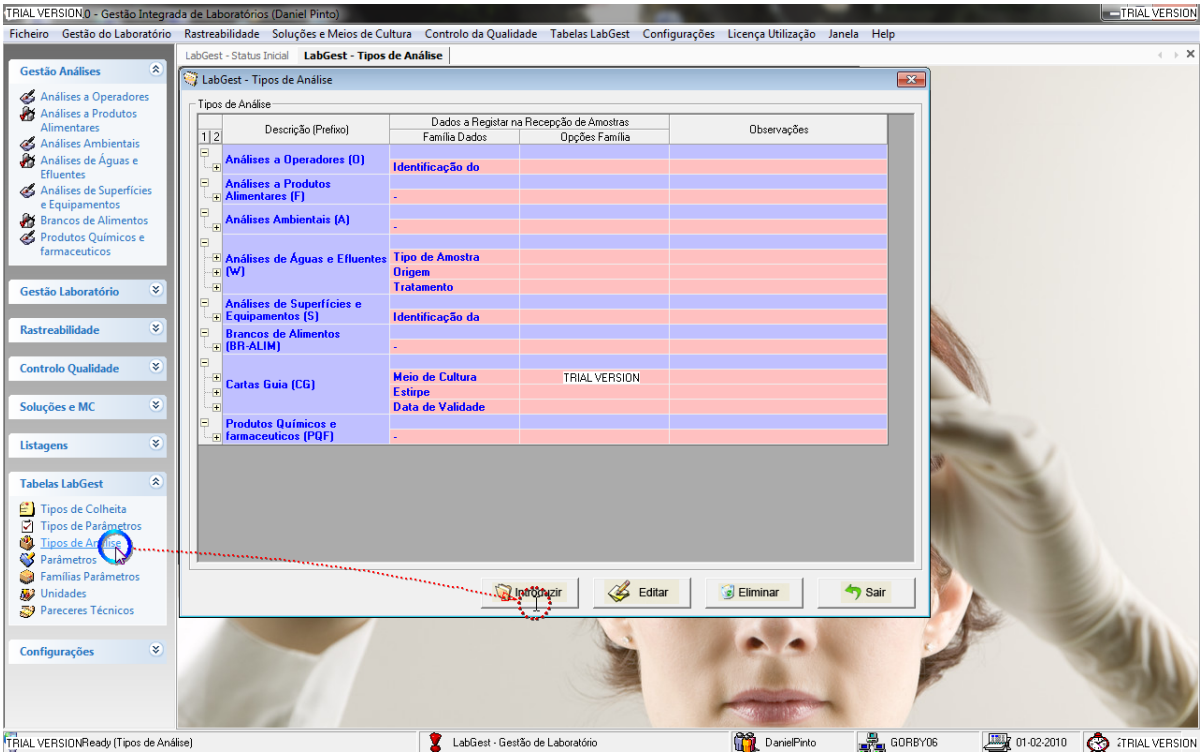


Figure 5.5: Analysis Type Management and creation of Analysis Management Options Group Step 2 of 4: introducing a new "Analysis Type"

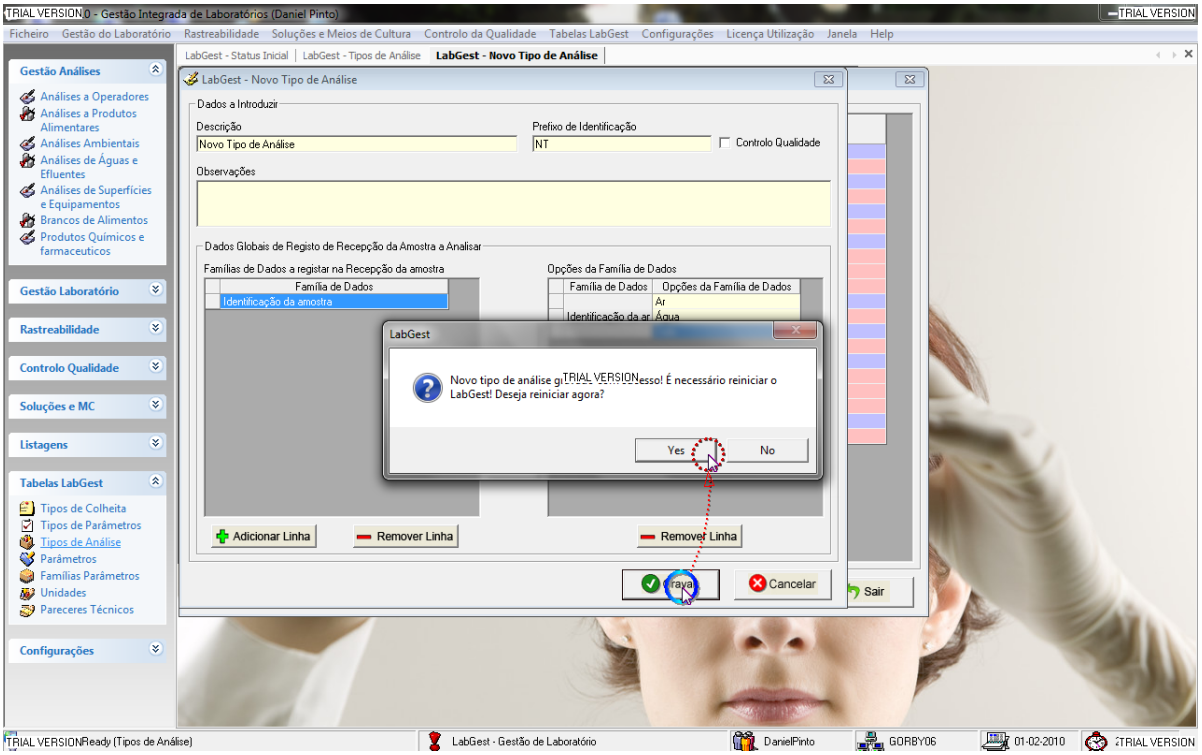


Figure 5.6: Analysis Type Management and creation of Analysis Management Options Group Step 3 of 4: restarting the program in order to refresh the menu

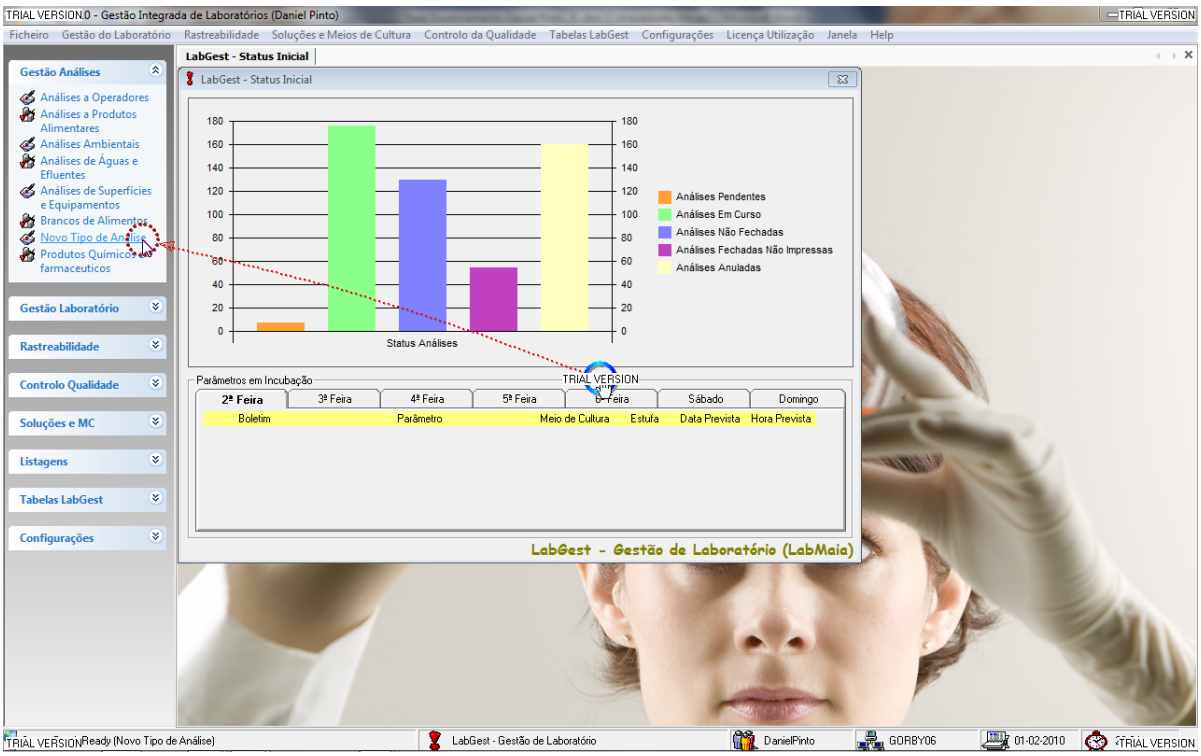


Figure 5.7: Analysis Type Management and creation of Analysis Management Options Group Step 4 of 4: customized menu is ready to use

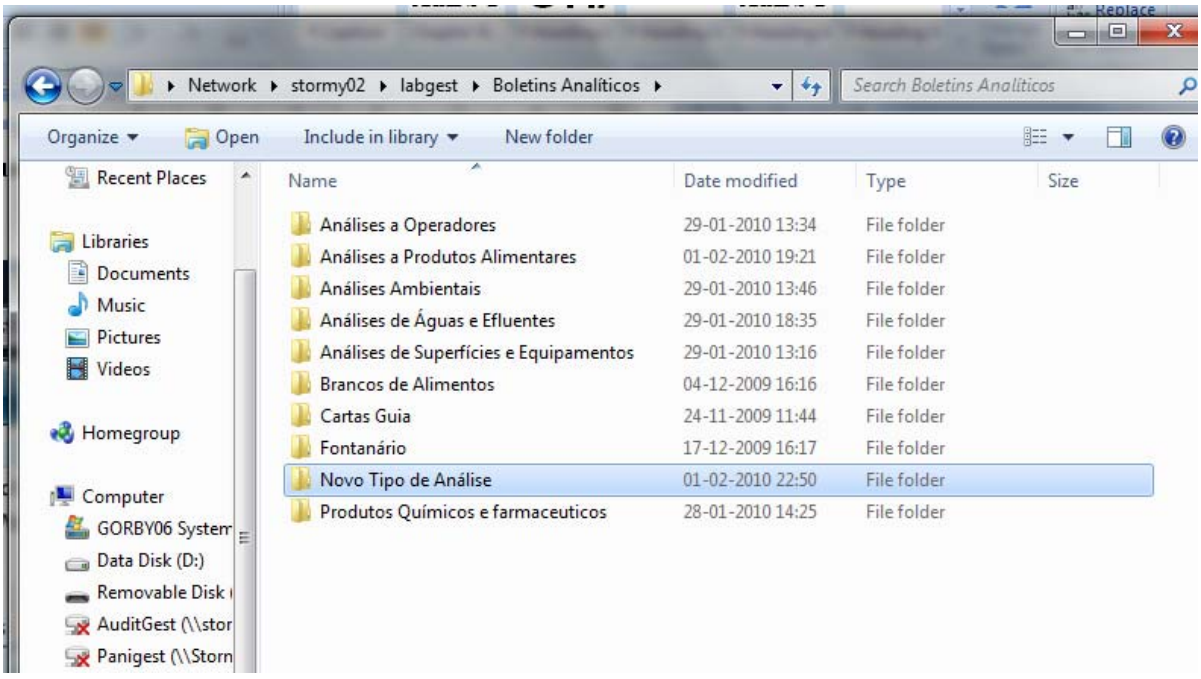


Figure 5.8: New folder at LabGest® Main path

3.1.2. Section "Configurations"

The configurations section is the startup section of this program. It is used to configure users and permissions, WEB users (to access this software from web browser), and other system configurations like the application main path to save all the reports and certificates. One of the most interesting options of this section is located at System Configurations. Here, the user can configure external data access (for example to a commercial management software) in order to read general information, for example customers, suppliers, raw-materials, etc. (Figure 5.9).

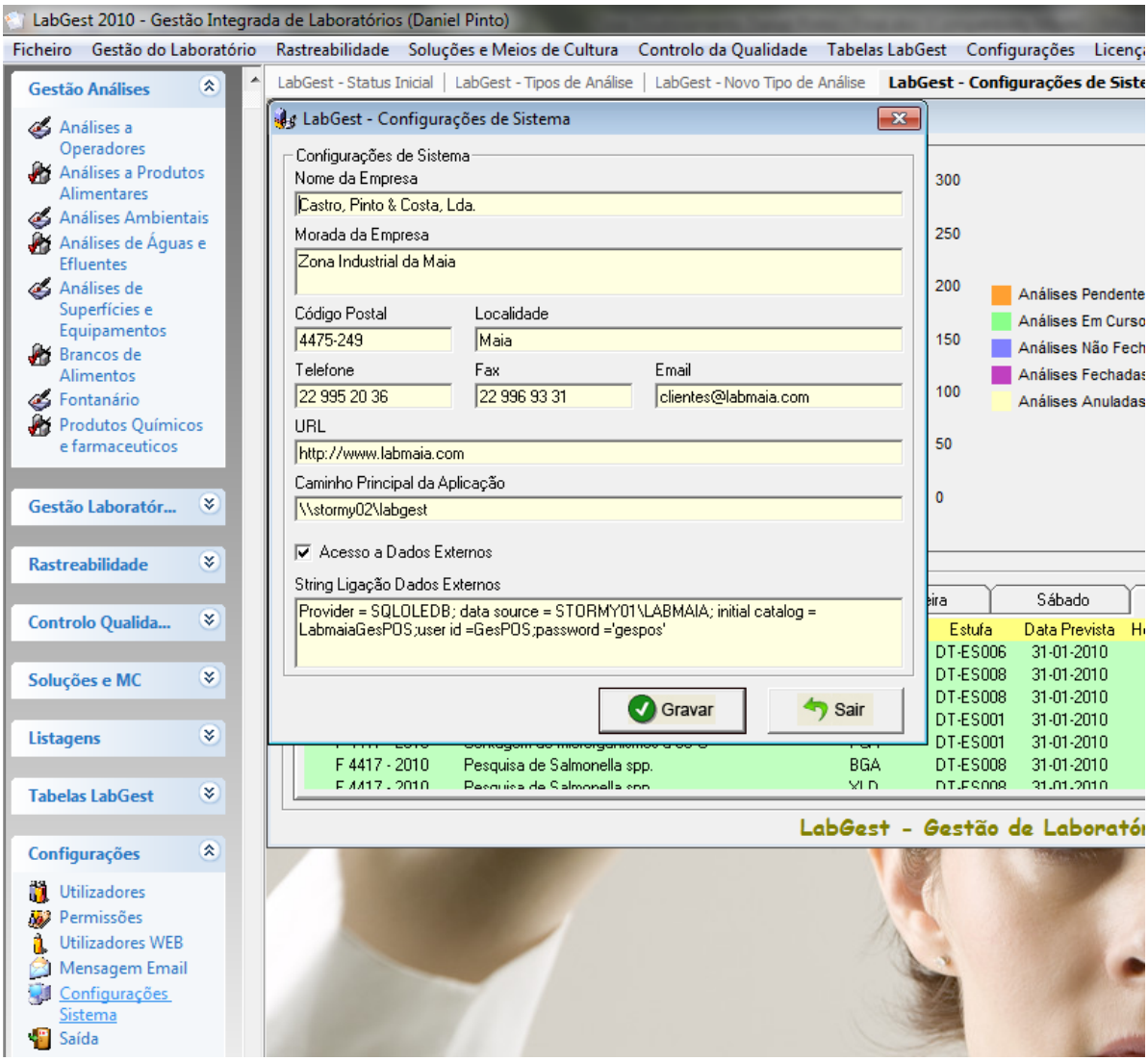


Figure 5.9: System Configuration of external data access

3.1.3. Section "LabGest Tables"

Under Tables section, useful information can be stored which will be used by other sections. Some examples are sampling type; parameters type; analysis type; parameters; parameters families; units and technical opinions about the results. These sections are essential once all the other features of the program use the informations that the user inputs into these tables. Each of the sections, which will be explained in detail further ahead, have the following available options:

- Add New – to add a new record
- Edit – to edit the selected record
- Delete – to mark as inactive the selected record. Note that nothing is deleted from database. It's simply marked as inactive in order to achieve total traceability as requested by NP EN ISO/IEC 17025
- Print – to print a list of what is showed on the screen
- Exit – to exit that particular section

The options presented at Tables section are:

- Sampling Type – define samplings types and configure the sampling operator e-mail in order to receive automatic e-mail information concerning the certificate (Figure 5.10);
- Parameters Type – in order to help to organize information the program can aggregate parameters by types (Figure 5.11) for example "water microbiology";
- Analysis Types – as explain above, this section allow to manager analysis types that will appear in the menu (e.g. water, food products);
- Parameters – one of the most important sections of this menu. This is were the parameter is configurated including the method or standard used, format of results, units and reference value (if applicable).

  Moreover, a mathematical formula can be defined (e.g. calibration curve, average, etc) to allow automated calculation of results. Also, if it is a microbiological parameter it is possible to configure the culture media, dilution for each media and incubation's times (Figure 5.12); It is in this section that a parameter is identified as accreditated ou subcontracted so that this information appears in the test report as required by NP EN ISO/IEC 17025 (Figure 5.12).

- Parameters Families - in order to facilitate reception of samples it is possible to define families by types of product that aggregate typical parameters for those products (Figure 5.13);
- Units – to define units that can be used;
- Technical Opinions – it allows the introduction of several common appreciations of the results in order to simplify the emission of the test reports. When all assay are completed and results validated the Technical Director can choose a pre-defined appreciation which will be included and printed into the test report annex.

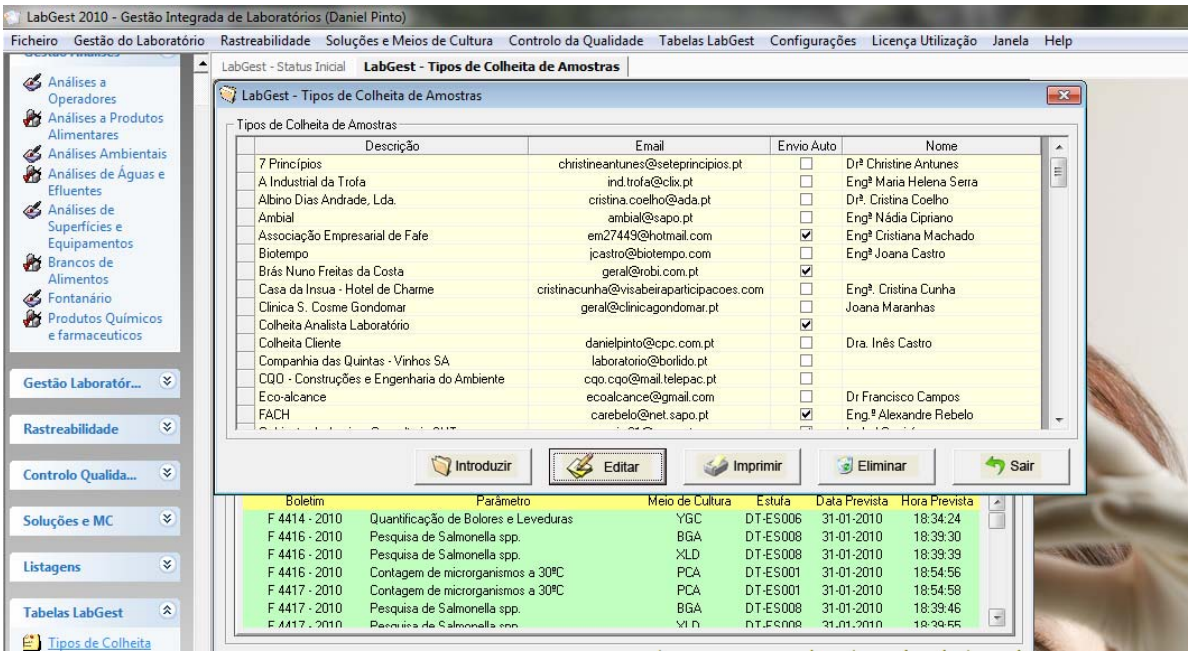


Figure 5.10: Sampling Types

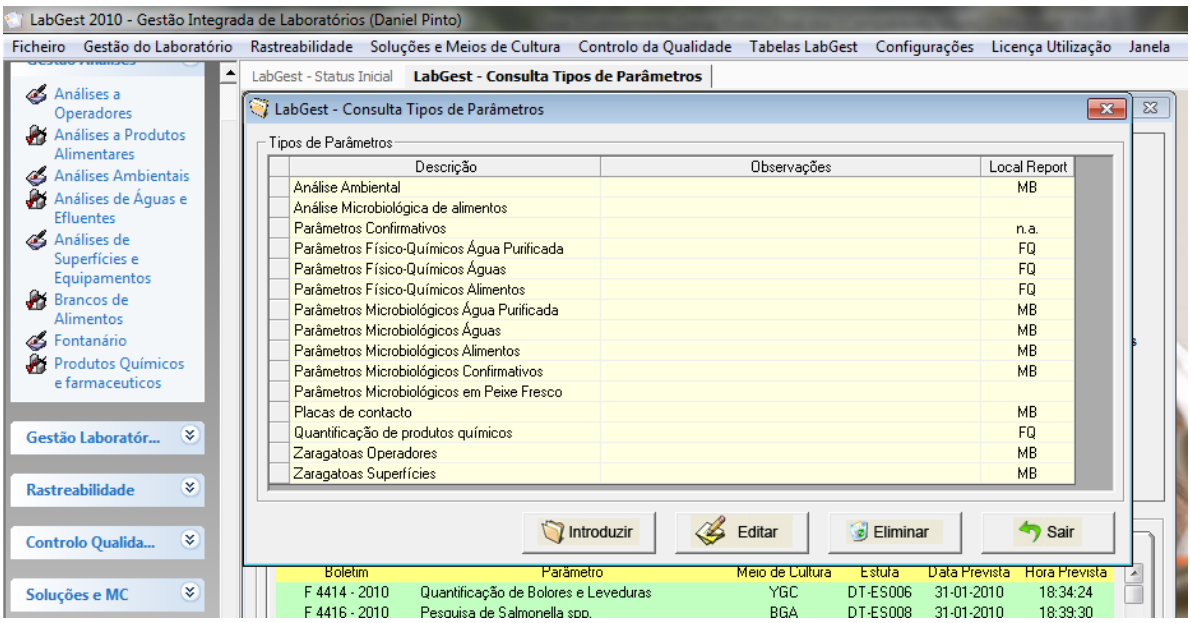


Figure 5.11: Parameters Types

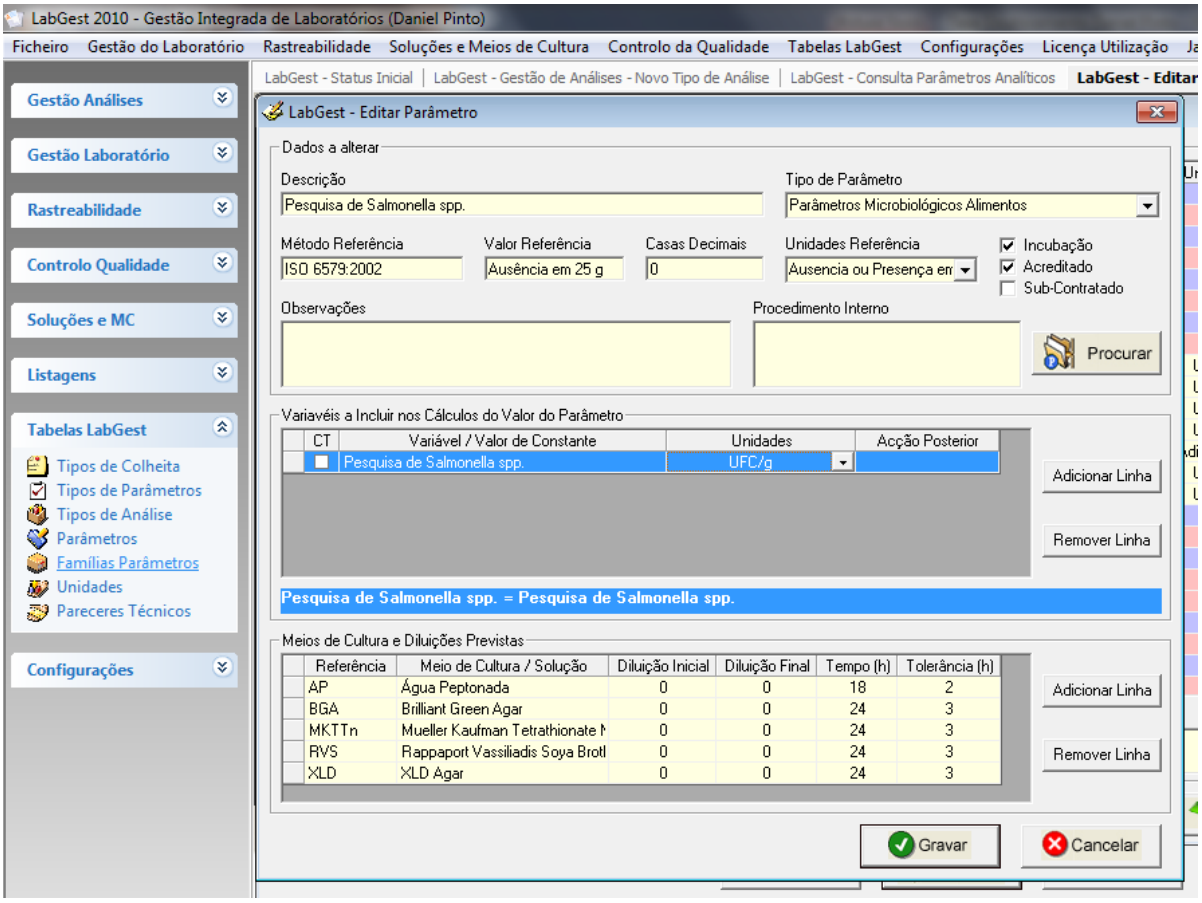


Figure 5.12: Parameter configuration

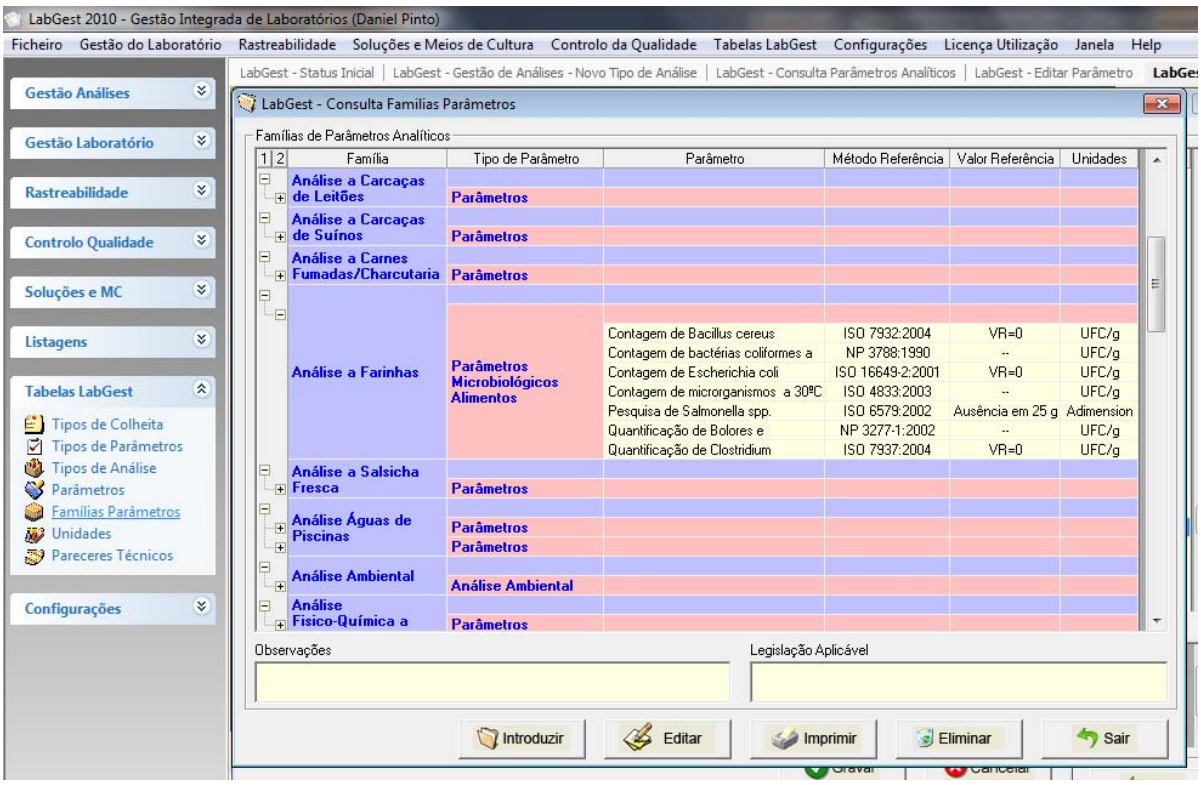


Figure 5.13: Parameters families' configuration

3.1.4. Section "Laboratory Management"

In this section the user has several options at his/her disposal for managing the laboratory, including:

- Customers' Management - allows the input, editing and maintenance of customer data;
- Suppliers' Management - allows the input, editing and maintenance of suppliers data;
- Planning – This function is helpful when there are pre-defined dates for sample reception. It is possible to input the date of sampling and reception and also the parameters to be analized simplifying the reception step and also contributing to better organize lab work once it is possible to visualize the incoming samples. Duplication of samples changing only arrival date is also available.
- Management of Test reports – helps to organize the sending of test reports to the client by traditional mail or e-mail (Figure 5.14);

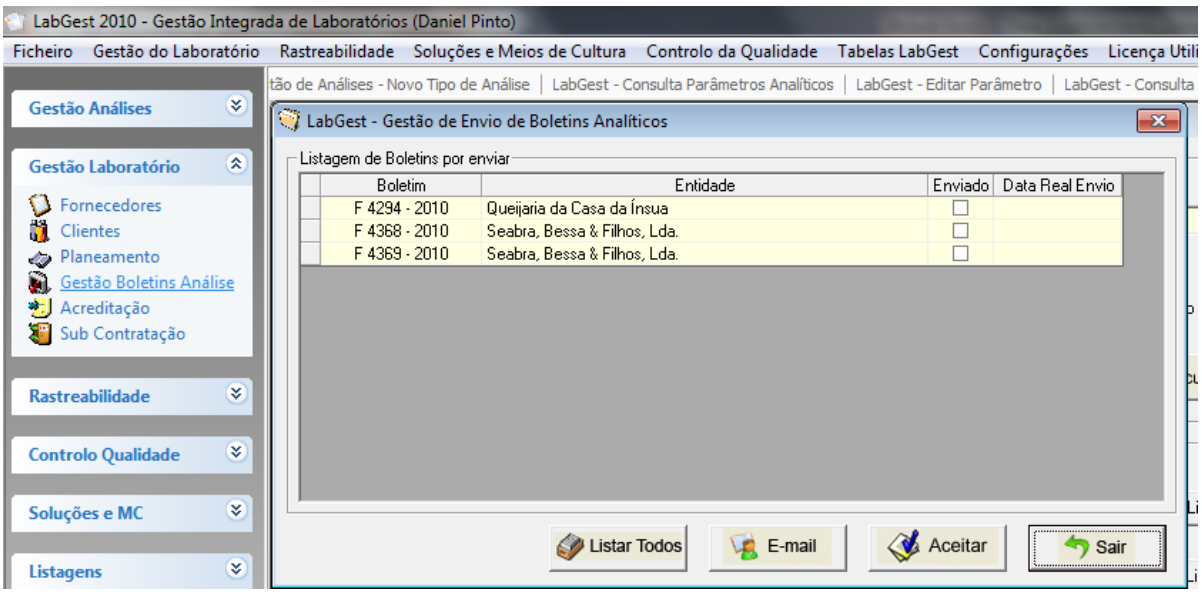


Figure 5.14: Certificate management form

- Accreditation - the software allows information on parameters that are accredited to NP EN ISO/IEC 17025 so that the overall program can correct the certificate of analysis (with or without accreditation symbol). This is a mandatory requirement for NP EN ISO/IEC 17025 (Figure 5.15);

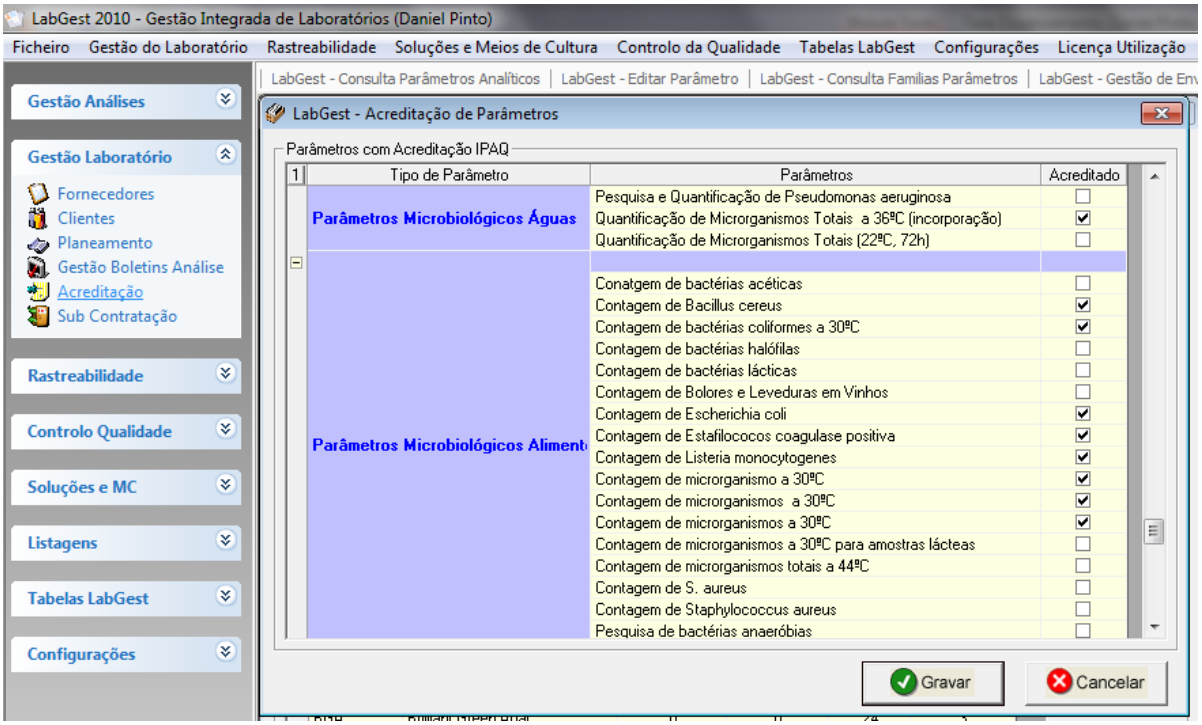


Figure 5.15: Parameters accreditation form

- Sub-contracting - another requirement of NP EN ISO/IEC 17025, which requires that the laboratory inform customer of the tests that have been sub-contracted to other suppliers. By using the options available at this section, software LabGest® refers sub-contracted of tests on the test report (Figure 5.16).

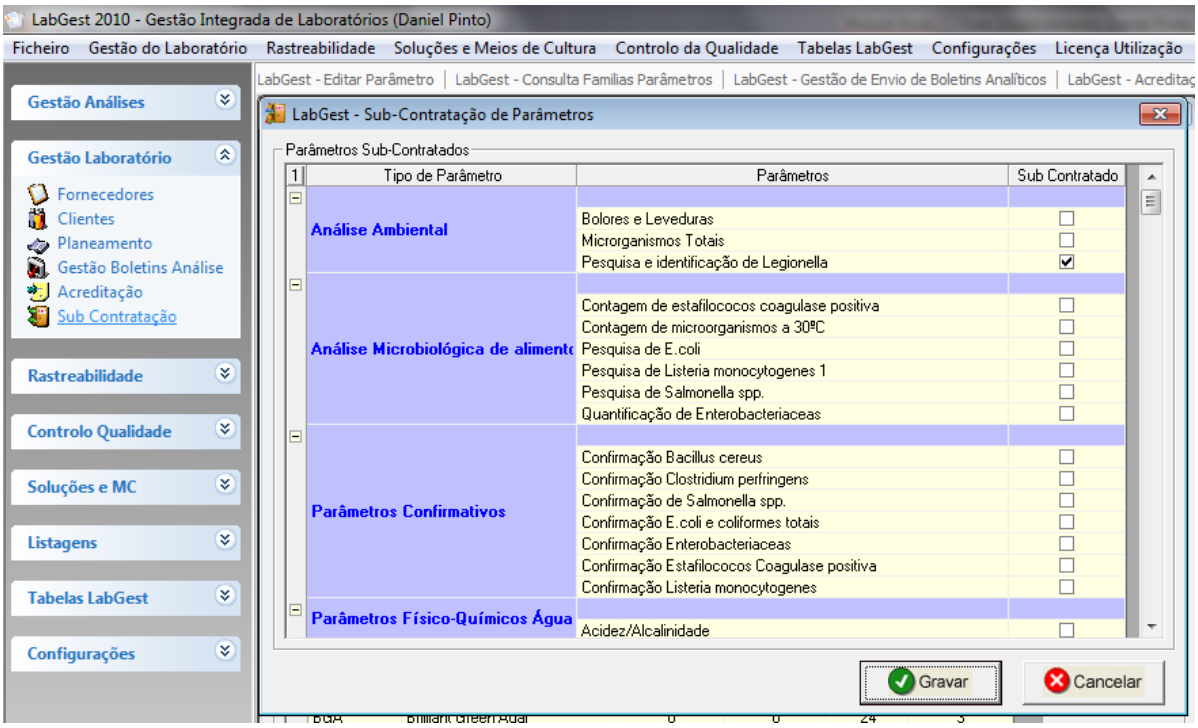


Figure 5.16: Subcontracted parameters form

3.1.5. Section "Analysis Management"

On a daily basis it is important to easily know which are the samples waiting to be processed, the ones being analized and the ones that were already sent to the Clients. In this section there are five tabs based on the status of a particular analysis (Figure 5.17). In the first tab it is possible view received samples but not yet under analysis. In this tab, new analysis can be inserted or edited, print labels for identifying the sample and print reception confirmation sheet. There is also the possibility to accept or reject a sample for analysis. By accepting, the sample in question is going to be transferred for the ongoing analysis tab. In parallel to this action of accepting the sample, the software, using stored procedures in database, calculates, in the case of microbiological parameters, based on the definitions of the parameters, the necessary data for incubation (culture media, temperatures, time and dilutions) which will be used in the traceability/incubation section. To facilitate the use of the software, upon acceptance of a given sample, the software allows the acceptance of

several simultaneously. Finally, it is possible for the operator of receipt of the sample import photos of it (Picture command).

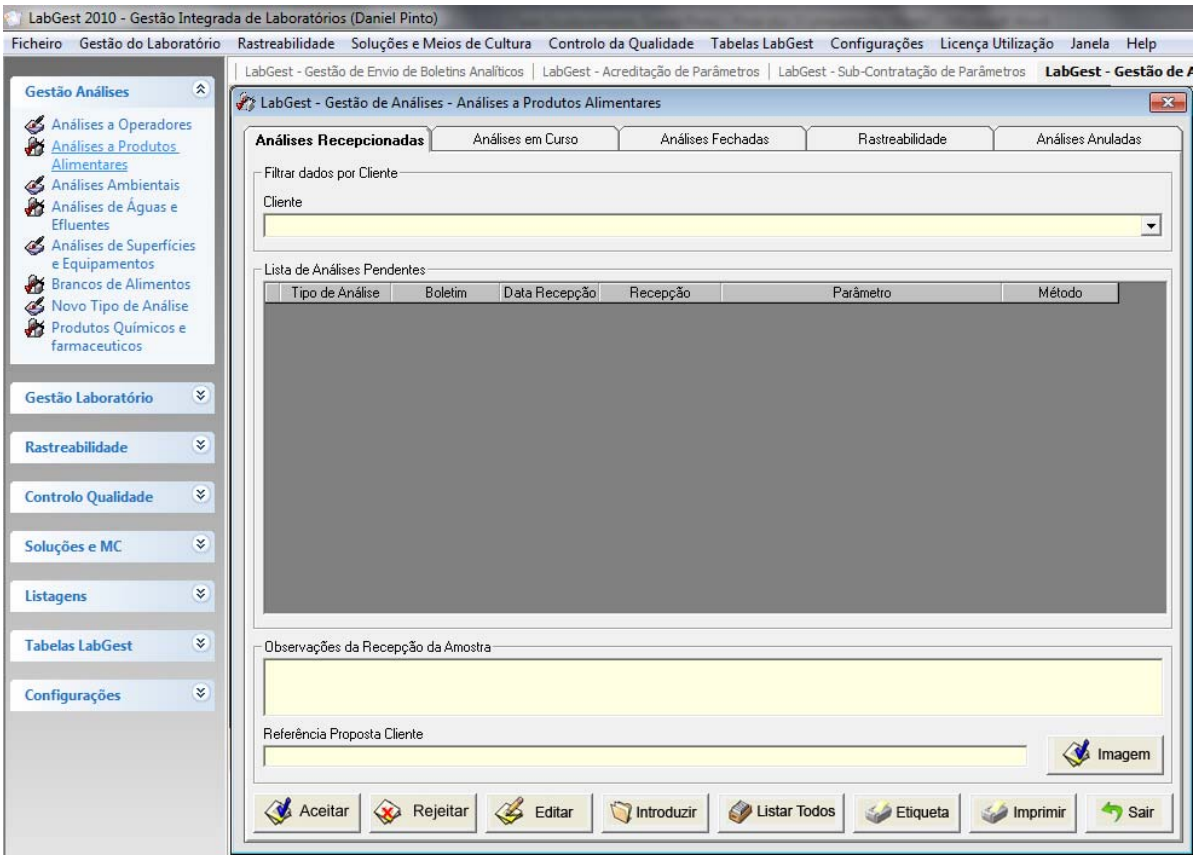


Figure 5.17: Analysis Management form

To introduce a new sample click add new and it apperars the form of a new sample. The introduction of new sample for analysis follows three basic steps: the first which identifies general data of the sample (Figure 5.18) as a customer reference, description of the sample, temperature of receipt, date of receipt, date of sampling, date of delivery of results and other customer comments. This information is required by NP EN ISO/IEC 17025 once several parameters have a maximum time between sampling and the beginning of assays. Moreover, if there are some relevant aspect which may be useful for the interpretation of results, that should be written at the observation field.

When a new sample is introduced two options may be activated:

- Option of planning - in order to plan reception of sample over time; The calendar is activated when this option is selected;
- Option of registering in analysis in serial mode – the program can make copies of general data (e.g. Customer information) and make several samples in serie

so that will not benecessary to repeat the same data, thus reducing significantly time spent in sample insertion.

- Sampling plans associated with microbiological criteria often require five (*n*=5) or more samples (to determine acceptability of lots) which have the same information and by activation of serial insertion in less then 2 minutes is possible to feed samples to the lab accompanied by the data sheets and labels.

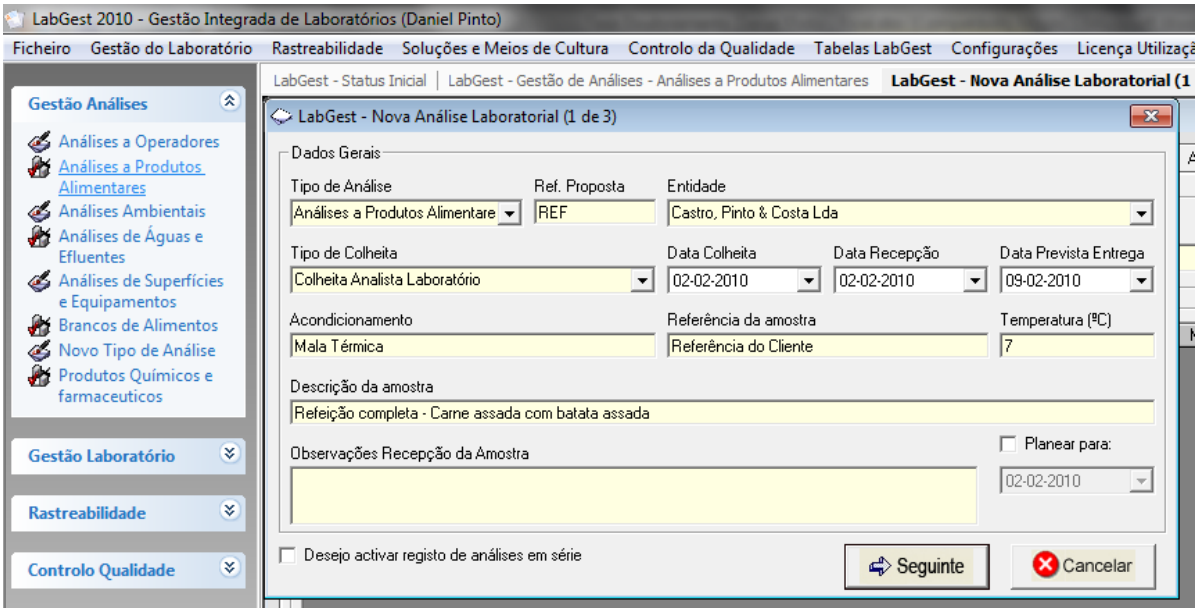


Figure 5.18: Insert of a new analysis (step 1 of 3)

In the second step of sample insertion it is possible to select options that are predefined for that type of analysis, namely for water analysis: the origin of the water (tap water, well, mine); treatment if applicable; use of the water: human consumption, process, gardening, etc.

Finally, the third and final stage of introducing a sample, the operator selects the parameters to be analyzed. It is possible either to select an individual parameter (Figure 5.19) or a family of parameters (previously defined in the configuration section) which the software automatically interprets (Figure 5.20).

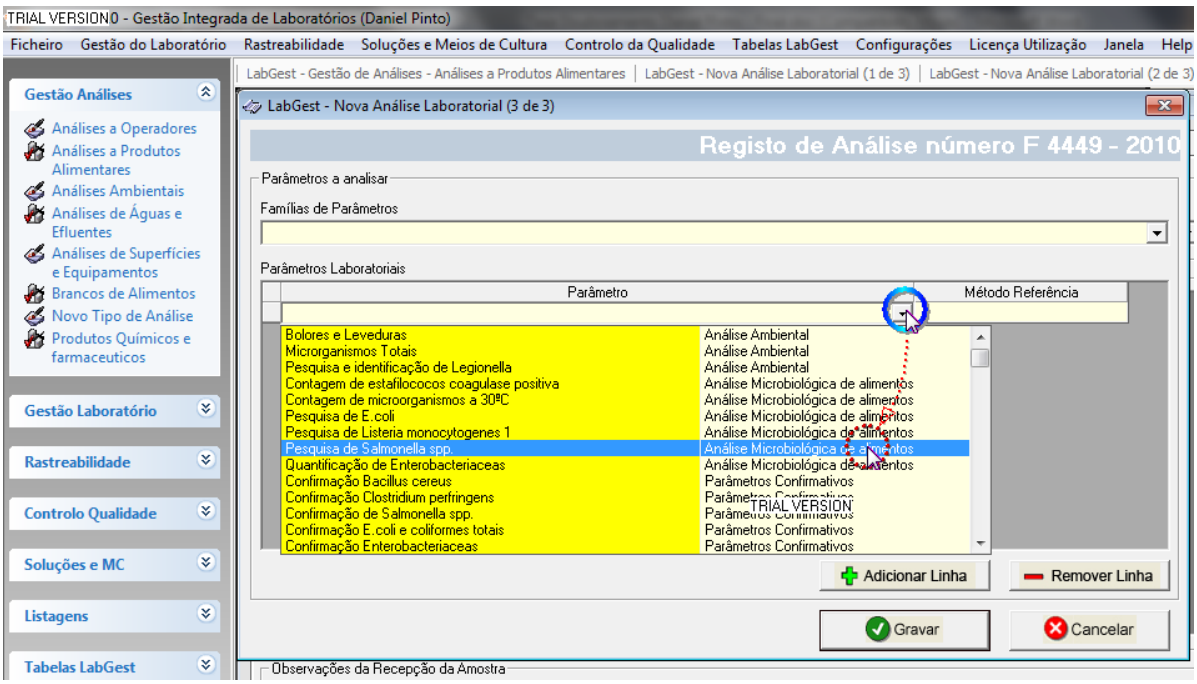


Figure 5.19: Insert of a new analysis (step 2 of 3) – select and individual parameter

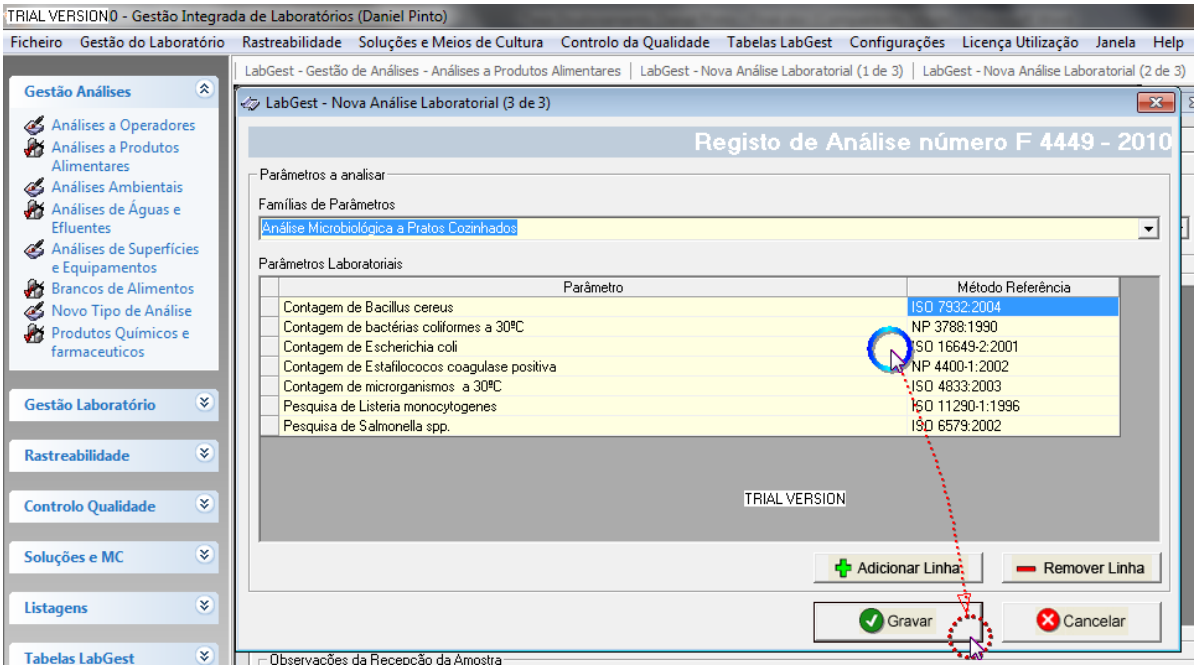


Figure 5.20: Insert of a new analysis (step 3 of 3) – select a parameter family

Now, the sample that we have just inserted appears on the received analysis tab (Figure 5.21).

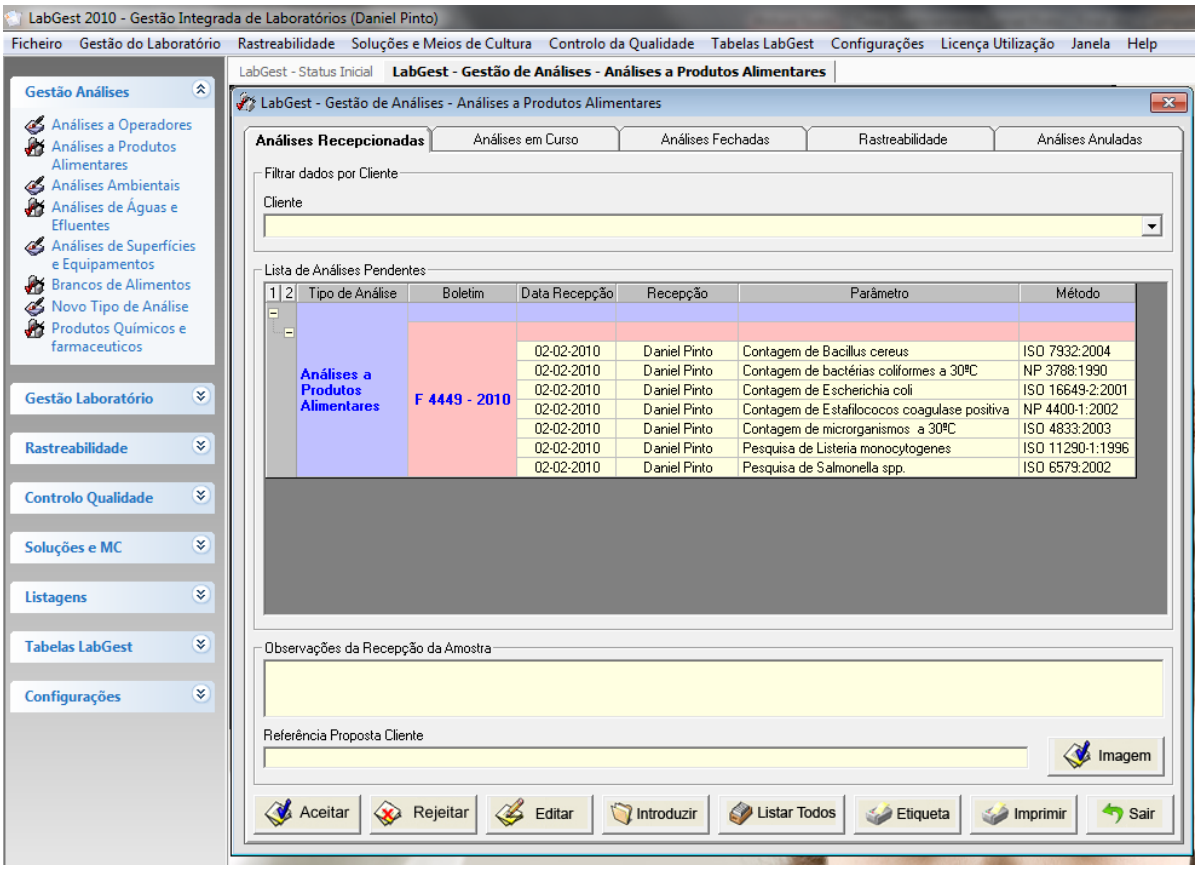


Figure 5.21: Received analysis tab

As shown in Figure 5.22 if the sample is accepted, it is transferred to the ongoing analysis tab; simultaneously, the program allows printing the "results sheet" in which the primary results will be written by the anslists.

Also, in the ongoing analysis tab, the operator can perform several actions (always depending on permission issued by system administrator):

- Close – to close the analysis and issue the certificate;
- Print – to print, as explained above, the "results sheet";
- Labels – if there are microbiological parameters, it's possible to print labels for petri dishes in order to correctly identify the plates in laboratory;
- Edit – to add or remove parameter or change general sample information;
- Delete – to delete the selected parameter from analysis;
- Picture – to insert picture of a result, for example a picture of a microscopic field;

- Confirm – to add test needed to confirm a presuntive result.

As shown in Figure 5.23, general information of the sample, parameters and also several blank fields (to be completed by analists) are printed on the sheet. There is also the possibility to read a determined parameter in order to input results to the program using barcode readers.

As soon as a sample is received an autonumber will be associated with it being the number of the final test report.

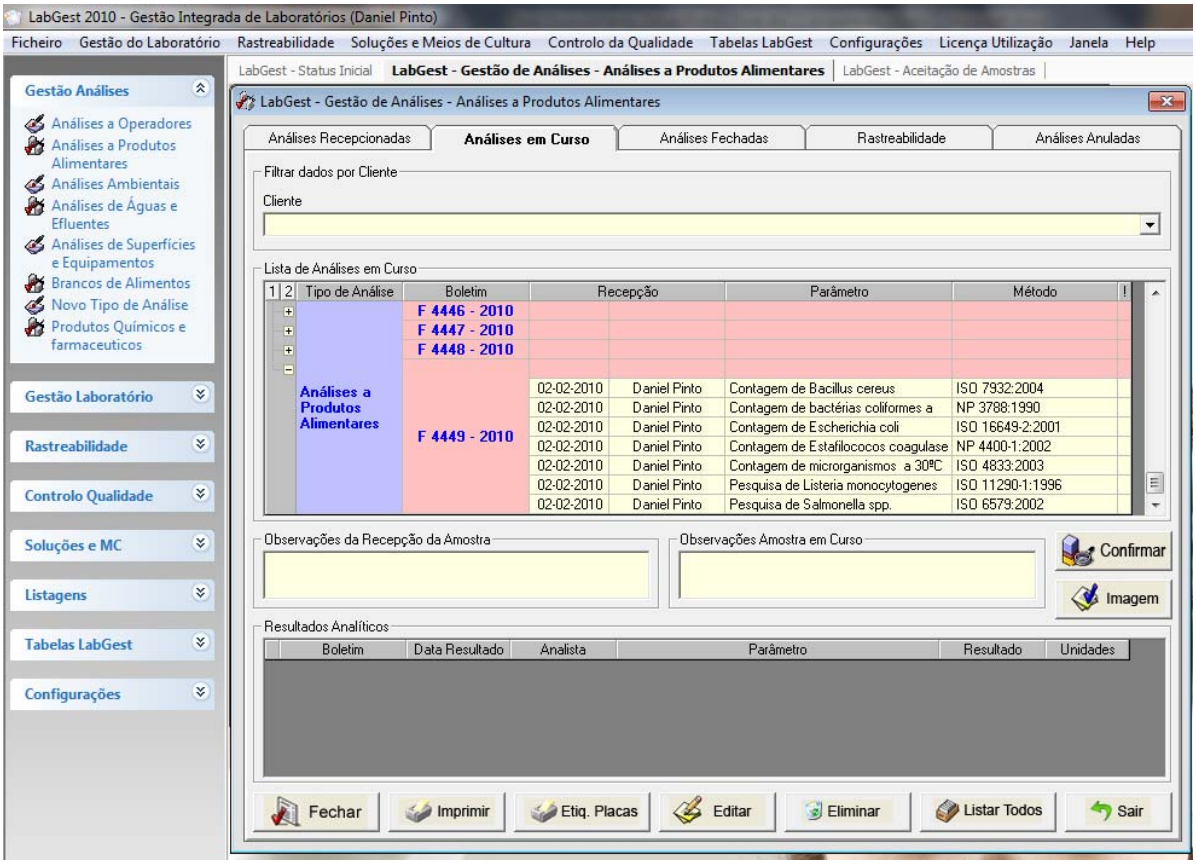
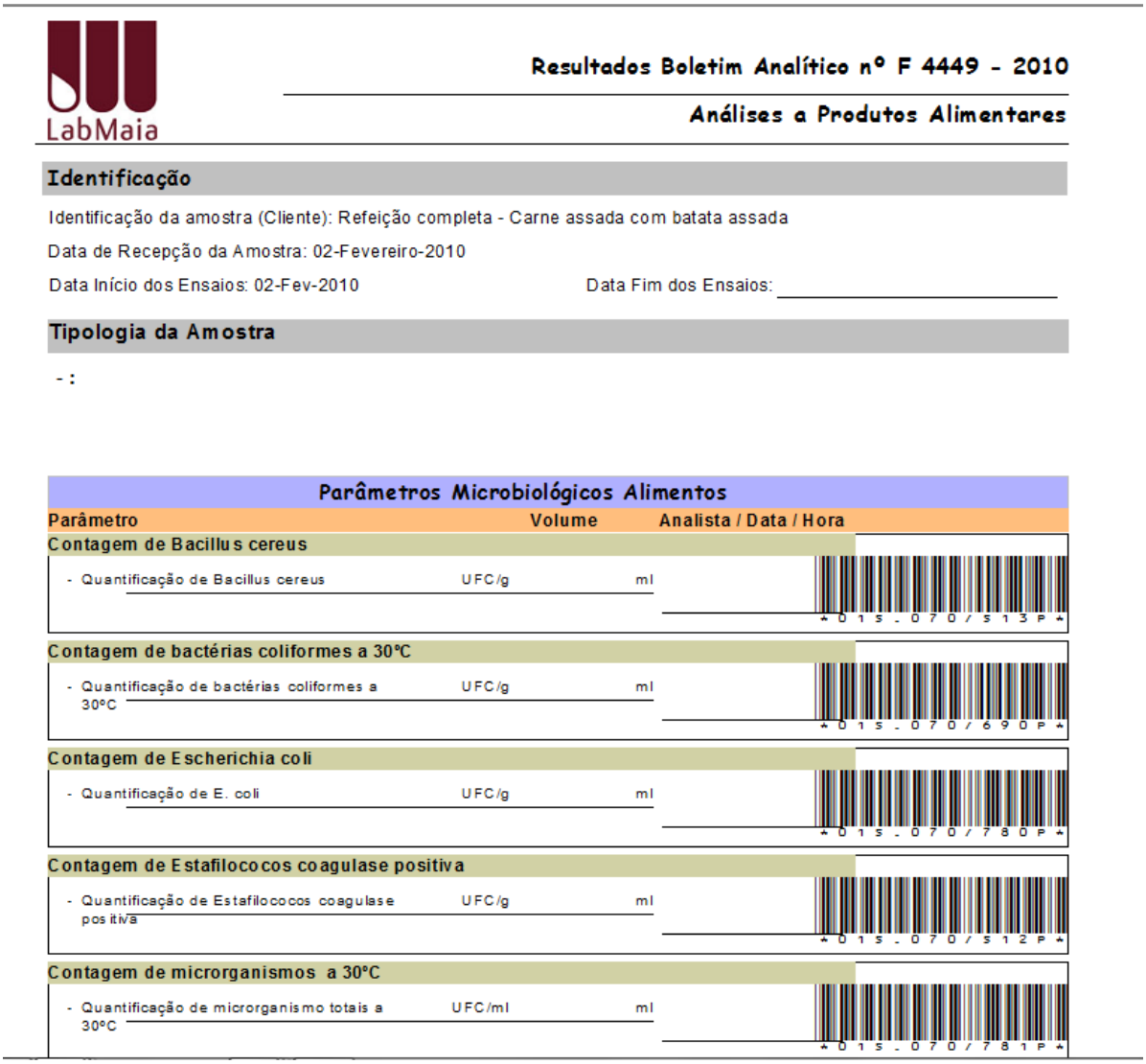


Figure 5.22: Ongoing analysis tab



LabMaia

Resultados Boletim Analítico nº F 4449 - 2010

Análises a Produtos Alimentares

Identificação

Identificação da amostra (Cliente): Refeição completa - Carne assada com batata assada

Data de Recepção da Amostra: 02-Fevereiro-2010

Data Início dos Ensaios: 02-Fev-2010 Data Fim dos Ensaios: _____

Tipologia da Amostra

- :

Parâmetros Microbiológicos Alimentos

Parâmetro		Volume	Analista / Data / Hora
Contagem de Bacillus cereus			
- Quantificação de Bacillus cereus	UFC/g	ml	
Contagem de bactérias coliformes a 30ºC			
- Quantificação de bactérias coliformes a 30ºC	UFC/g	ml	
Contagem de Escherichia coli			
- Quantificação de E. coli	UFC/g	ml	
Contagem de Estafilococos coagulase positiva			
- Quantificação de Estafilococos coagulase positiva	UFC/g	ml	
Contagem de microrganismos a 30ºC			
- Quantificação de microrganismo totais a 30ºC	UFC/ml	ml	

Figure 5.23: Ongoing analysis sheet

To introduce results it is as simple as a double click on the analysis and it pops up the form for its registration (Figure 5.24).

Depending on the defined formula for a given parameter (please refer to section parameters configuration) the use must introduce one or more values that will be asked by LabGest®. There is also the possibility of updating the formula which may be useful is a calibration curve changes during the time the sample is under analysis.

In order to meet the requirements of NP EN ISO/IEC 17025, namely traceability and technical validation of analists, it is also mandatory to introduce for each parameter the analyst who carried out the assay. If a sample is still incubated it is impossible to introduce results. Again, at this stage can be inserted an image associated with a particular result.

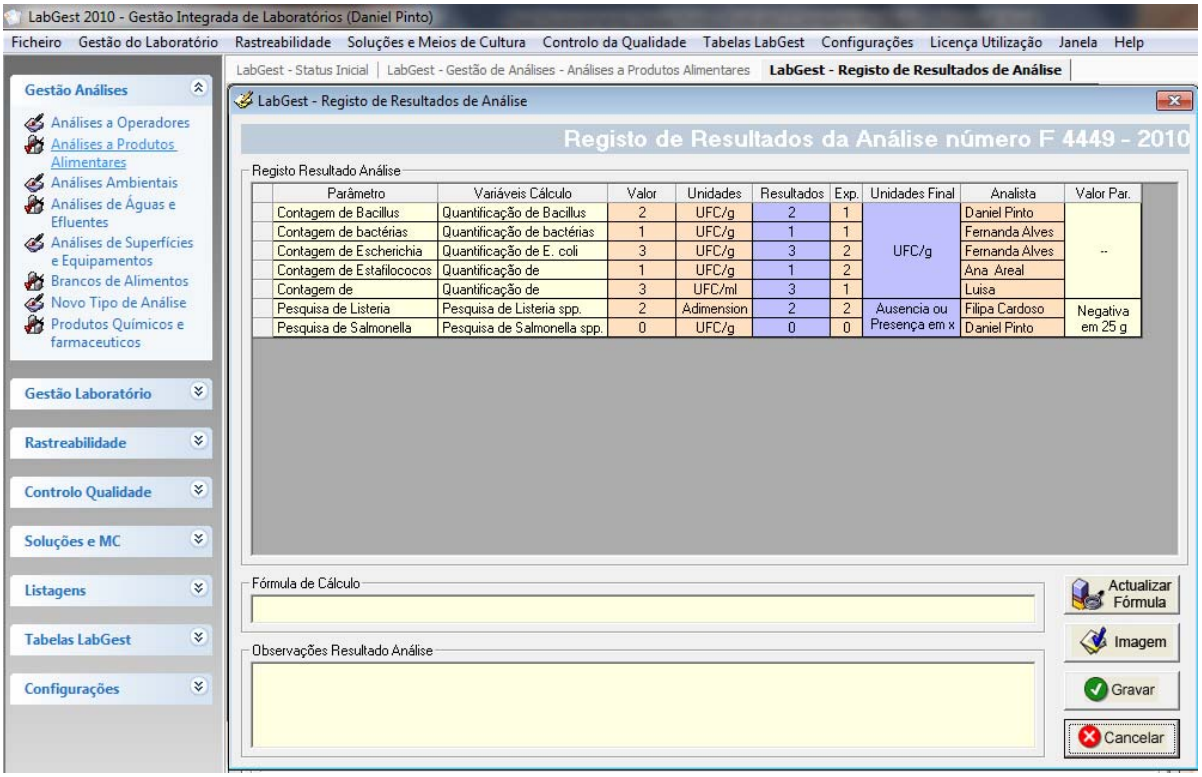


Figure 5.24: Record Analysis Results

When we save the results we go back to the Ongoing Analysis tab (Figure 5.25) and if you click on the analysis number can see the results obtained so far. Whenever is necessary to add new results just double click and repeat the procedure. The analysis is editable until the close button is activated.

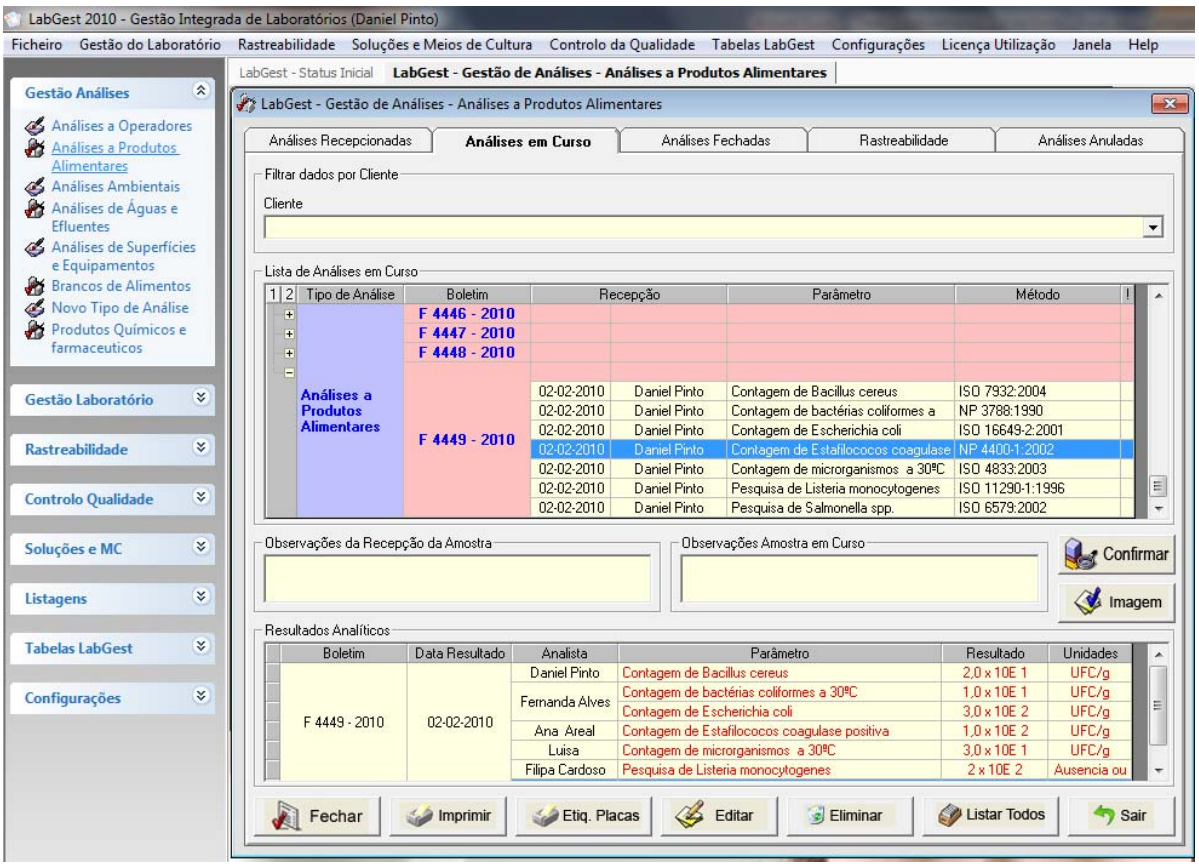


Figure 5.25: Ongoing analysis tab with results entered

When a result is registered the software compares it with a reference value (introduced in parameter configuration as explained above), if it exists, and the non-conform results are shown in red (Figure 5.25).

When all the results are completed, just by clicking "Close" the analysis report appears allowing the introduction of an opinion by the Technical Director of the Laboratory (Figure 5.26). There are several pre-defined sentences (please report to configurations section) and it is also possible to display the results and reference values (Show Details) to facilitate the interpretation of results.

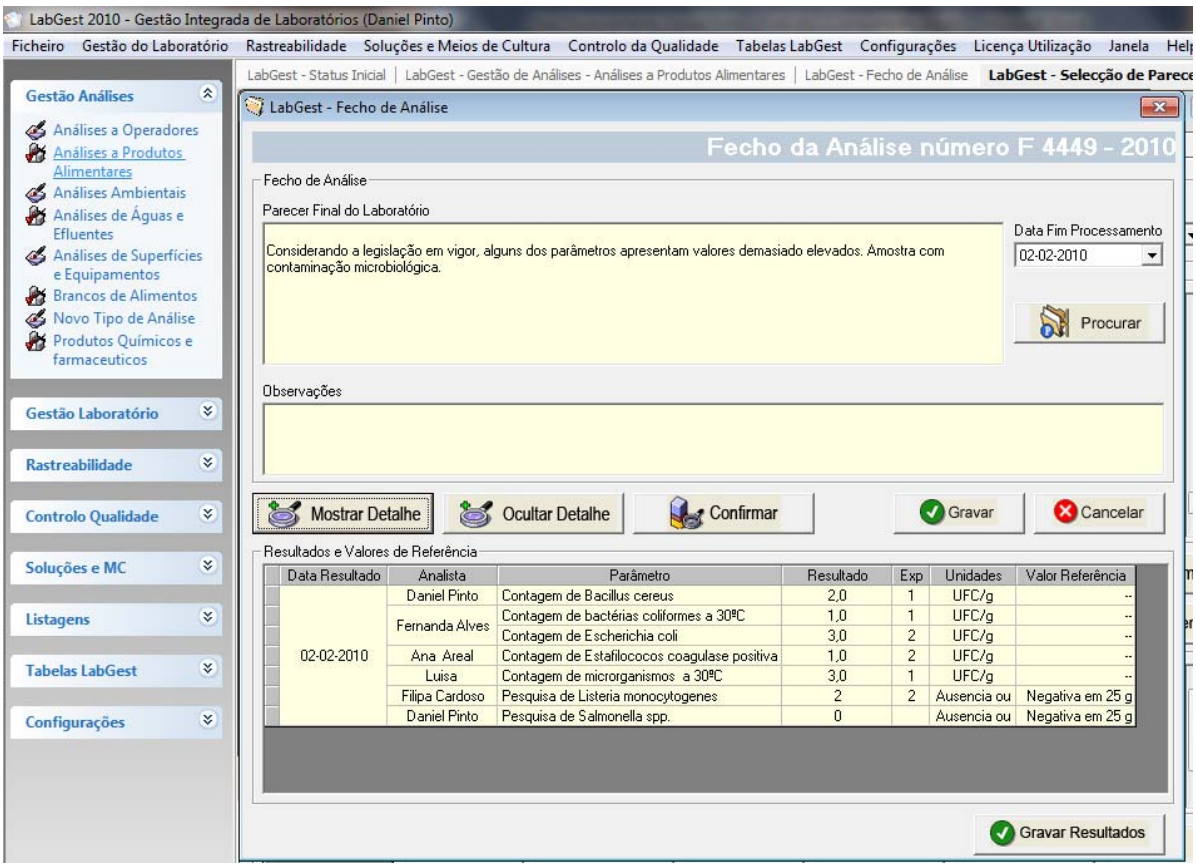


Figure 5.26: Close Analysis Form

The Techical Director may refine results (e.g. introduce < QL when the result is lower then the quantification level) by changing directily into the table results and by clicking "Save Results". When the Technical Director press "Save" the analysis report is closed and the analysis is transfered to the closed analysis tab. In this tab, there are several options available:

- Reopen – To transfer the selected analysis to the ongoing status;
- View – to view, if already generated, the analysis certificate;
- Certificates – to generate several (serial mode) analysis certificate;
- PDF certificate – to generate the selected analysis sertificate.

It is also possible to search a analysis number or to filter data by several available options.

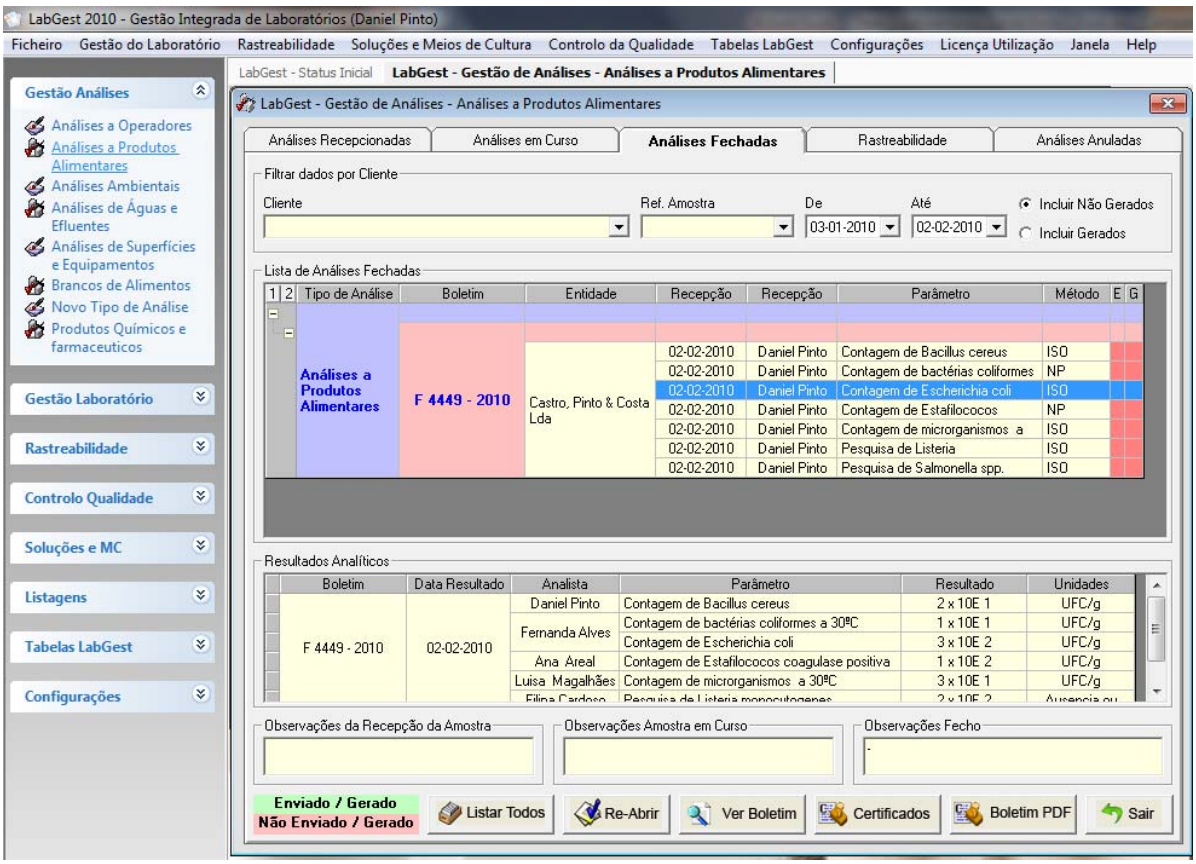


Figure 5.27: Analysis Management form – Closed Analysis Tab

When the "PDF Certificate" button is pressed, the Test report is generated in PDF format. It is saved at the main path identified at the System Configuration Section and is sent by e-mail to the customer. All this actions ends when the generated certificate opens (Figure 5.28).

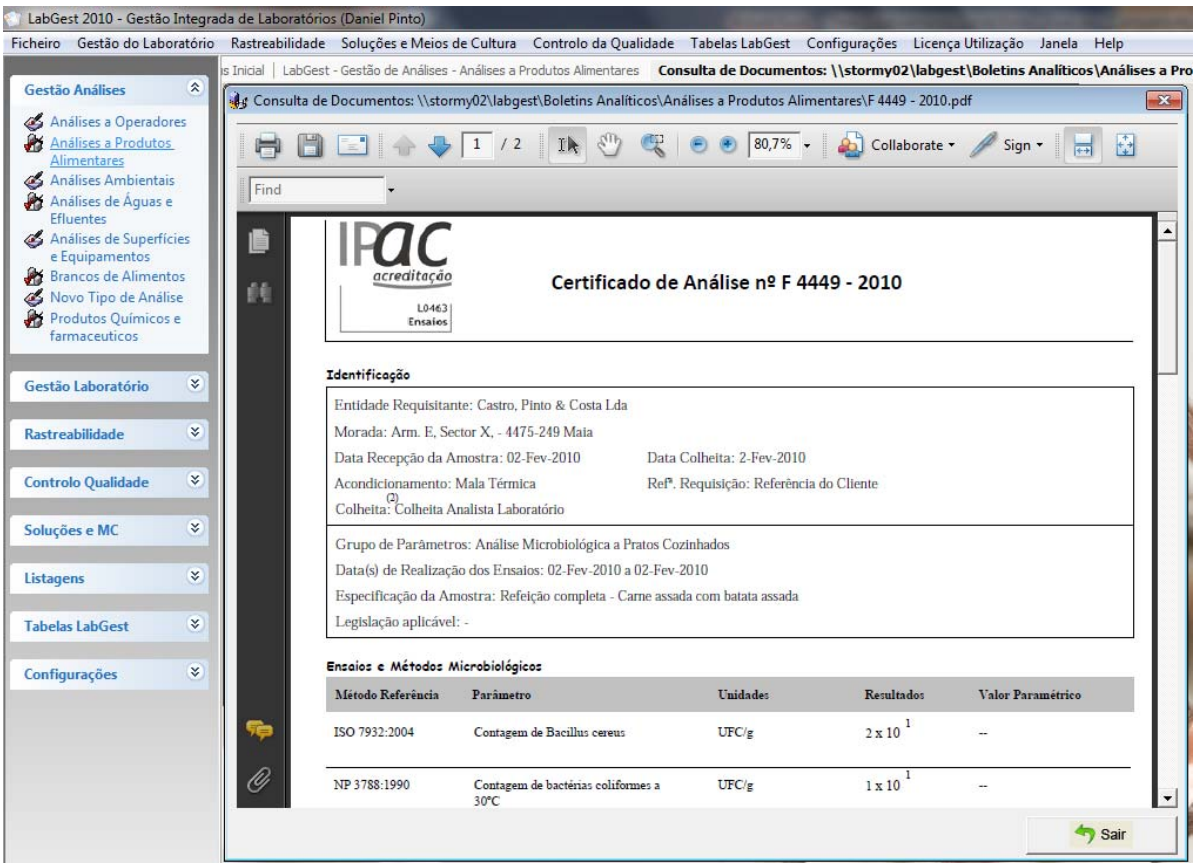


Figure 5.28: Generating analysis certificate

NP EN ISO/IEC 17025 requests that a traceability methodology is implemented in order to control Test reports versions. LabGest® complies with this requirement by creating a new version of the Test report each time an analysis is reopened. The information about report's versions is saved in the database and printed at the botton of the Test report. If you press the "view" button it appears a form with a table showing the different Test reports versions.

3.1.6. Section "Solutions and Culture Medium"

The objective of this section is to help managing the reagents (solutions, culture media, etc.) purchasing, production and quality control.

Purchasing Services and Supplies addressed in NP EN ISO/IEC 17025 refers that the quality of incoming material should be verified against predefined specifications and traceability must be maintained.

Laboratory have several options under this section that allow to complie with several NP EN ISO/IEC 17025 requirements, namely the ones who refer traceability. Being so, the laboratory can:

- Reagents – to manage reagents information, including expiration date, lot, etc;
- Regents stocks – manage reception of reagents and stocks. Perhaps the most important feature of this section is the reagents reception (Figure 5.29). This is made automatically by the attribution of a sequential number, the reception number (RN), by supplier lot which is the internal lot (auto-number). To easily guarantee the reagents traceability it is advisable to attribute a new number (auto-number) for each supplier lot even if it corresponds to the same delivery. Furthermore, this sequential number will be used to maintain traceability and stocks (Figure 5.29). Also, when a reagent is received several quality parameters (transport temperature, expiration date, etc.) are check and it only enters into stock if there are not non-conformacies.

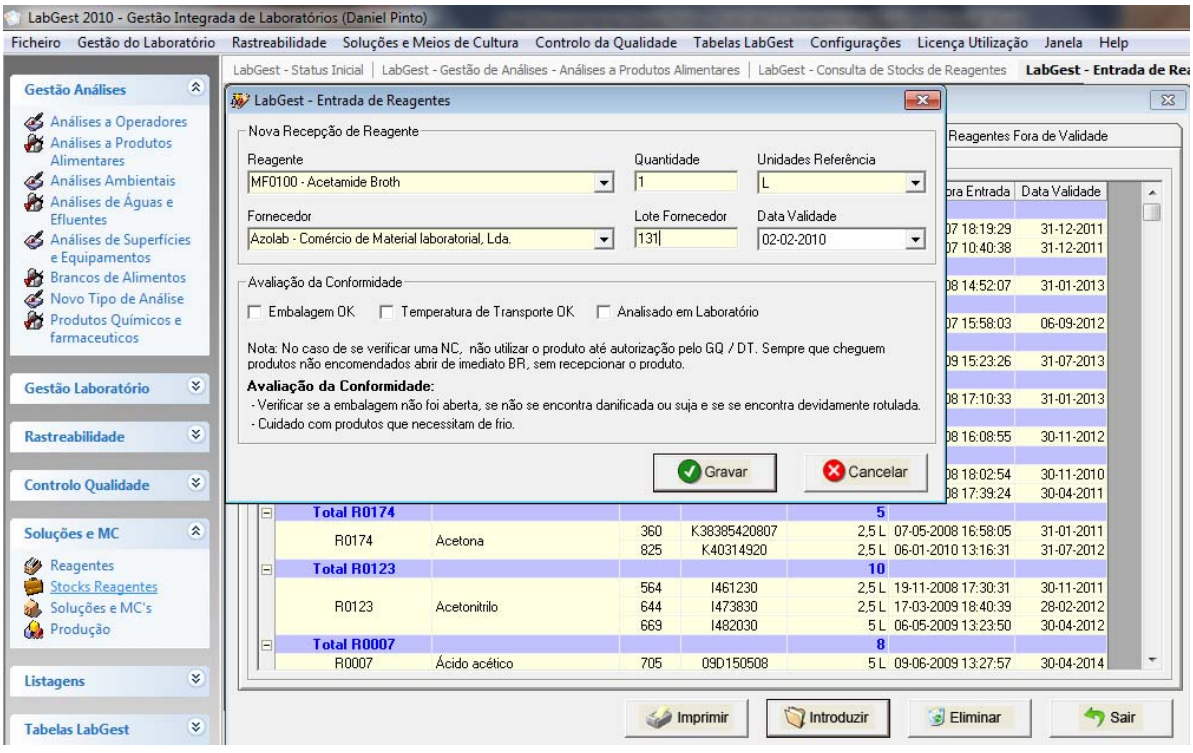


Figure 5.29: New reagent reception

- Solutions and CM – at this section, the laboratory can configure culture medium and checmical solutions formulation. Laboratory also configures, at this section,

quality control paramaters of that particular culture medium or chemical solution.

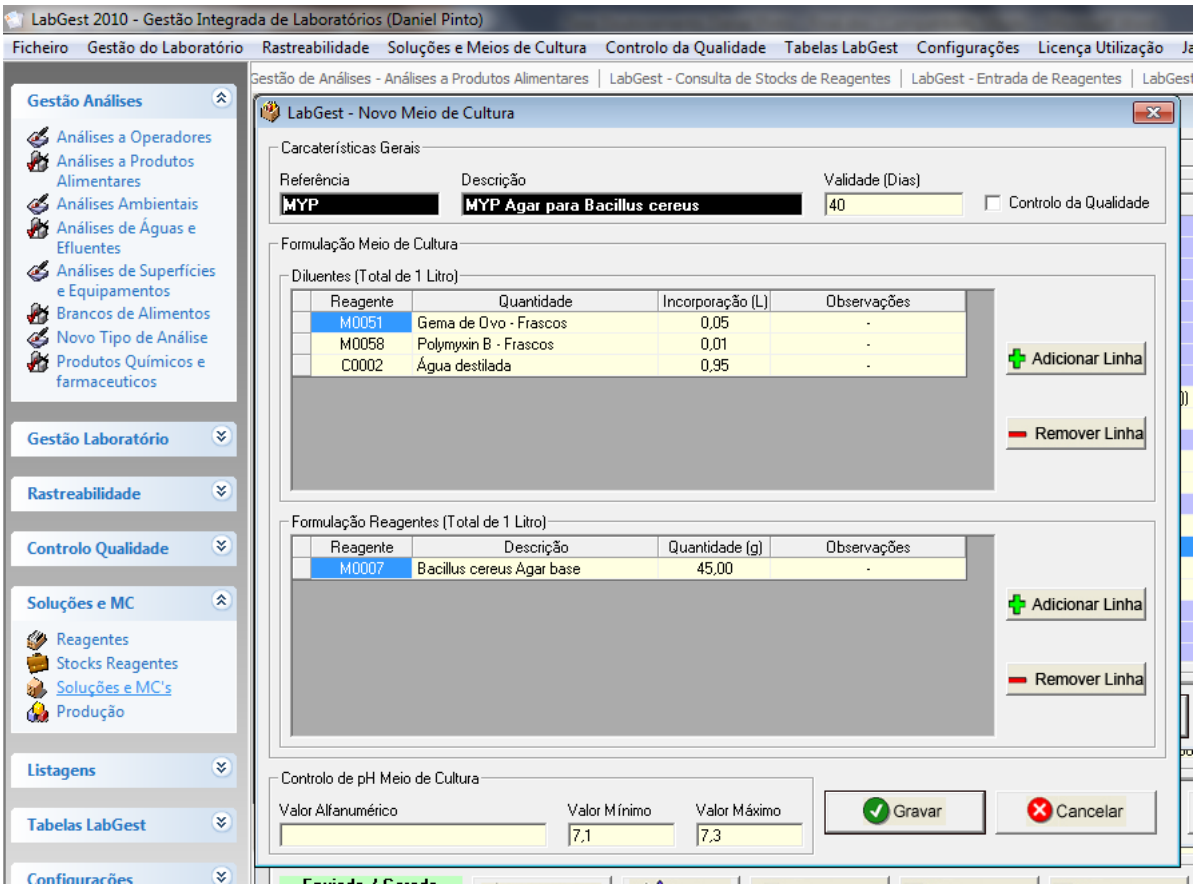


Figure 5.30: Culture medium formulation configuration

- Production – Production orders (Figure 5.31) automatically indicate the reagents type, quantity and suggest the reagents internal lot to be used as well as define the lot number (auto-number). The suggestion of the internal lot is made by a very complex algorithm that calculates raw-materials distribution according to FIFO (First in – First out) and expiration dates. If there are not enough reagents an error message will be generated with the information of the missing quantity of the reagent in question. The production order is a printable output used by the laboratory production section as a registry.

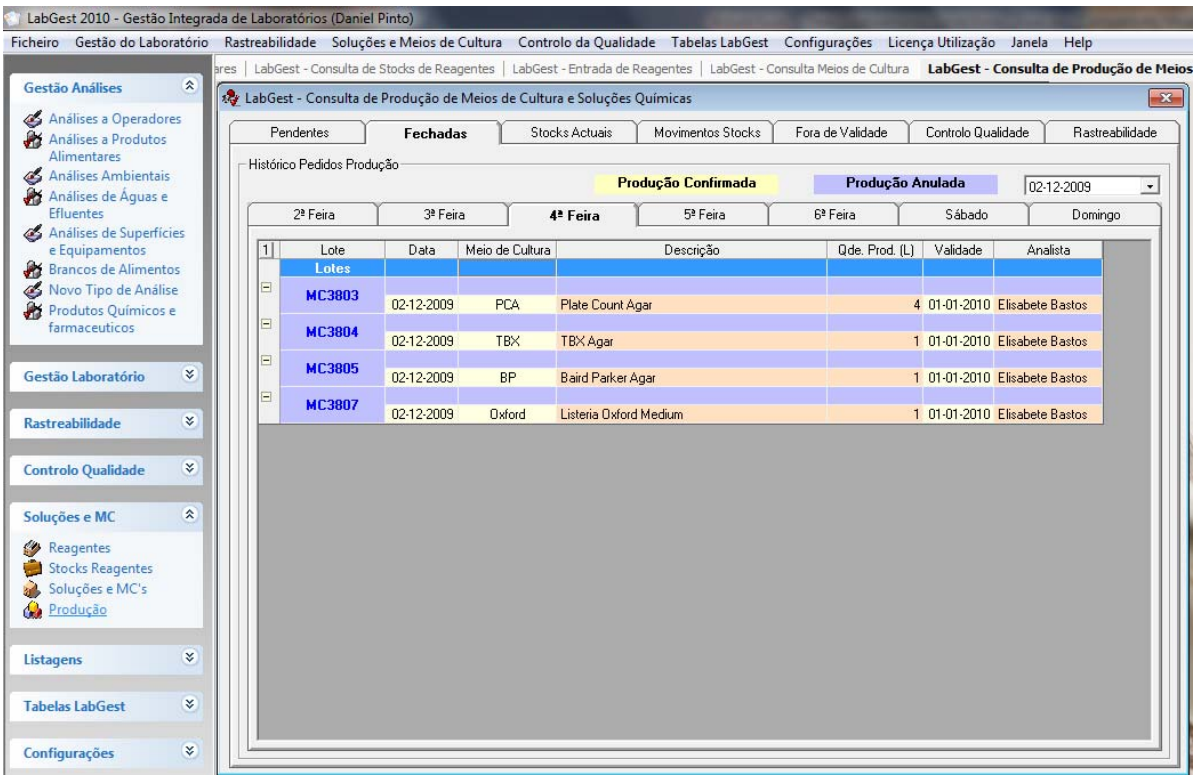


Figure 5.31: Closed production orders

The production operator should write down in the referenced paper form the exact quantity of the used reagents and the correct internal lot when there are changes to the original order.

After confirmation of the exact reagent consumption the production manager closes the production order. It is at this time that reagent stock movement is finally saved and the solution or culture medium becomes available to be used at the incubation section.

Also, in this section is possible to trace back all the production process and results of quality control performed to a culture medium. To do so, press the traceability tab and select a culture medium or a specified lot to view traceability information (Figure 5.32 and Figure 5.33).

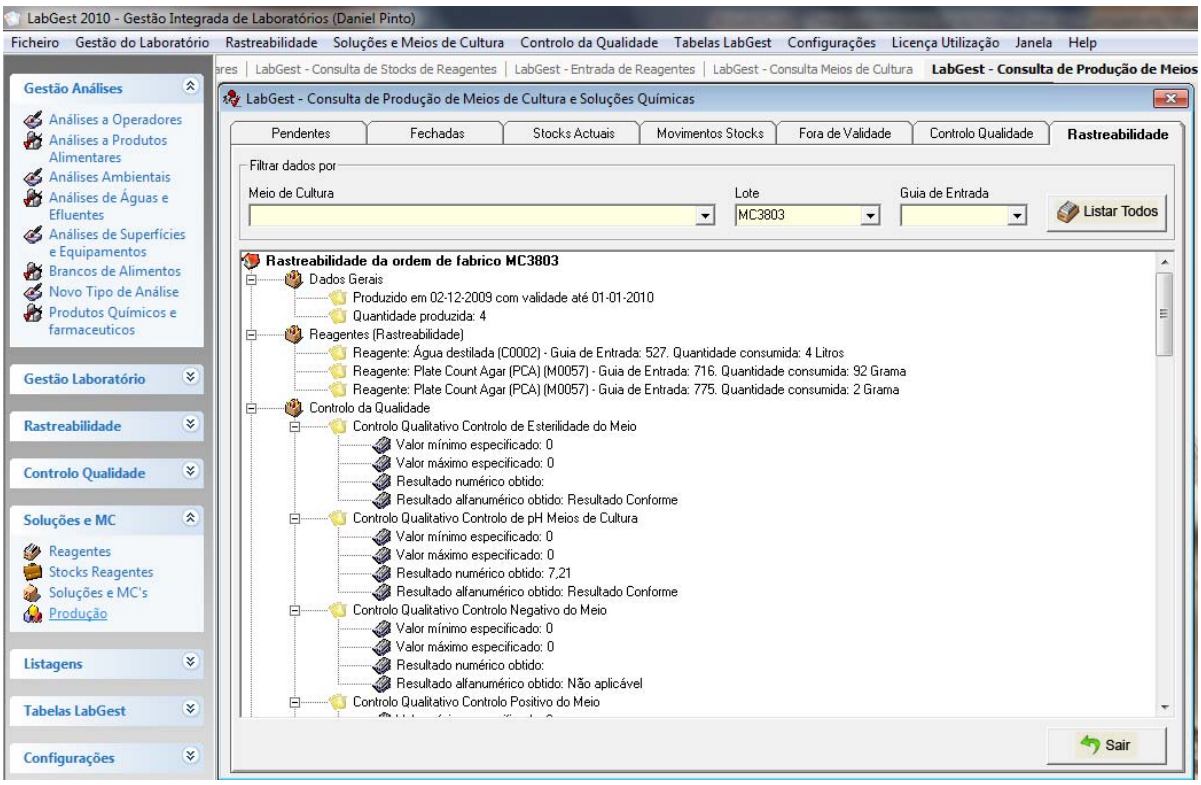


Figure 5.32: Culture medium traceability information

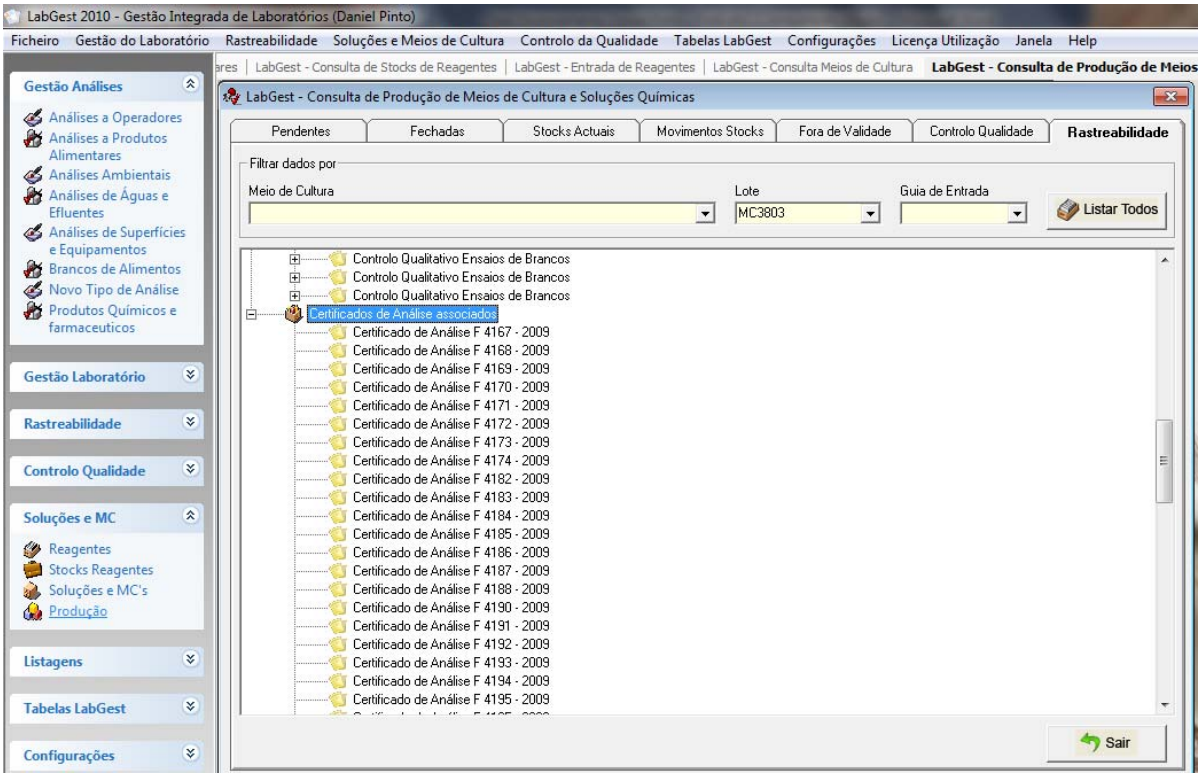


Figure 5.33: Culture medium traceability information (continue)

3.1.7. Section "Traceability"

After configuration of the culture medium and after the activation of the Incubation option, it is possible to open the section regarding global laboratory traceability. In this section, laboratory users can configure incubators and temperatures. At the temperatures section, LabGest® is prepared to connect to a temperature sensor in order to monitor the incubator temperature automatically Figure 5.34).

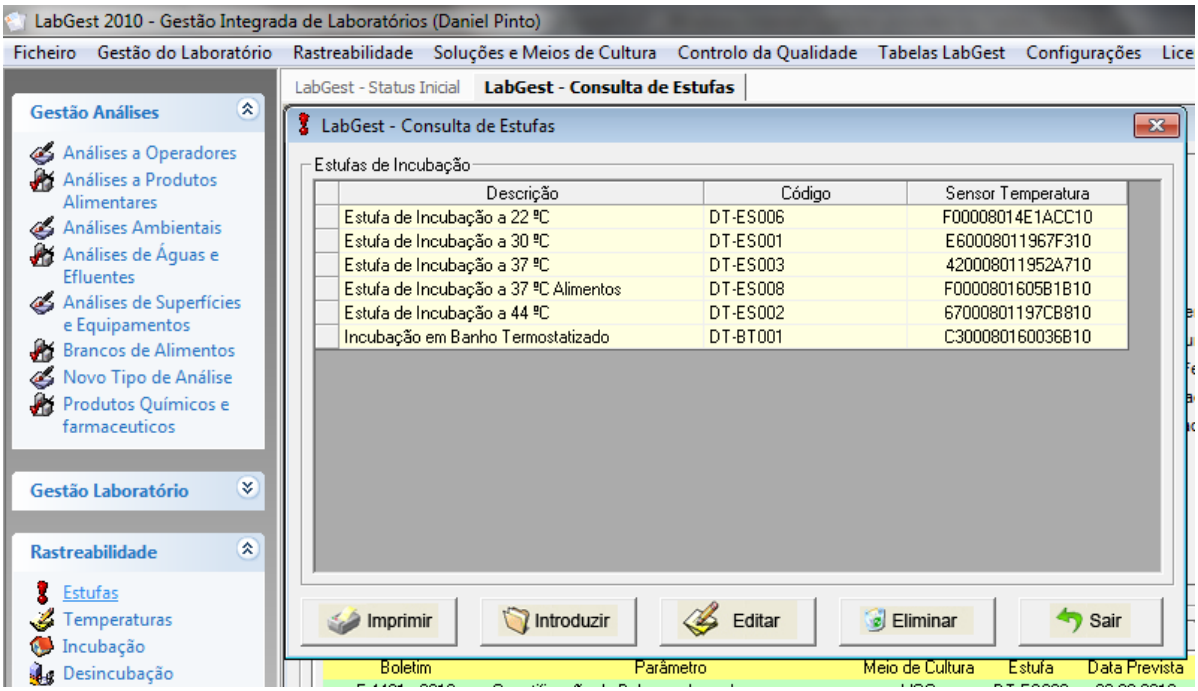


Figure 5.34: Incubators form with information regarding temperature sensors

When the users enter the Incubation section, it's visible the analyses that have Incubation activated as previously configured are shown. To incubate a parameter, simply select it, choose incubator, operator and culture medium and lot and press Incubate Parameter (Figure 5.35). After this, the user should remove that parameter from Incubation status in order to input the results. To do this, LabGest® has a specific section.

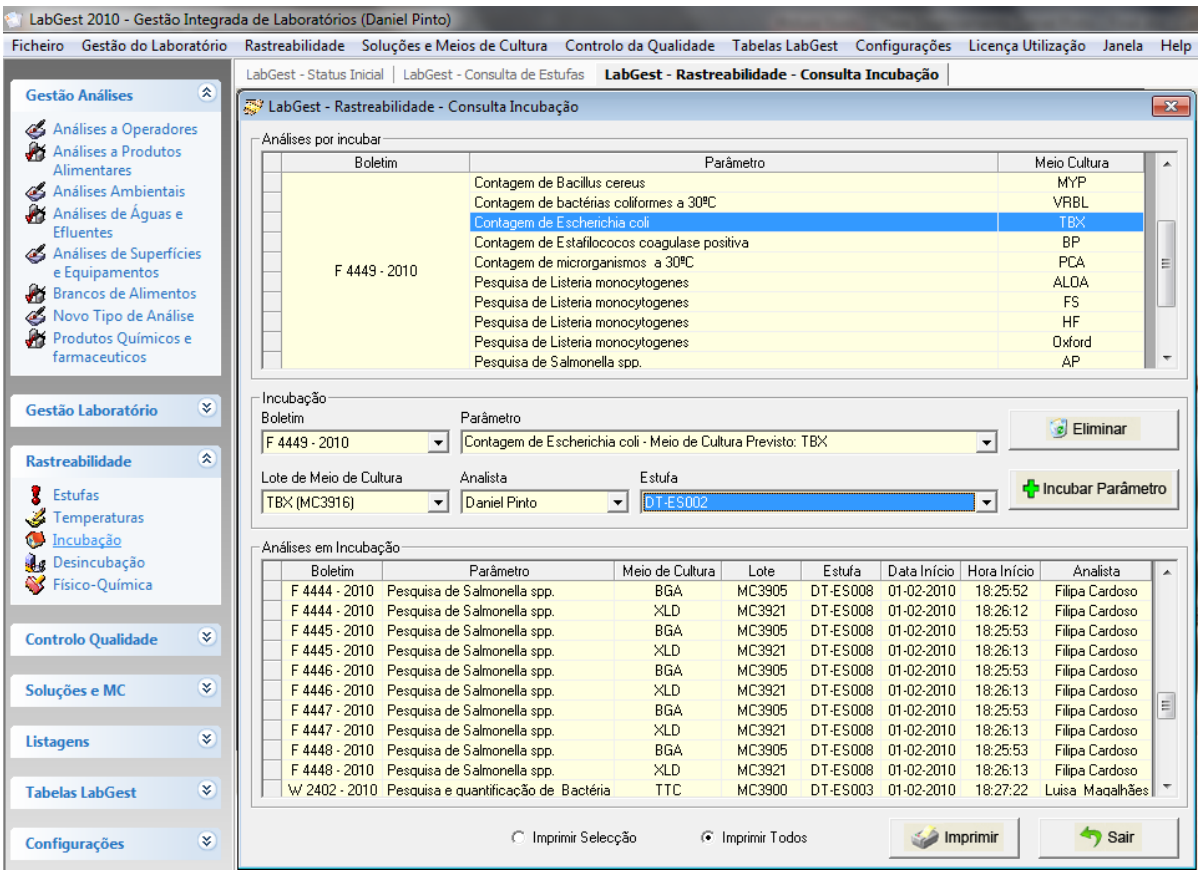


Figure 5.35: Incubation form

After this, at any time, on the analysis management section can be consulted all traceability of the laboratory, as follows:

1. Go to the analysis management section and click the desired analysis type;
2. Got to closed analysis tab and select the desired analysis
3. Click once at the analysis number. At the botton table appears the parameters results (Figure 5.36);
4. Double click the analysis number to show a most complete form of the parameters results (Figure 5.37);
5. Double click on a microbiological parameter to view the incubation information as time and temperature;

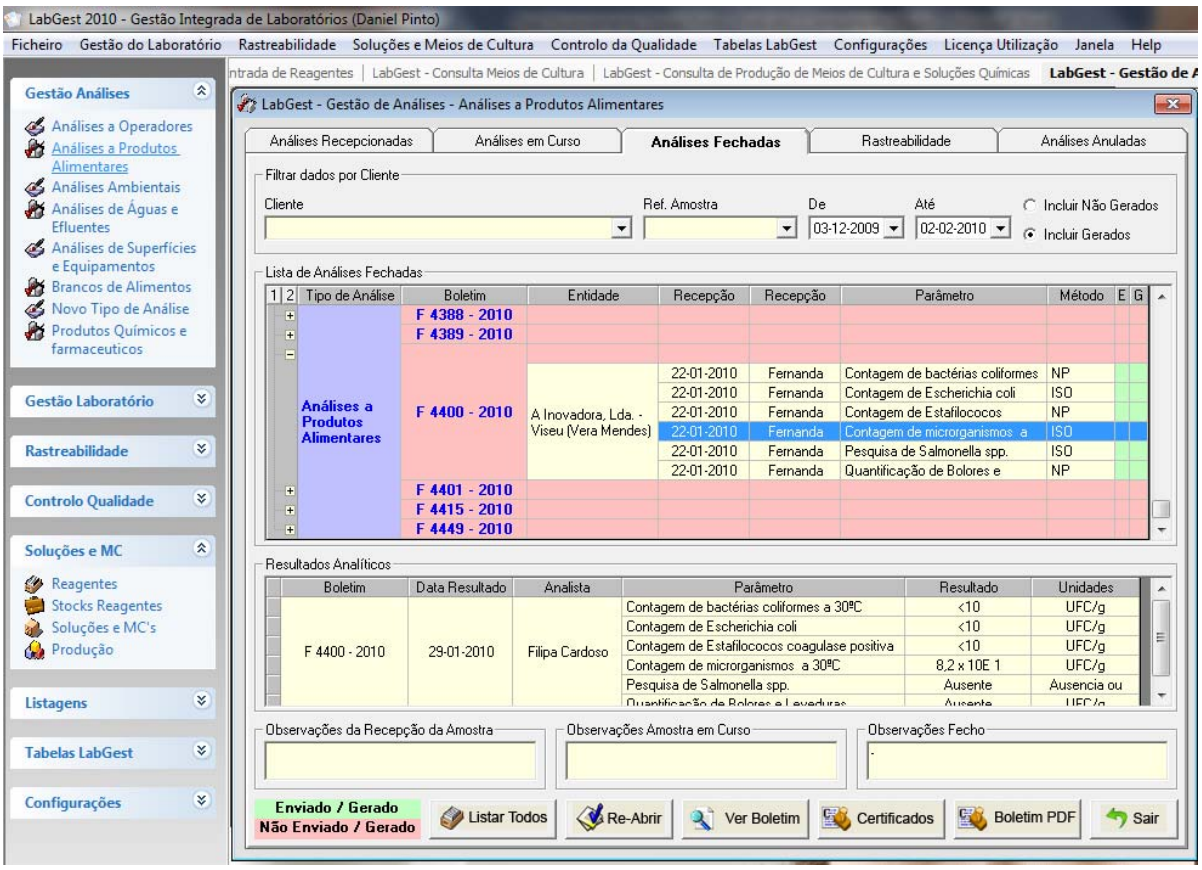


Figure 5.36: Traceability – view results

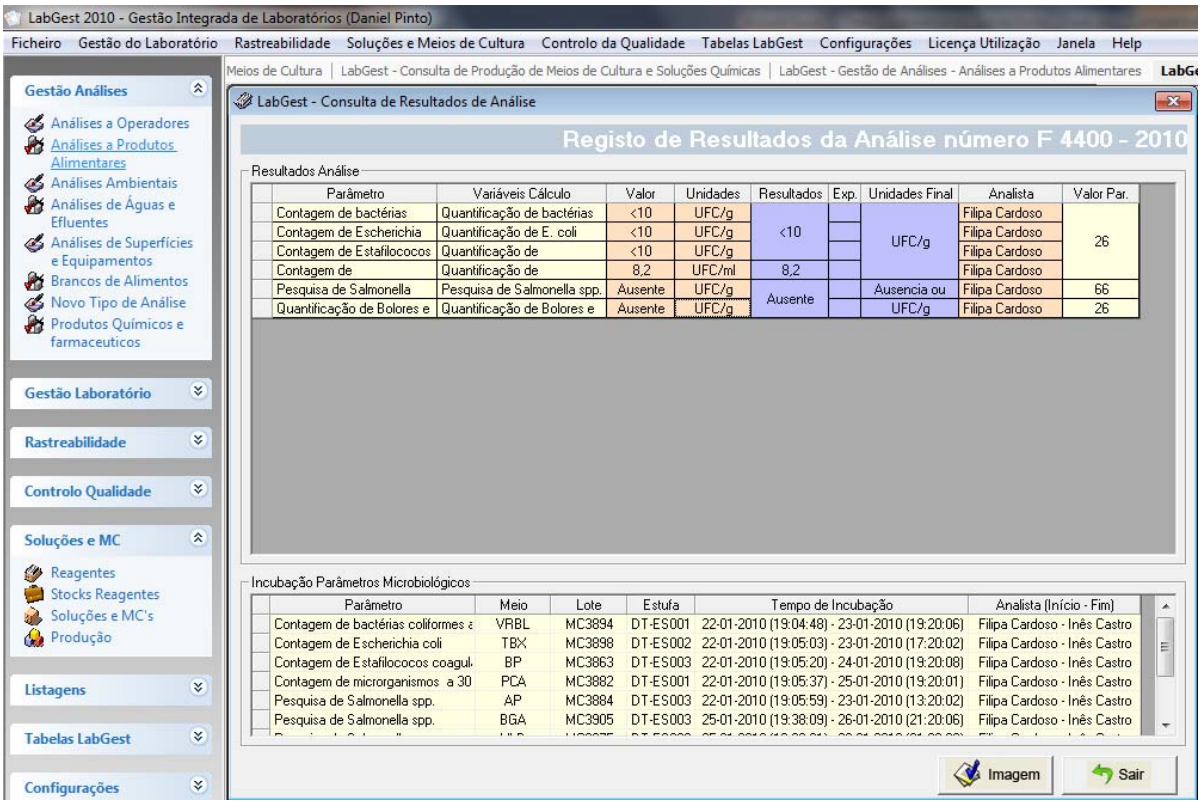


Figure 5.37: Traceability – detail results view

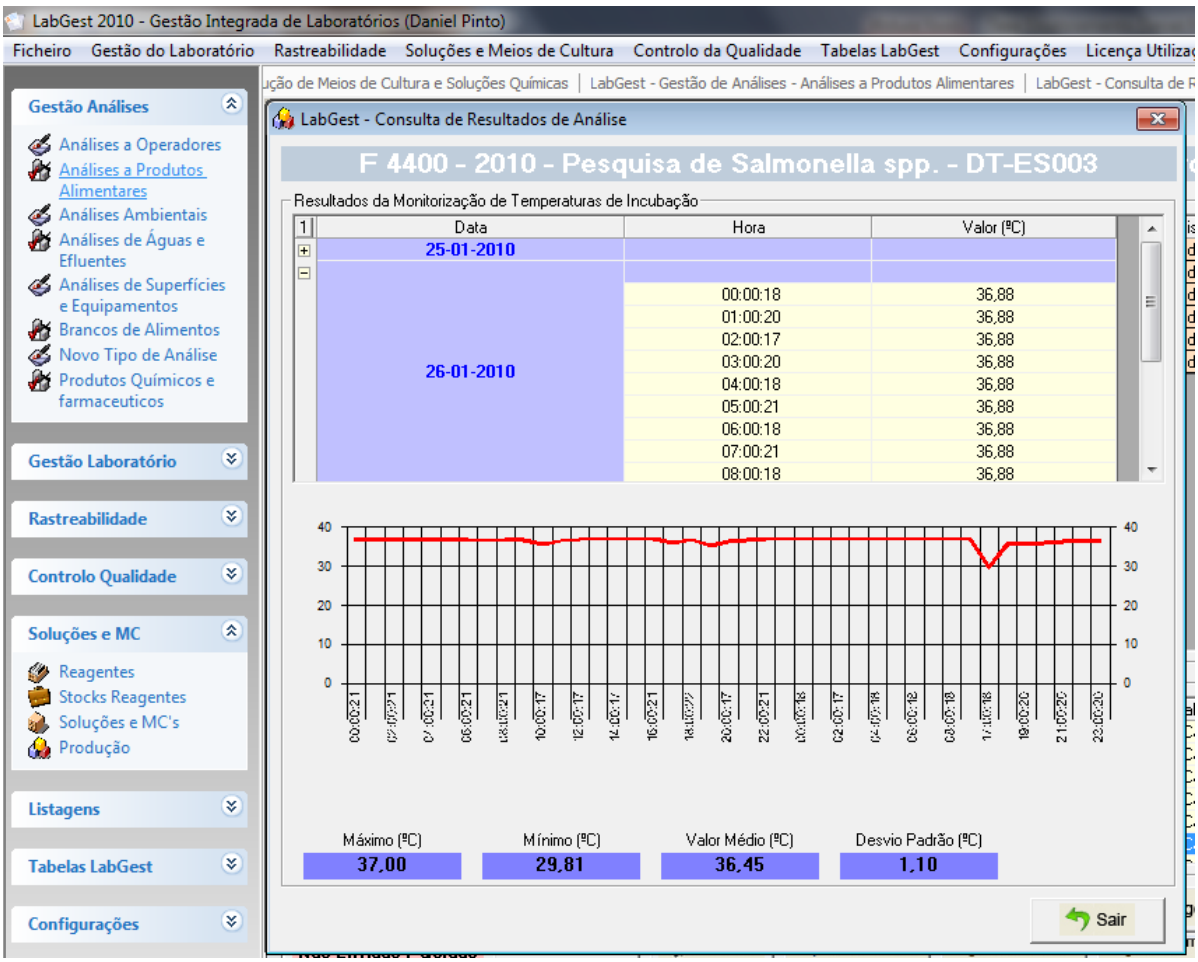


Figure 5.38: Traceability – incubation information

Another way to achieve this is by pressing the Traceability tab and select an analysis certificate number to view all traceability information (Figure 5.39).

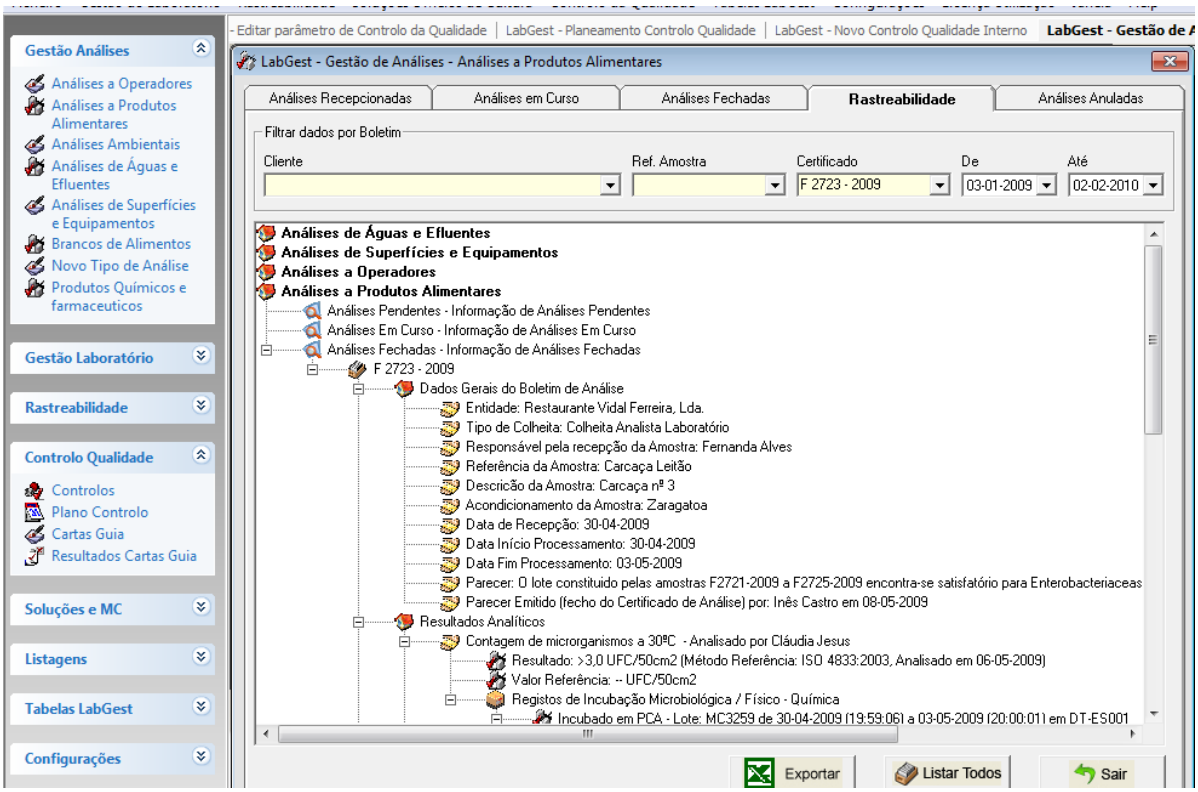


Figure 5.39: Traceability

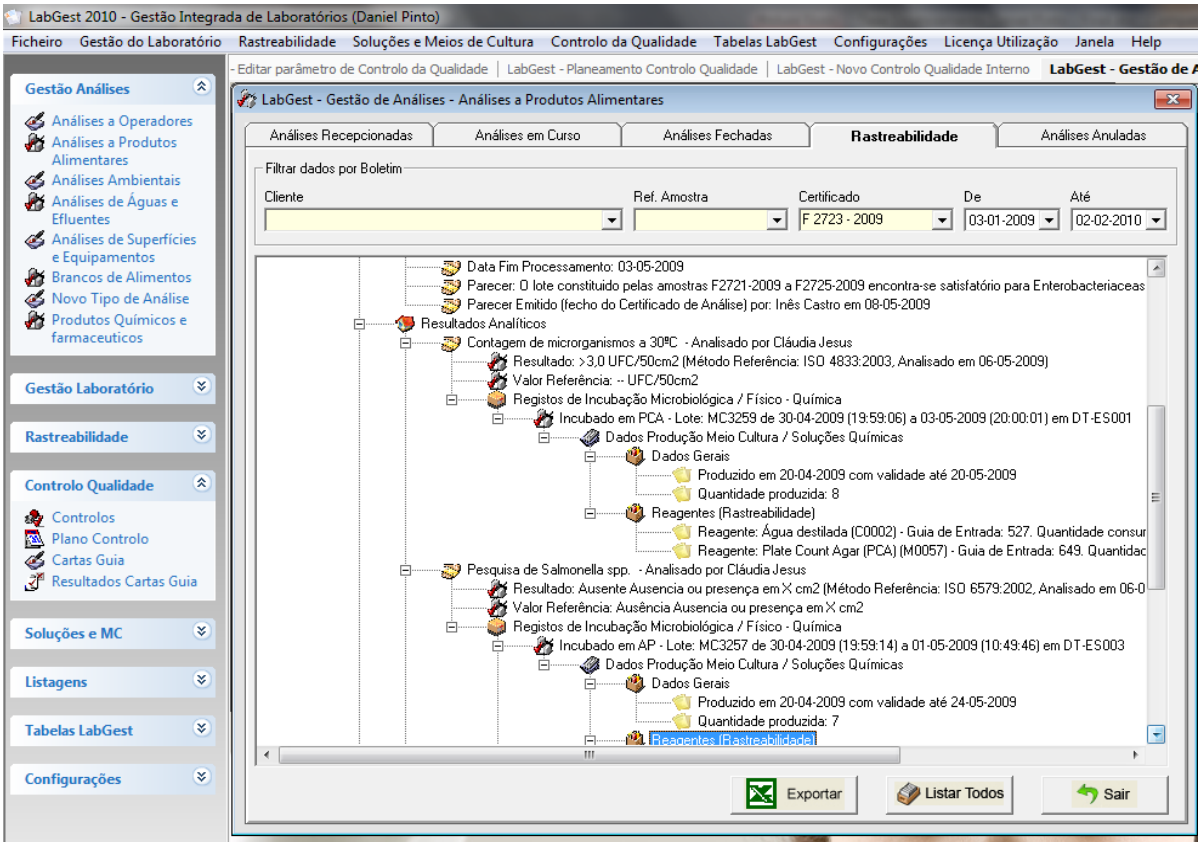


Figure 5.40: Traceability (continued)

3.1.8. Section "Quality Control"

The main goal of this section is to document quality control operations namely acceptation/rejection decisions, release procedures, among others (Figure 5.41).

The quality control is made accordingly to the specifications that were inserted and the Laboatory must define its control plan. To configure quality control parametes, simply go to controls section and insert a new control. Once the control is created there is the need to specify its limits/ expected result and periodicity (Figure 5.41). Internal quality control is one of the most important requirements of NP EN ISO/IEC 17025 and LabGest® is able to give response to this requirement reducing considerably paperwork and avoids forgting the controls.

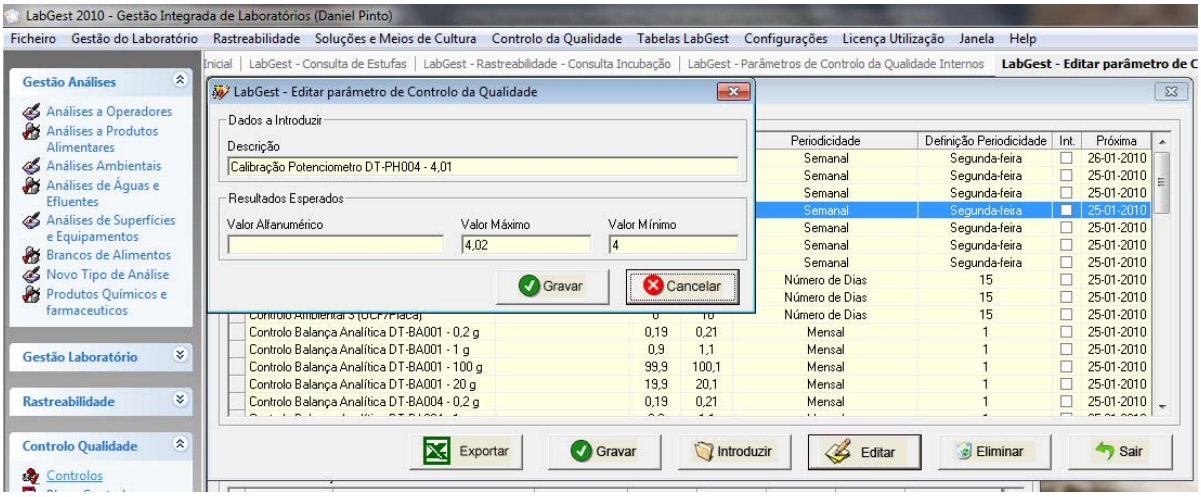


Figure 5.41: Internal quality controls form

When all the controls are configured, it is possible to consult the quality control plan. At this form, the laboratory user can query the quality control by three different ways:

- General daily table – controls to be executed at that day;
- Weekly planning – a plan for all week concerning internal quality controls;
- Monthly planning – a plan for all month concerning internal quality controls;

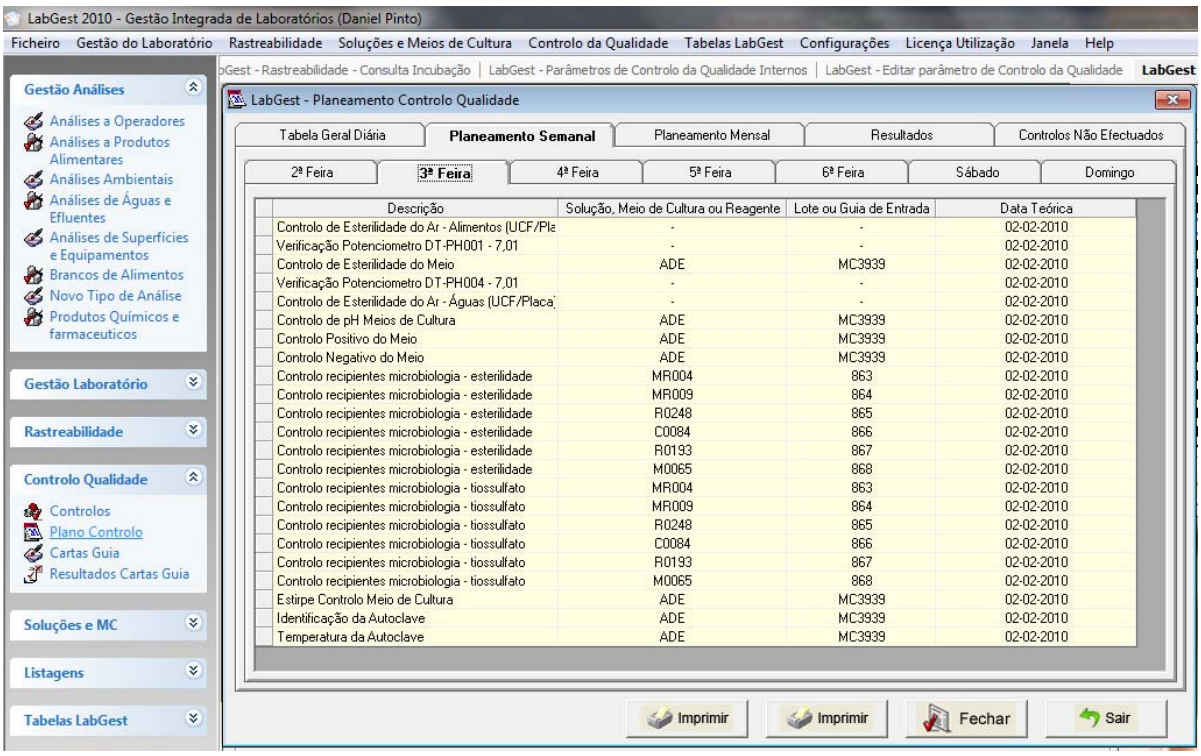


Figure 5.42: Internal quality controls plan

Nevertheless, LabGest® automatically sends and e-mails information regarding the controls to be performed.

To insert a control result, the users simply double clicks that control and enter the results (Figure 5.43).

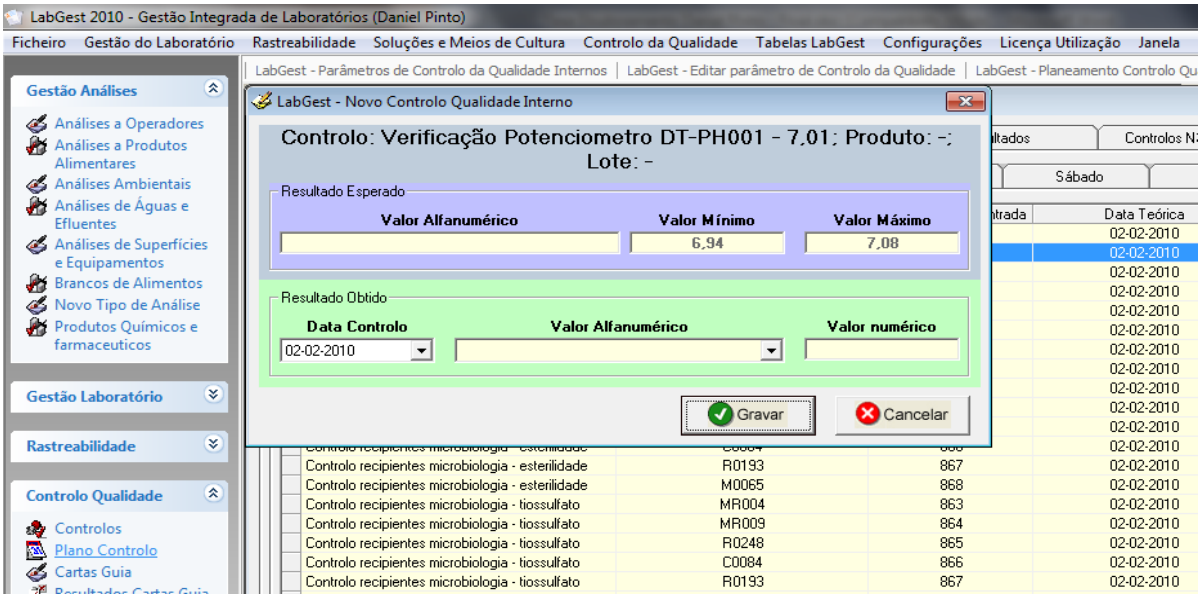


Figure 5.43: Insert internal quality control results

4. Conclusions

The developed computer package, LabGest®, is currently used by Portuguese laboratories and it is fully implemented in LabMaia. LabMaia and other clients using LabGest® are accreditated laboratories according to NP EN ISO/IEC 17025 and LabGest® has been audited the last few years and the auditor's feedback is also very good, especially because it reduces the amount of paperwork and allows to clearly identifying responsibilities and is extremely useful when implementing and being audit by NP EN ISO/IEC 17025. Also, the application allows implementing traceability in the laboratory without a large number of human resources.

LabGest® is very user friendly, especially because it runs under a common Microsoft Windows environment and uses common computer user controls. Another important advantage is the use of a web browser to easily query information by users who do not need the program installed on their personal computer. It is designed to meet the needs of the accredited NP EN ISO/IEC 17025 laboratories (see Table 5.3).

Table 5.3: Relationship between NP EN ISO/IEC 17025 requirements and LabGest®

NP EN ISO/IEC 17025 Requirements		LabGest®
4.5	Subcontracting of Tests and Calibrations	Subcontracted parameters management and identification
4.6	Purchasing Services and Supplies	The quality of incoming material is verified against predefined specifications with LabGest® reagent management and reception
4.13	Record Control	With user level security implemented, LabGest® ensure that original records are not overwritten by the system and that corrections are recorded together with the original records. It also uses a system that prevents overwriting original records and stores changes in an electronic audit trail that can be viewed and printed
5.2	Personnel	With user level security implemented and user permissions management, with LabGest®, only authorize personnel can perform specific tasks, for example, to issue test reports or to interpret specific test results.
5.6	Measurement Traceability	All actions made in LabGest® are traceable.

Table 5.4: Relationship between NP EN ISO/IEC 17025 requirements and LabGest® (continued)

NP EN ISO/IEC 17025 Requirements		LabGest®
5.8	Handling Test and Calibration Items	As requested by NP EN ISO/IEC 17025, test and calibration items should be uniquely identified. LabGest® allows the unique sample identification since reception to analysis certificate issue.
5.9	Assuring the Quality of Test and Calibration Results	According to NP EN ISO/IEC 17025, laboratory should ensure the quality of results on an ongoing basis, for example, through regular analysis of quality control samples. With LabGest®: • The validity of test results are monitored on an ongoing basis; • The type and frequency of tests are planned and documented through LabGest® Quality Control Management
5.10	Reporting of Results	Concerning results reporting, as requested by NP EN ISO/IEC 17025, LabGest® analysis certificates includes: • The name and address of the laboratory; • Unique identification of the test report or calibration certificate; • The name and address of the client; • Identification of the method; • A description and identification of the item(s) tested; • Reference to the sampling plan and procedures used by the laboratory; • The test results with the units of measurement; • The name(s), function(s) and signature(s) or equivalent identification of person(s) authorizing the analysis certificate; • When opinions and interpretations are included, LabGest® can document the basis upon which the opinions and interpretations have been made; • Opinions and interpretations are clearly marked as such in a analysis certificate.

5. References

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Chapter 6.

General Conclusions and Future Perspectives

1. General Conclusions and Future Perspectives

This thesis focused on four major themes: formation of acrylamide in French fries and baking products as a function of processing conditions; enrichment of baking products with dietary supplements such as vitamin E; development of the computer application Panigest® to manage food safety requisites in the baking industry; development of the computer application Labgest® to support NP EN ISO/IEC 17025 requirements in laboratories. The general conclusions of the work are presented below.

Acrylamide formation in French fries is highly related with the quality of oils used, namely in terms of polar compounds, and a direct relation has been found between them, i.e. the worse the oil quality in terms of polar compounds, the higher the amount of acrylamide in the product. Moreover, it was also found that during the frying cycle the formation of acrylamide increases rapidly in the beginning and stabilizes when the cycle reaches about two thirds of its duration. Temperature was confirmed to play an important role: higher temperatures are responsible for increased levels of acrylamide and, simultaneously, for accelerated degradation of oils

Different conclusions regarding acrylamide formation kinetics were drawn for baking products; in this case no significant changes were found when using different baking temperatures. It has also been concluded, while studying acrylamide behavior during shelf-life of traditional bakery products, that acrylamide concentration increases only during the heating process and it remains stable during storage.

Regarding α- and δ-tocopherols, different contents of these compounds were found in different traditional bakery products and in different parts of those products: higher α-tocopherol concentrations were registered in corn bread, in the crust of corn bread and in the crust of “bijou” bread than in corn bread dough and “bijou” bread dough.

Future perspectives point to the need for a deeper knowledge concerning mathematical modeling of the acrylamide formation into several food models taking into account processing conditions such as temperature and type of heating and the production steps as key aspects of the model. Furthermore, considering increasing demands for safer and healthier foods, it will be of extreme interest to optimize/change products formulations in order to minimize the concentrations of harmful compounds present in the final product.

Future work should also consider monitoring levels of precursors of acrylamide in bakery products and pastries during their processing, in order to correlate acrylamide formation with changes in those levels. On the other hand, it should be studied the possibility of increasing the amount of α- and δ-tocopherols in traditional bakery products by adding vitamin E directly to the product at an early stage of production and following its fate throughout the production line.

Concerning the software developing work, two important software packages were developed: PaniGest® and LabGest®. Both have the main objectives of helping industry and laboratories to comply with regulatory or normative requirements. PaniGest® allows implementing traceability in the food (baking) industry without a large number of technical and human resources and it facilitates recall procedures with economical and safety benefits.

LabGest® reduces the amount of paperwork and allows to clearly identify responsibilities and is extremely useful both when implementing NP EN ISO/IEC 17025 and when being audited. Also, this application allows implementing traceability in the laboratory without a large number of human resources.

PaniGest® future developments should include a software package allowing to receive data from HACCP systems (e.g. level of risks) and reporting all the preventive controls associated with those systems.

LabGest® is continuously improving once it is fully implemented in LabMaia® and almost daily several new functions are requested. In a few months it will be prepared for automatically construct control charts and duplicate charts, being also able to validate results.

APPENDIX

1. Calibration Curves

1.1. Acrylamide calibration curve

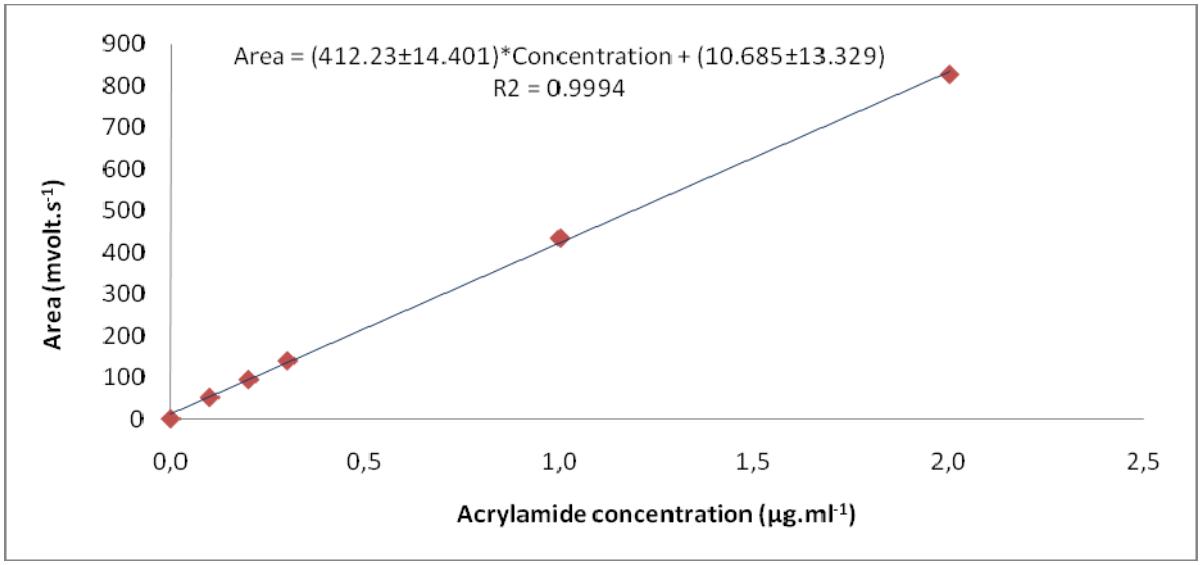

Figure A.0.1: Acrylamide calibration curve for concentration values between 0.0 µg.ml^{-1} and 2.0 µg.ml^{-1}

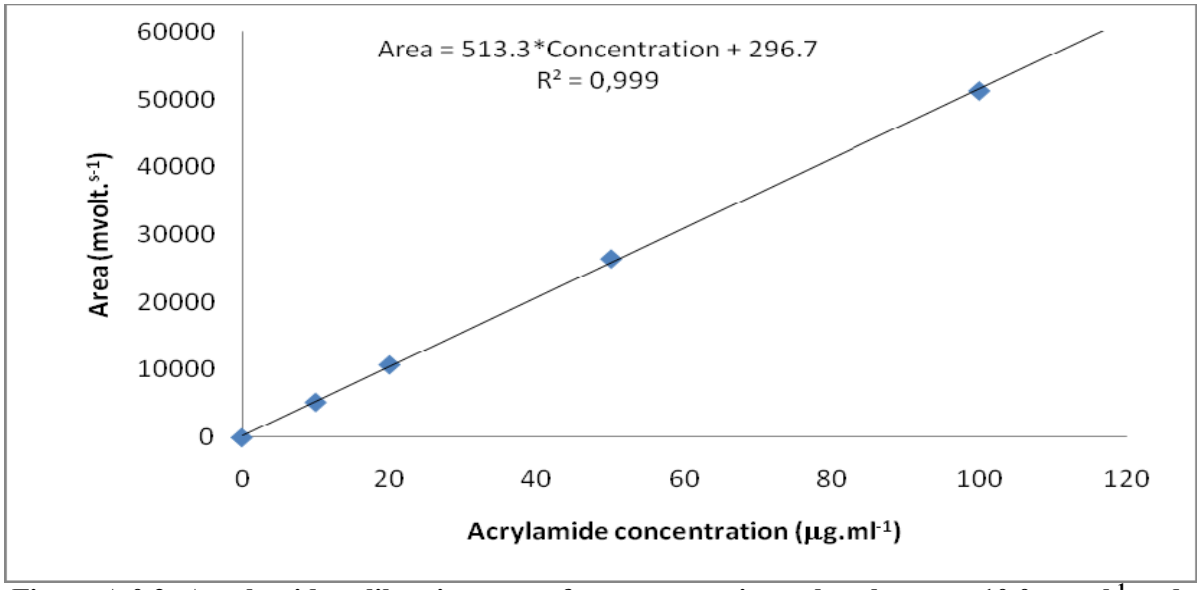

Figure A.0.2: Acrylamide calibration curve for concentration values between 10.0 µg.ml^{-1} and 100.0 µg.ml^{-1}

1.2. α and δ Tocopherol calibration curve

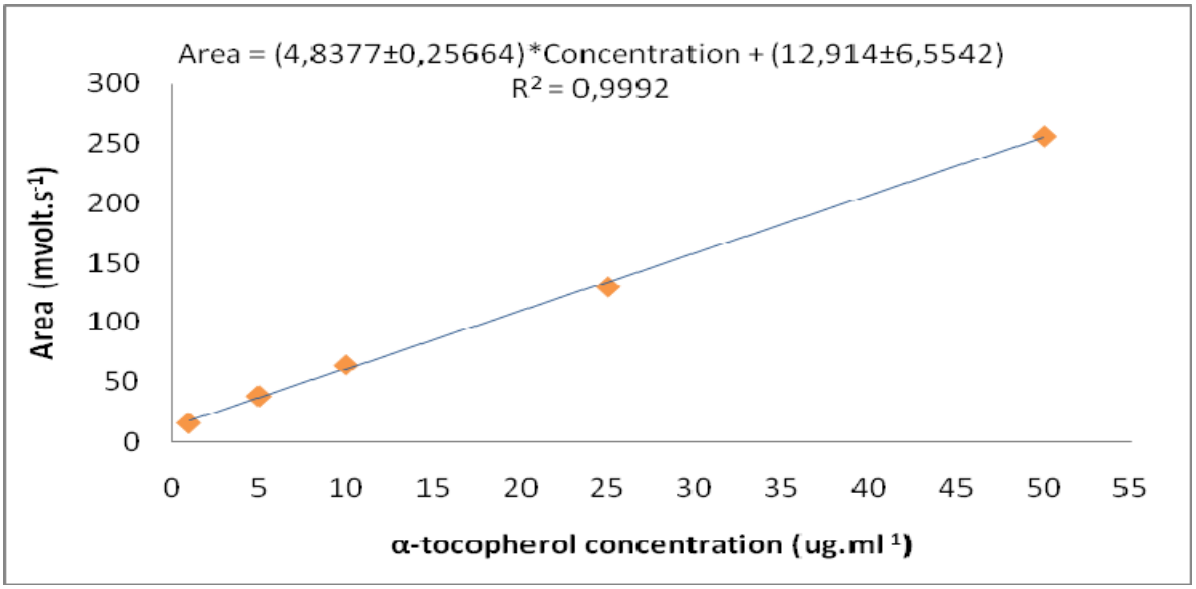

Figure A.0.3: α-tocopherol calibration curve

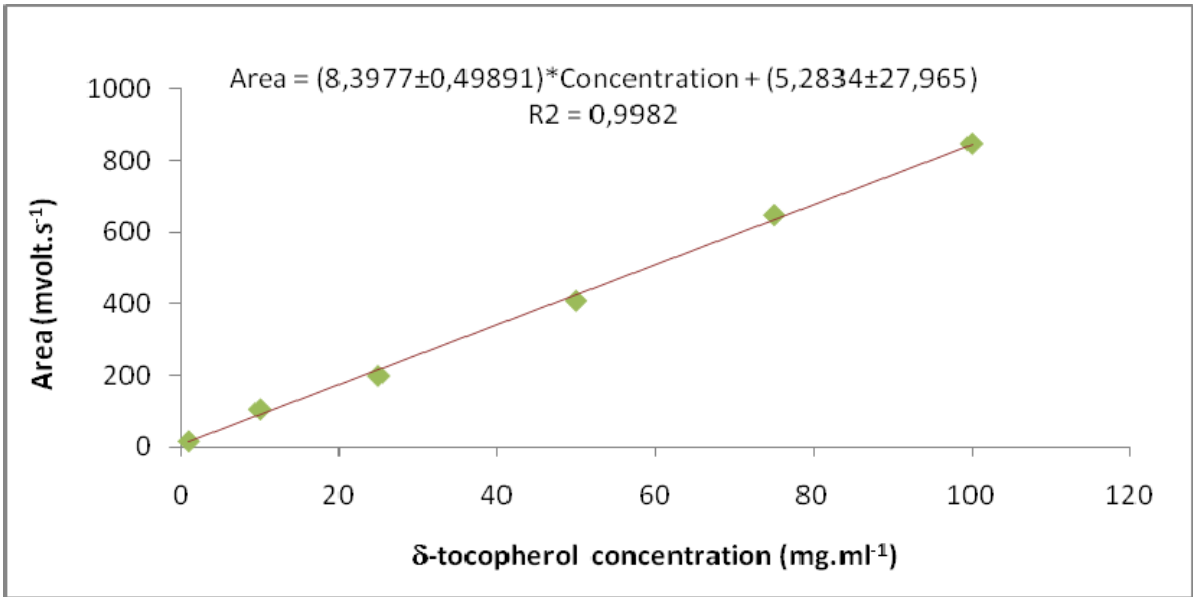

Figure A.0.4: δ-tocopherol calibration curve

2. PaniGest® code sample

Here is a sample of the code computed at the raw material reception form.

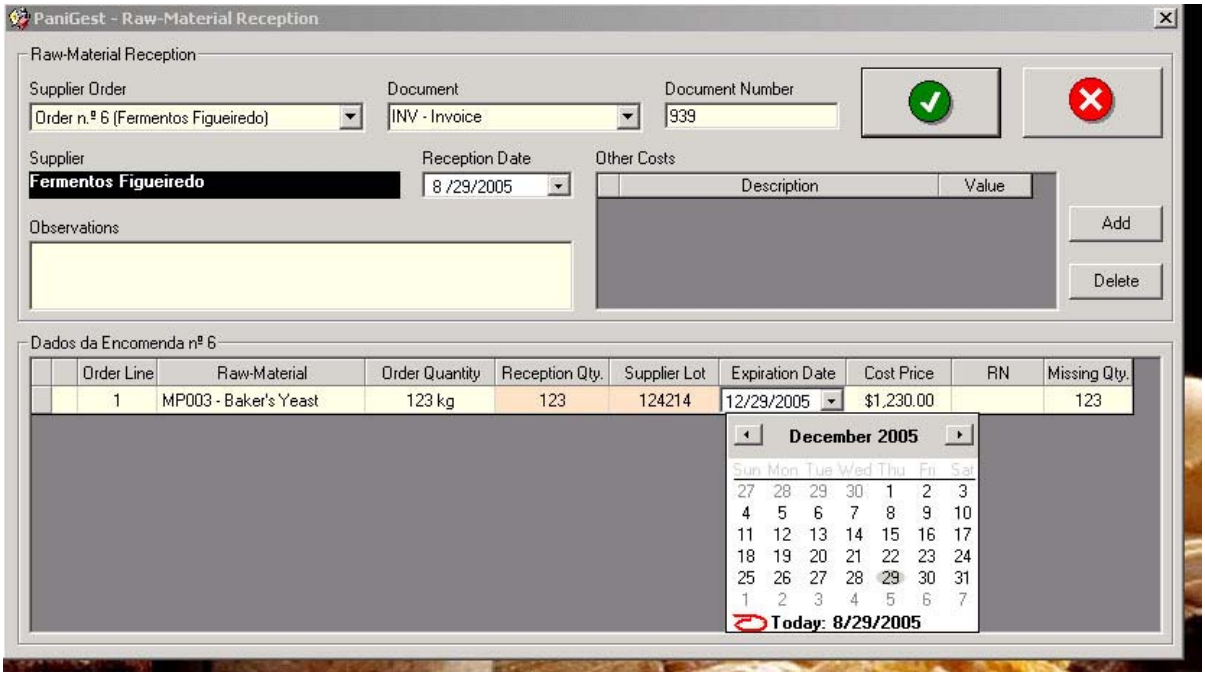

Figure A.0.5: Example of a raw-material reception

```
Dim getultima As ADODB.Recordset
Dim GR As Integer
Dim ne As ADODB.Recordset
Dim idne As Integer
Dim linhasne As ADODB.Recordset
Dim mp As ADODB.Recordset
Dim forne As ADODB.Recordset
Dim Doc As ADODB.Recordset
Dim Conf As Integer
Dim gravaGR As ADODB.Recordset
Dim stocks As ADODB.Recordset
Dim precos As ADODB.Recordset
Dim Validade As Date
Dim LoteF As String
Dim qde As Currency
Dim CodigoMP As String
```

```
Dim unidade As String
Dim Preco As Currency
Dim stockGR As Currency
Dim linNE As Integer
Dim iddoc As String
Dim stockreal As Currency
Dim pcms As ADODB.Recordset
Dim pcm As Currency
Dim pcmfinal As Currency
Dim encomendas As ADODB.Recordset
Dim encomendas2 As ADODB.Recordset
Dim estado As Integer
Dim Estadoencomenda As Integer
Dim encomendas3 As ADODB.Recordset
Dim cq As ADODB.Recordset
Dim imprimido As Integer
Dim estadoCQ As Integer
Dim ultimaana As ADODB.Recordset
Dim numanalise As Integer
Dim idforne As Integer
Dim getpreco As ADODB.Recordset
Dim tempgr As ADODB.Recordset
Dim vertemp As ADODB.Recordset
Dim enca As ADODB.Recordset
Dim getnumdoc As ADODB.Recordset
Dim ValidadeActiva As Integer
Dim verqualidade As ADODB.Recordset
Dim conforme As Integer
Dim gravaanalises As ADODB.Recordset
Dim getespe As ADODB.Recordset
Dim gravapara As ADODB.Recordset
Dim movimentos As ADODB.Recordset
Dim movimentos2 As ADODB.Recordset
```

```
Private Sub Combo1_Click()
On Error GoTo Erro
    Dim pretendido As Integer
    pretendido = Combo1.ListIndex + 1
    Dim inicial As Variant
    ne.MoveFirst
    inicial = ne.Bookmark
    ne.Move pretendido - 1, inicial
    idne = ne.Fields("numencomenda").Value
    Frame2.Caption = "Dados da Encomenda nº " & idne
    Set forne = New ADODB.Recordset
    forne.Open "select * from SMP_Tabela_Fornecedores where codfornecedor = " &
ne.Fields("codfornecedor").Value & "", cpcOT, adOpenStatic
    Text3.Text = forne.Fields("NomeEmpresa").Value
    idforne = forne.Fields("CodFornecedor").Value
    forne.Close
    Set linhasne = New ADODB.Recordset
    linhasne.Open "select * from SMP_Linhas_NotaEncomenda where ne = " & idne & "
and estadolinha <> 1 and estadolinha <> 4", cpcOT, adOpenStatic
    Tabela.Clear
    Tabela.TextMatrix(0, 2) = "Linha NE"
    Tabela.TextMatrix(0, 3) = "Matéria Prima"
    Tabela.TextMatrix(0, 4) = "Qde Encomenda"
    Tabela.TextMatrix(0, 5) = "Qde Recepção"
    Tabela.TextMatrix(0, 6) = "Lote Forn."
    Tabela.TextMatrix(0, 7) = "Validade"
    Tabela.TextMatrix(0, 8) = "PC (s/IVA)"
    Tabela.TextMatrix(0, 9) = "GR a atribuir"
    Tabela.TextMatrix(0, 10) = "Qde falta"
    Tabela.ColDataType(1) = flexDTBoolean
    Tabela.ColDataType(7) = flexDTDate
    linha = 1
    linhasne.MoveFirst
```

```
        Tabela.Rows = linhasne.RecordCount + 1
        Set mp = New ADODB.Recordset
        Do While Not linhasne.EOF
           Tabela.TextMatrix(linha, 2) = linhasne.Fields("IDLInha").Value
           Tabela.TextMatrix(linha, 11) = linhasne.Fields("CodigoMP").Value
           mp.Open "select * from SMP_Tabela_Mprimas where codigoMP = '" &
linhasne.Fields("CodigoMP").Value & "'", cpcOT, adOpenStatic
           If mp.Fields("ControloValidade").Value = False Then
              ValidadeActiva = 0
              Tabela.TextMatrix(linha, 7) = Date
           Else
              ValidadeActiva = 1
           End If
           Tabela.TextMatrix(linha, 14) = ValidadeActiva
           Tabela.TextMatrix(linha, 3) = linhasne.Fields("CodigoMP").Value & " - " &
mp.Fields("Descricao").Value
           mp.Close
           Tabela.TextMatrix(linha, 4) = linhasne.Fields("Quantidade").Value & " " &
linhasne.Fields("unidades").Value
           Tabela.TextMatrix(linha, 8) = linhasne.Fields("precofinal").Value
           Tabela.TextMatrix(linha, 10) = linhasne.Fields("qdefalta").Value
           Tabela.TextMatrix(linha, 12) = linhasne.Fields("unidades").Value
           Tabela.TextMatrix(linha, 13) = linhasne.Fields("Quantidade").Value
           linhasne.MoveNext
           linha = linha + 1
        Loop
        For i = 1 To Tabela.Rows - 1
           Tabela.Row = i
           Tabela.Col = 5
           Tabela.CellBackColor = &HC0E0FF
           Tabela.Col = 6
           Tabela.CellBackColor = &HC0E0FF
           Tabela.Col = 7
           Tabela.CellBackColor = &HC0E0FF
```

```
        Next i
        Tabela.SelectionMode = flexSelectionFree
    Erro:
        If Err = 0 Then
            Err.Clear
        Else
            MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number), vbCritical, App.Title
            Err.Clear
        End If
    End Sub

    Private Sub Combo2_Click()
    On Error GoTo Erro
        Dim pretendido1 As Integer
        pretendido1 = Combo2.ListIndex + 1
        Dim inicial1 As Variant
        Doc.MoveFirst
        inicial1 = Doc.Bookmark
        Doc.Move pretendido1 - 1, inicial1
        iddoc = Doc.Fields("idtipodocumento").Value
        If Doc.Fields("numeracaoautomatica").Value = True Then
            Set getnumdoc = New ADODB.Recordset
            getnumdoc.Open "select * from SMP_Tabela_NumerosDocumentos where
idtipodocumento = '" & iddoc & "'", cpcOT, adOpenStatic
            Text2.Text = getnumdoc.Fields("ultimonumero").Value + 1
            Text2.Enabled = False
        Else
            Text2.Text = ""
            Text2.Enabled = True
        End If
    Erro:
        If Err = 0 Then
            Err.Clear
```

```
        Else
            MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number), vbCritical, App.Title
            Err.Clear
        End If
    End Sub

    Private Sub Command1_Click()
    On Error GoTo Erro
        Conf = 0
        For i = 1 To Tabela.Rows - 1
            If Tabela.ValueMatrix(i, 1) <> 0 Then
                Conf = Conf + 1
            End If
        Next i
        If Conf < 1 Then
            MsgBox "Seleccione uma Linha de Nota de Encomenda a Recepcionar!",
vbInformation, App.Title
            Exit Sub
        End If
        If Combo1.ListIndex = -1 Then
            MsgBox "Seleccione Nota de Encomenda!", vbInformation, App.Title
            Exit Sub
        End If
        If Combo2.ListIndex = -1 Then
            MsgBox "Seleccione Tipo de Documento que acompanha a encomenda!",
vbInformation, App.Title
            Exit Sub
        End If
        If Text4.Text = "" Then
            Text4.Text = "-"
        End If
        If Text2.Text = "" Then
```

```
        MsgBox "Introduza número do documento que acompanha a mercadoria!", vbInformation, App.Title
        Exit Sub
      End If

      If Encargos.Rows > 1 Then
        For l = 1 To Encargos.Rows - 1
          If Encargos.TextMatrix(l, 1) = "" Then
            MsgBox "Introduza descrição do encargo ou elimine linhas desnecessárias!", vbInformation, App.Title
            Exit Sub
          End If
          If Encargos.TextMatrix(l, 2) = "" Then
            MsgBox "Introduza custo do encargo " & Encargos.TextMatrix(l, 1) & "!", vbInformation, App.Title
            Exit Sub
          End If
        Next l
      End If

      For k = 1 To Tabela.Rows - 1
        If Tabela.ValueMatrix(k, 1) = -1 Then
          If Tabela.TextMatrix(k, 5) = "" Then
            MsgBox "Introduza quantidade a recepcionar de " & Tabela.TextMatrix(k, 3) & "!", vbExclamation, App.Title
            Exit Sub
          Else
            qde = Tabela.TextMatrix(k, 5)
            stockGR = qde
          End If
          If Tabela.TextMatrix(k, 6) = "" Then
            MsgBox "Introduza Lote do Fornecedor de " & Tabela.TextMatrix(k, 3) & "!", vbExclamation, App.Title
            Exit Sub
```

```
            Else
               LoteF = Tabela.TextMatrix(k, 6)
            End If
            If Tabela.TextMatrix(k, 7) = "" Then
               MsgBox "Introduza Data de Validade da Matéria Prima " &
Tabela.TextMatrix(k, 3) & "!", vbExclamation, App.Title
               Exit Sub
            Else
               Validade = Tabela.TextMatrix(k, 7)
            End If
            CodigoMP = Tabela.TextMatrix(k, 11)
            unidade = Tabela.TextMatrix(k, 12)
            Preco = Tabela.TextMatrix(k, 8)
            linNE = Tabela.TextMatrix(k, 2)
            If qde < Tabela.TextMatrix(k, 10) Then
               estado = 3
            ElseIf qde >= Tabela.TextMatrix(k, 10) Then
               estado = 1
            End If
         End If
      Next k

      For k = 1 To Tabela.Rows - 1
         If Tabela.ValueMatrix(k, 1) = -1 Then
            If Tabela.TextMatrix(k, 5) = "" Then
               MsgBox "Introduza quantidade a recepcionar de " & Tabela.TextMatrix(k, 3)
& "!", vbExclamation, App.Title
               Exit Sub
            Else
               qde = Tabela.TextMatrix(k, 5)
               stockGR = qde
            End If
            If Tabela.TextMatrix(k, 6) = "" Then
```

```
            MsgBox "Introduza Lote do Fornecedor de " & Tabela.TextMatrix(k, 3) &
"!", vbExclamation, App.Title
            Exit Sub
          Else
            LoteF = Tabela.TextMatrix(k, 6)
          End If
          If Tabela.TextMatrix(k, 7) = "" Then
            MsgBox "Introduza Data de Validade da Matéria Prima " &
Tabela.TextMatrix(k, 3) & "!", vbExclamation, App.Title
            Exit Sub
          Else
            Validade = Tabela.TextMatrix(k, 7)
          End If
          CodigoMP = Tabela.TextMatrix(k, 11)
          unidade = Tabela.TextMatrix(k, 12)
          Preco = Tabela.TextMatrix(k, 8)
          linNE = Tabela.TextMatrix(k, 2)
          qdefalta = Tabela.TextMatrix(k, 13) - qde
          If qdefalta < 0 Then
            qdefalta = 0
          End If
          If qde < Tabela.TextMatrix(k, 10) Then
            estado = 3
          ElseIf qde >= Tabela.TextMatrix(k, 10) Then
            estado = 1
          End If
          If qdefalta = 0 Then
            estado = 1
          End If
          estadoCQ = 2
          Set gravaGR = New ADODB.Recordset
          gravaGR.Open "insert into SMP_Tabela_GuiasRecepcao (GR, CodigoMP,
Fornecedor, LoteFornecedor, " & _
          " DataEntrada, QdeEntrada, unidade, validade, PrecoCompra, " & _
```

```
" StockGR, LinNE, obs, Tipodoc, NumeroDoc, NEncomenda, EstadoCQ,DataSistemaEntrada, HoraSistemaEntrada, UserWindows, usercpc, Computador) " & _
" values ('" & Tabela.TextMatrix(k, 9) & "','" & CodigoMP & "','" & idforne & "','" & LoteF & "', " & _
" '" & Data.Value & "','" & qde & "','" & unidade & "','" & Validade & "', " & _
" '" & Preco & "','" & stockGR & "','" & linNE & "','" & Text4.Text & "', " & _
" '" & iddoc & "','" & Text2.Text & "','" & idne & "','" & estadoCQ & "', '" & Date & "','" & Time & "','" & Utilizador & "','" & userCQ & "','" & Computador & "')", cpcOT, adOpenStatic

'update Estados Linhas Notas Encomendas
Set encomendas = New ADODB.Recordset
encomendas.Open "update SMP_Linhas_NotaEncomenda set estadolinha = '" & estado & "', qdefalta = '" & qdefalta & "', L_DataSistemaEntrada = '" & Date & "', L_HoraSistemaEntrada = '" & Time & "' ,L_UserWindows = '" & Utilizador & "', L_UserCPC = '" & userCQ & "', L_Computador = '" & Computador & "' where ne = " & idne & " and idlinha = " & linNE & "", cpcOT, adOpenStatic

'Update COntrolo Qualidade
Set ultimaana = New ADODB.Recordset
ultimaana.Open "select * from CQ_Analises order by idanalise", cpcOT, adOpenStatic
If ultimaana.RecordCount = 0 Then
   numanalise = 1
Else
   ultimaana.MoveLast
   numanalise = ultimaana.Fields("IDanalise").Value + 1
End If
Set cq = New ADODB.Recordset
cq.Open "insert into CQ_Analises (IDanalise, Tipo, Documento,DataSistemaEntrada, HoraSistemaEntrada, UserWindows, usercpc, Computador) values ('" & numanalise & "','GR','" & Tabela.TextMatrix(k, 9) & "', '" & Date
```

& "'," & Time & "'," & Utilizador & "'," & userCQ & "'," & Computador & "')", cpcOT, adOpenStatic

'update precos

Set getpreco = New ADODB.Recordset

getpreco.Open "select * from SMP_Tabela_GuiasRecepcao where gr = " & Tabela.TextMatrix(k, 9) & "", cpcOT, adOpenStatic

Set precos = New ADODB.Recordset

precos.Open "update SMP_Tabela_Mprimas set PCU = '" & getpreco.Fields("PrecoCompra").Value / getpreco.Fields("Qdeentrada").Value & "', L_DataSistemaEntrada = '" & Date & "', L_HoraSistemaEntrada = '" & Time & "' ,L_UserWindows = '" & Utilizador & "', L_UserCPC = '" & userCQ & "', L_Computador = '" & Computador & "' where codigoMP = '" & CodigoMP & "'", cpcOT, adOpenStatic

Set pcms = New ADODB.Recordset

pcms.Open "select * from SMP_Tabela_GuiasRecepcao where codigoMP = '" & getpreco.Fields("CodigoMP").Value & "'", cpcOT, adOpenStatic

pcms.MoveFirst

pcm = 0

Do While Not pcms.EOF

pcm = pcm + (pcms.Fields("Precocompra").Value / pcms.Fields("qdeentrada").Value)

pcms.MoveNext

Loop

pcmfinal = pcm / pcms.RecordCount

pcms.Close

Set pcms = New ADODB.Recordset

pcms.Open "Update SMP_Tabela_Mprimas set pcm = '" & pcmfinal & "', L_DataSistemaEntrada = '" & Date & "', L_HoraSistemaEntrada = '" & Time & "' ,L_UserWindows = '" & Utilizador & "', L_UserCPC = '" & userCQ & "', L_Computador = '" & Computador & "' where codigomp = '" & getpreco.Fields("codigomp").Value & "'", cpcOT, adOpenStatic

If MsgBox("Matéria Prima Recepcionada. Deseja Imprimir Guia de Recepção " & Tabela.TextMatrix(k, 9) & "?", vbYesNo + vbQuestion, App.Title) = vbYes Then

```
                Cancel = False
                Source = App.Path & "\panigest.mdb"
                crt.ReportFileName = App.Path & "\Reports\GuiaRecepcao.rpt"
                crt.WindowState = crptMaximized
                crt.DiscardSavedData = True
                crt.WindowTitle = "Guia de Recepção"
                crt.SQLQuery = "SELECT " & _
                "                                                Query_Guias_de_Recepcao.`GR`,
Query_Guias_de_Recepcao.`CodigoMP`,          Query_Guias_de_Recepcao.`Descricao`,
Query_Guias_de_Recepcao.`LoteFornecedor`,  Query_Guias_de_Recepcao.`DataEntrada`,
Query_Guias_de_Recepcao.`QdeEntrada`,          Query_Guias_de_Recepcao.`Unidade`,
Query_Guias_de_Recepcao.`Validade`,          Query_Guias_de_Recepcao.`NumeroDoc`,
Query_Guias_de_Recepcao.`doc`, Query_Guias_de_Recepcao.`Obs` " & _
                " From `Query_Guias de Recepcao` Query_Guias_de_Recepcao " & _
                " where Query_Guias_de_Recepcao.`GR` = " & GR & ""
                crt.Action = 1
                Screen.MousePointer = 99
                Screen.MousePointer = 99
              Else
                Cancel = True
              End If
            End If
          Next k

          'Update Estados Notas Encomendas
          Set encomendas = New ADODB.Recordset
          Set encomendas2 = New ADODB.Recordset
          Set encomendas3 = New ADODB.Recordset
          encomendas.Open "select * from SMP_Linhas_NotaEncomenda where EstadoLinha
= 2 and ne = " & idne & "", cpcOT, adOpenStatic
          If encomendas.RecordCount = 0 Then
            encomendas2.Open "select * from SMP_Linhas_NotaEncomenda where
EstadoLinha = 3 and ne =" & idne & "", cpcOT, adOpenStatic
            If encomendas2.RecordCount = 0 Then
```

```
            Estadoencomenda = 1
          Else
            Estadoencomenda = 3
          End If
        Else
          Estadoencomenda = 2
        End If
        encomendas3.Open "update SMP_NE_Mprimas set estado = '" & Estadoencomenda
& "', L_DataSistemaEntrada = '" & Date & "', L_HoraSistemaEntrada = '" & Time & "'
,L_UserWindows = '" & Utilizador & "', L_UserCPC = '" & userCQ & "', L_Computador =
'" & Computador & "' where numencomenda = " & idne & "", cpcOT, adOpenStatic

        'grava encargos nota de encomenda
        If Encargos.Rows > 1 Then
          Set enca = New ADODB.Recordset
          For m = 1 To Encargos.Rows - 1
            enca.Open "insert into SMP_Encargos_NE (descricao, IDNE, valor) values ('"
& Encargos.TextMatrix(m, 1) & "'," & idne & ",'" & Encargos.TextMatrix(m, 2) & "')",
cpcOT, adOpenStatic
          Next m
        End If
      Erro:
        If Err = 0 Then
          Err.Clear
          Qualidade
          Form_Load
        Else
          MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number) & " - Transacção não efectuada!", vbCritical, App.Title
          Err.Clear
          Form_Load
          Exit Sub
        End If
      End Sub
```

```
Private Sub Qualidade()
On Error GoTo Erro
   For k = 1 To Tabela.Rows - 1
      If Tabela.ValueMatrix(k, 1) = -1 Then
         Set verqualidade = New ADODB.Recordset
         verqualidade.Open "select * from SMP_Tabela_MPrimas where CodigoMP = '" & Tabela.TextMatrix(k, 11) & "'", cpcOT, adOpenStatic
         If verqualidade.Fields("controloqualidade").Value = False Then
            Set getultima = New ADODB.Recordset
            getultima.Open "select * from CQ_Analises where tipo = 'GR' and documento = " & Tabela.TextMatrix(k, 9) & "", cpcOT, adOpenStatic
            getultima.MoveLast
            analise = getultima.Fields("idanalise").Value
            conforme = 1
            estadoCQ = 1
            Set gravaanalises = New ADODB.Recordset
            gravaanalises.Open "update CQ_Analises set conforme = '" & conforme & "', dataanalise = '" & Date & "', obs = 'Sem Controlo Qualidade', L_DataSistemaEntrada = '" & Date & "', L_HoraSistemaEntrada = '" & Time & "' ,L_UserWindows = '" & Utilizador & "', L_UserCPC = '" & userCQ & "', L_Computador = '" & Computador & "' where idanalise = " & analise & "", cpcOT, adOpenStatic
            Set gravapara = New ADODB.Recordset
            gravapara.Open "insert into CQ_Analises_Registos (IDanalise, IDEspecificacao, ValorObservado,DataSistemaEntrada, HoraSistemaEntrada, UserWindows, usercpc, Computador) values ('" & analise & "','26','OK', '" & Date & "','" & Time & "','" & Utilizador & "','" & userCQ & "','" & Computador & "')", cpcOT, adOpenStatic

            'update Estado CQ Guias Recepção
            Set cq = New ADODB.Recordset
            cq.Open "update SMP_Tabela_GuiasRecepcao set EstadoCQ = '" & estadoCQ & "', L_DataSistemaEntrada = '" & Date & "', L_HoraSistemaEntrada = '" & Time & "' ,L_UserWindows = '" & Utilizador & "', L_UserCPC = '" & userCQ & "',
```

```
L_Computador = '" & Computador & "' where GR = " & Tabela.TextMatrix(k, 9) & "", cpcOT, adOpenStatic


            'update stocks
            Set stocks = New ADODB.Recordset
            stocks.Open "select * from SMP_Tabela_Mprimas where codigoMP = '" & Tabela.TextMatrix(k, 11) & "'", cpcOT, adOpenStatic
            stockreal = stocks.Fields("Stockreal").Value + Tabela.TextMatrix(Tabela.RowSel, 5)
            stocks.Close
            Set stocks = New ADODB.Recordset
            stocks.Open "update SMP_Tabela_Mprimas set stockreal = '" & stockreal & "', ultimaentrada='" & Date & "', L_DataSistemaEntrada = '" & Date & "', L_HoraSistemaEntrada = '" & Time & "' ,L_UserWindows = '" & Utilizador & "', L_UserCPC = '" & userCQ & "', L_Computador = '" & Computador & "' where codigoMP = '" & Tabela.TextMatrix(k, 11) & "'", cpcOT, adOpenStatic
            'update movimentos MP's
            Set movimentos = New ADODB.Recordset
            Set movimentos2 = New ADODB.Recordset
            movimentos.Open "select * from SMP_Tabela_GuiasRecepcao where GR = " & Tabela.TextMatrix(k, 9) & "", cpcOT, adOpenStatic
            If movimentos.Fields("Nencomenda").Value = 0 Then
              Set movimentos2 = New ADODB.Recordset
              movimentos2.Open "insert into SMP_MovimentosSTocks (CodigoMP, Documento, " & _
              " numerodoc, quantidade, obs, data, DataSistemaEntrada, HoraSistemaEntrada, UserWindows, usercpc, Computador) values ('" & Tabela.TextMatrix(k, 11) & "', " & _
              " '" & movimentos.Fields("tipodoc").Value & "','" & movimentos.Fields("numerodoc").Value & "','" & Tabela.TextMatrix(Tabela.RowSel, 5) & "','-', '" & Date & "', '" & Date & "','" & Time & "','" & Utilizador & "','" & userCQ & "','" & Computador & "')", cpcOT, adOpenStatic
            Else
```

```
                movimentos2.Open "insert into SMP_MovimentosSTocks (CodigoMP,
Documento, " & _
                " numerodoc, quantidade, obs, data, DataSistemaEntrada,
HoraSistemaEntrada, UserWindows, usercpc, Computador) values ('" &
Tabela.TextMatrix(k, 11) & "', " & _
                " 'NE','" & movimentos.Fields("NEncomenda").Value & "','" &
Tabela.TextMatrix(Tabela.RowSel, 5) & "','-','" & Date & "', '" & Date & "','" & Time & "','"
& Utilizador & "','" & userCQ & "','" & Computador & "')", cpcOT, adOpenStatic
                End If
              End If
            End If
          Next k
      Erro:
          If Err = 0 Then
            Err.Clear
            Form_Load
          Else
            MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number) & " - Transacção não efectuada!", vbCritical, App.Title
            Err.Clear
            Form_Load
            Exit Sub
          End If
      End Sub

      Private Sub Command2_Click()
          Unload Me
      End Sub




      Private Sub Command3_Click()
          Encargos.Rows = Encargos.Rows + 1
      End Sub
```

```
Private Sub Command4_Click()
   If Encargos.Rows = 1 Then
      MsgBox "Não pode eliminar mais linhas!", vbInformation, App.Title
      Exit Sub
   Else
      Encargos.RemoveItem (Encargos.RowSel)
   End If
End Sub

Private Sub Dvalidade_Change()
On Error GoTo Erro
   Tabela.TextMatrix(Tabela.RowSel, 7) = DValidade.Value

Erro:
   If Err = 0 Then
      Err.Clear
   Else
      MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number), vbCritical, App.Title
      Err.Clear
      Exit Sub
   End If
End Sub

Private Sub Form_Activate()
   Form_Load
End Sub

Private Sub Form_Load()
On Error GoTo Erro
   Me.Top = 10
   Me.Left = 10
   DValidade.Value = Date
```

```
        Data = Date
        Combo1.Clear
        Combo2.Clear
        Text2.Text = ""
        Encargos.Clear
        Encargos.Rows = 1
        Encargos.TextMatrix(0, 1) = "Descrição"
        Encargos.TextMatrix(0, 2) = "Valor (Euro)"
        Tabela.Rows = 1
        GR = 0
        Set ne = New ADODB.Recordset
        ne.Open "select * from SMP_NE_Mprimas where estado <> 1 and estado <> 4 order
by numencomenda ", cpcOT, adOpenStatic
        If ne.RecordCount = 0 Then
           Exit Sub
        Else
           ne.MoveFirst
           Do While Not ne.EOF
              Set forne = New ADODB.Recordset
              forne.Open "select * from SMP_Tabela_Fornecedores WHERE CodFornecedor
= " & ne.Fields("CodFornecedor").Value & "", cpcOT, adOpenStatic
              Combo1.AddItem "Encomenda " & ne.Fields("numencomenda").Value & " ("
& forne.Fields("nomeempresa").Value & ")"
              ne.MoveNext
           Loop
           Set Doc = New ADODB.Recordset
           Doc.Open "select * from SMP_Tabela_TiposDocumentos", cpcOT, adOpenStatic
           Doc.MoveFirst
           Do While Not Doc.EOF
              Combo2.AddItem Doc.Fields("IDTIpoDocumento") & " - " &
Doc.Fields("descricao").Value
              Doc.MoveNext
           Loop
        End If
```

```
Erro:
    If Err = 0 Then
        Err.Clear
    Else
        MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" & Str(Err.Number), vbCritical, App.Title
        Err.Clear
    End If
End Sub


Private Sub Form_Unload(Cancel As Integer)
    Unload Me
End Sub


Private Sub Tabela_DblClick()
On Error GoTo Erro
    If Tabela.ColSel = 1 Then
        Tabela.Editable = flexEDKbdMouse
        Set getultima = New ADODB.Recordset
        getultima.Open "select * from SMP_Tabela_GuiasRecepcao order by GR", cpcOT, adOpenStatic
        If getultima.RecordCount = 0 Then
            GR = 0
        Else
            getultima.MoveLast
            GR = getultima.Fields("GR").Value
        End If
        For l = 1 To Tabela.Rows - 1
            Tabela.TextMatrix(l, 9) = ""
        Next l
        For k = 1 To Tabela.Rows - 1
            If Tabela.ValueMatrix(k, 1) = -1 Then
```

```
                GR = GR + 1
                Tabela.TextMatrix(k, 9) = GR
              End If
            Next k
          End If
        Erro:
          If Err = 0 Then
            Err.Clear
          Else
            MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number), vbCritical, App.Title
            Err.Clear
          End If
        End Sub

        Private Sub tabela_KeyPressEdit(ByVal Row As Long, ByVal Col As Long, KeyAscii
As Integer)
        On Error GoTo Erro
          If Tabela.Col = 5 Then
            mvalidacao.ValidaDecimalNoGrid Tabela, KeyAscii, 6
          End If
          If Tabela.Col = 4 Then
            mvalidacao.ValidaDecimalNoGrid Tabela, KeyAscii, 6
          End If
          If Tabela.Col = 7 Then
            mvalidacao.ValidaDecimalNoGrid Tabela, KeyAscii, 6
          End If
          If Tabela.ColSel = 1 Then
            Tabela.Editable = flexEDKbdMouse
            Set getultima = New ADODB.Recordset
            getultima.Open "select * from SMP_Tabela_GuiasRecepcao order by GR",
cpcOT, adOpenStatic
            If getultima.RecordCount = 0 Then
              GR = 0
```

```
            Else
               getultima.MoveLast
               GR = getultima.Fields("GR").Value
            End If
            For l = 1 To Tabela.Rows - 1
               Tabela.TextMatrix(l, 9) = ""
            Next l
            For k = 1 To Tabela.Rows - 1
               If Tabela.ValueMatrix(k, 1) = -1 Then
                  GR = GR + 1
                  Tabela.TextMatrix(k, 9) = GR
               End If
            Next k
         End If
      Erro:
         If Err = 0 Then
            Err.Clear
         Else
            MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number), vbCritical, App.Title
            Err.Clear
         End If
      End Sub


      Private Sub Tabela_Click()
      On Error GoTo Erro
         Tabela.Editable = flexEDNone
         If Tabela.ColSel = 1 Then
            Tabela.Editable = flexEDKbdMouse
            Set getultima = New ADODB.Recordset
            getultima.Open "select * from SMP_Tabela_GuiasRecepcao order by GR",
cpcOT, adOpenStatic
            If getultima.RecordCount = 0 Then
               GR = 0
```

```
            Else
               getultima.MoveLast
               GR = getultima.Fields("GR").Value
            End If
            For l = 1 To Tabela.Rows - 1
               Tabela.TextMatrix(l, 9) = ""
            Next l
            For k = 1 To Tabela.Rows - 1
               If Tabela.ValueMatrix(k, 1) = -1 Then
                  GR = GR + 1
                  Tabela.TextMatrix(k, 9) = GR
               End If
            Next k
         End If
         If Tabela.ColSel = 5 Then
            Tabela.Editable = flexEDKbdMouse
         End If
         If Tabela.ColSel = 6 Then
            Tabela.Editable = flexEDKbdMouse
         End If
         If Tabela.ColSel = 7 Then
            If Tabela.TextMatrix(Tabela.RowSel, 14) = 1 Then
               Tabela.Editable = flexEDKbdMouse
            Else
               Tabela.Editable = flexEDNone
            End If
         End If
      Erro:
         If Err = 0 Then
            Err.Clear
         Else
            MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" &
Str(Err.Number), vbCritical, App.Title
            Err.Clear
```

```
        End If
    End Sub

    Private Sub Tabela_BeforeEdit(ByVal Row As Long, ByVal Col As Long, Cancel As Boolean)
    On Error GoTo Erro
        If Tabela.ColDataType(Col) = flexDTDate Then
            If Tabela.TextMatrix(Tabela.RowSel, 14) = 1 Then
                DValidade.Move Tabela.CellLeft, Tabela.CellTop, Tabela.CellWidth, Tabela.CellHeight
            'If Tabela <> "" Then
                'DataP.Value = Tabela.Text
                'DataP.Tag = Tabela.Text
            'Else
            '   Tabela = DataP.Value
            'End If
                DValidade.Visible = True
                DValidade.SetFocus
            End If
        Else
            DValidade.Visible = False
        End If
    Erro:
        If Err = 0 Then
            Err.Clear
        Else
            MsgBox "Ocorreu o seguinte erro: " & Err.Description & " - Nr.:" & Str(Err.Number), vbCritical, App.Title
            Err.Clear
            Exit Sub
        End If
    End Sub
```

```
Private Sub encargos_KeyPressEdit(ByVal Row As Long, ByVal Col As Long, KeyAscii As Integer)
    If Encargos.Col = 2 Then
        mvalidacao.ValidaDecimalNoGrid Encargos, KeyAscii, 7
    End If
End Sub
```

3. LabGest® code sample

Here is a sample of the code computed at the production form.

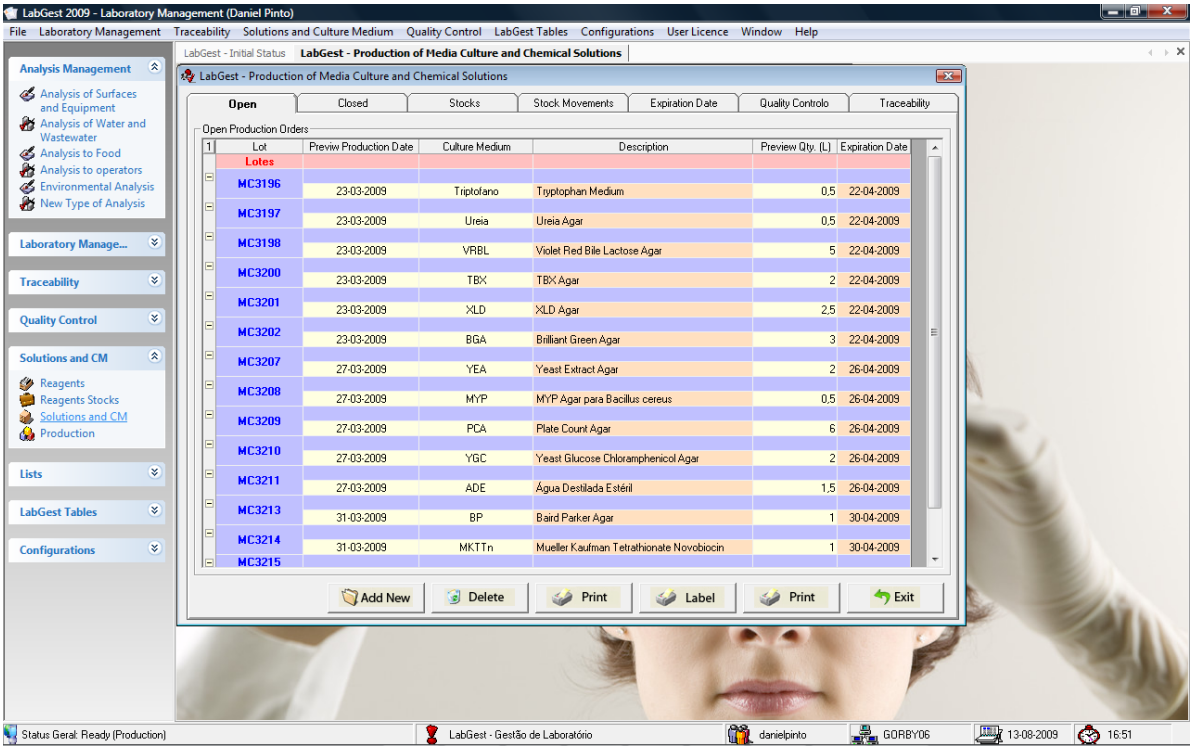

Figure A.0.6: Production form

```
Dim pp As ADODB.Recordset
Dim pp1 As ADODB.Recordset
Dim pp2 As ADODB.Recordset
Dim anula As ADODB.Recordset
Dim stocksemiactual As Integer
Dim datainisem As String
Dim datafimsem As String
Dim Data1 As Date
Dim Data2 As Date
Dim getstock As ADODB.Recordset
Dim elimina As ADODB.Recordset
Dim reagentes1 As ADODB.Recordset
Dim IDReagente As String
Dim lertodosv As Integer
```

```
Dim abrir As Word.Application
Dim i As Integer
Dim ppCQ As ADODB.Recordset
Public origem As Integer
Dim lotes As ADODB.Recordset
Dim meios As ADODB.Recordset
Dim guias As ADODB.Recordset
Dim leumeios As Integer
Dim leulotes As Integer
Dim leuguias As Integer
Dim producao As ADODB.Recordset
Dim idmeio As String
Dim getb As ADODB.Recordset

Private Sub cMP_Click()
On Error GoTo erro
   If cMP.ListIndex = -1 Then
      Exit Sub
   End If
   Dim pretendido As Integer
   pretendido = cMP.ListIndex + 1
   Dim inicial As Variant
   reagentes1.MoveFirst
   inicial = reagentes1.Bookmark
   reagentes1.Move pretendido - 1, inicial
   IDReagente = reagentes1.Fields("referencia").Value
   lertodosv = 0
   LerMovimentos
erro:
   ErrosGerais
End Sub

Private Sub Combo1_Click()
   If Combo1.ListIndex = -1 Then
```

```
      Exit Sub
   End If
   Dim pretendido As Integer
   pretendido = Combo1.ListIndex + 1
   Dim inicial As Variant
   meios.MoveFirst
   inicial = meios.Bookmark
   meios.Move pretendido - 1, inicial
   idmeio = meios.Fields("referencia").Value
   LerFiltro2
End Sub

Private Sub Combo1_GotFocus()
On Error GoTo erro
   If leumeios = 0 Then
      Combo1.Clear
      Set meios = New ADODB.Recordset
      meios.Open "EXEC Get_MeiosCultura_Codigos", LabDB, adOpenStatic
      If meios.RecordCount = 0 Then
         Exit Sub
      Else
         Do While Not meios.EOF
            Combo1.AddItem meios.Fields("referencia").Value & " - " &
meios.Fields("descricao").Value
            meios.MoveNext
         Loop
      End If
      leumeios = 1
   End If
erro:
   ErrosGerais
End Sub

Private Sub Combo2_Click()
```

```
    If Combo2.ListIndex = -1 Then
        Exit Sub
    End If
    LerFiltro
End Sub

Private Sub Combo2_GotFocus()
On Error GoTo erro
    If leulotes = 0 Then
        Combo2.Clear
        Set lotes = New ADODB.Recordset
        lotes.Open "EXEC Get_MeiosCultura_Lotes", LabDB, adOpenStatic
        If lotes.RecordCount = 0 Then
            Exit Sub
        Else
            Do While Not lotes.EOF
                Combo2.AddItem lotes.Fields("lote").Value
                lotes.MoveNext
            Loop
            leulotes = 1
        End If
    End If
erro:
    ErrosGerais
End Sub

Private Sub Combo3_Click()
    If Combo3.ListIndex = -1 Then
        Exit Sub
    End If
    LerFiltro3
End Sub

Private Sub Combo3_GotFocus()
```

```
On Error GoTo erro
   If leuguias = 0 Then
      Combo3.Clear
      Set guias = New ADODB.Recordset
      guias.Open "select * from tabela_reagentes_ge order by ge", LabDB, adOpenStatic
      If guias.RecordCount = 0 Then
         Exit Sub
      Else
         Do While Not guias.EOF
            Combo3.AddItem guias.Fields("ge").Value
            guias.MoveNext
         Loop
         leuguias = 1
      End If
   End If
erro:
   ErrosGerais
End Sub

Private Sub Command1_Click()
On Error GoTo erro
   Set VerPerm = New ADODB.Recordset
   VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador
& "", LabDB, adOpenStatic
   If VerPerm.Fields("Check69").Value = 0 Then
      MsgBox "You do not have permissions to access the requested. Please contact System
Administrator!", vbInformation, App.Title
      Exit Sub
   End If
   If TabelaA.RowSel >= 1 Then
      If TabelaA.TextMatrix(TabelaA.RowSel, 2) = "" Then
         Exit Sub
      End If
      Set imprime = New ADODB.Recordset
```

```
        imprime.Open "update tabela_MeiosCultura_Producao set aimprimir = 0 ", LabDB, adOpenStatic
        Set imprime = New ADODB.Recordset
        imprime.Open "update tabela_Meioscultura_Producao set aimprimir = 1 where pproducao = " & TabelaA.TextMatrix(TabelaA.RowSel, 2) & "", LabDB, adOpenStatic
        crt1.ReportFileName = caminhoprincipal & "\Modelos Crystal\OrdemFabrico.rpt"
        crt1.WindowState = crptMaximized
        crt1.DiscardSavedData = True
        crt1.WindowTitle = "Produção de Meio de Cultura"
        crt1.Action = 1
        Screen.MousePointer = 99
        Screen.MousePointer = 99
    Else
        Exit Sub
    End If
erro:
    ErrosGerais
End Sub

Private Sub Command11_Click()
On Error GoTo erro
    lertodosv = 1
    cMP.Text = ""
    LerMovimentos
erro:
    ErrosGerais
End Sub

Private Sub Command12_Click()
    Unload Me
End Sub

Private Sub Command13_Click()
On Error GoTo erro
```

```
Set VerPerm = New ADODB.Recordset
VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador & "", LabDB, adOpenStatic
If VerPerm.Fields("Check70").Value = 0 Then
   MsgBox "You do not have permissions to access the requested. Please contact System Administrator!", vbInformation, App.Title
   Exit Sub
End If
If TabelaA.RowSel >= 1 Then
   If TabelaA.TextMatrix(TabelaA.RowSel, 2) = "" Then
      Exit Sub
   End If
Else
   Exit Sub
End If
Set imprime = New ADODB.Recordset
imprime.Open "update tabela_MeiosCultura_Producao set aimprimir = 0 ", LabDB, adOpenStatic
Set imprime = New ADODB.Recordset
imprime.Open "update tabela_Meioscultura_Producao set aimprimir = 1 where pproducao = " & TabelaA.TextMatrix(TabelaA.RowSel, 2) & "", LabDB, adOpenStatic
Set abrir = New Word.Application
abrir.Documents.Open caminhoprincipal & "\Modelos Word\Etiquetas_MC.docx"
abrir.Visible = False
abrir.Windows("Etiquetas_MC.docx").Activate
abrir.ActiveDocument.MailMerge.MainDocumentType = wdFormLetters

abrir.Windows("Etiquetas_MC.docx").Activate
abrir.ActiveDocument.MailMerge.OpenDataSource Name:= _
caminhoprincipal & "\Modelos Word\ImpressoEtiquetasMC.odc" _
, ConfirmConversions:=False, ReadOnly:=False, LinkToSource:=True, _
AddToRecentFiles:=False, PasswordDocument:="", PasswordTemplate:="", _
WritePasswordDocument:="", WritePasswordTemplate:="", Revert:=False, _
Format:=wdOpenFormatAuto, Connection:= _
```

```
   "Provider=MSDASQL.1;Persist Security Info=True;Extended
Properties=""DSN=LabGest;UID=Labgest;APP=2007 Microsoft Office
system;WSID=GORBY02;DATABASE=LabGest;Trusted_Connection=Yes"";Initial
Catalog=LabGest" _
   , SQLStatement:="SELECT * FROM ""ImpressoEtiquetasMC""", SQLStatement1 _
   :="", SubType:=wdMergeSubTypeOther
   abrir.ActiveDocument.MailMerge.DataSource.QueryString = _
   "SELECT * FROM [ImpressoEtiquetasMC]"
   With abrir.ActiveDocument.MailMerge
      .Destination = wdSendToNewDocument
      .SuppressBlankLines = True
      With .DataSource
         .FirstRecord = wdDefaultFirstRecord
         .LastRecord = wdDefaultLastRecord
      End With
      .Execute pause:=False
   End With
   abrir.Windows("Etiquetas_MC.docx").Activate
   abrir.Windows("Etiquetas_MC.docx").Close (n)
   abrir.ActiveDocument.Activate
   abrir.Visible = True
   Set abrir = Nothing
erro:
   ErrosGerais
End Sub

Private Sub Command14_Click()
On Error GoTo erro
   Set VerPerm = New ADODB.Recordset
   VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador
& "", LabDB, adOpenStatic
   If VerPerm.Fields("Check71").Value = 0 Then
      MsgBox "You do not have permissions to access the requested. Please contact System
Administrator!", vbInformation, App.Title
```

```
    Exit Sub
  End If

  Set imprime = New ADODB.Recordset
  imprime.Open "update tabela_MeiosCultura_Producao set aimprimir = 0 ", LabDB, adOpenStatic
  Set imprime = New ADODB.Recordset
  imprime.Open "update tabela_Meioscultura_Producao set aimprimir = 1 where pproducao = " & TabelaA.TextMatrix(TabelaA.RowSel, 2) & "", LabDB, adOpenStatic
  Set abrir = New Word.Application
  abrir.Documents.Open caminhoprincipal & "\Modelos Word\Etiquetas_MC.docx"
  abrir.Visible = False
  abrir.Windows("Etiquetas_MC.docx").Activate
  abrir.ActiveDocument.MailMerge.MainDocumentType = wdFormLetters

  abrir.Windows("Etiquetas_MC.docx").Activate
  abrir.ActiveDocument.MailMerge.OpenDataSource Name:= _
  caminhoprincipal & "\Modelos Word\ImpressoEtiquetasMC.odc" _
  , ConfirmConversions:=False, ReadOnly:=False, LinkToSource:=True, _
  AddToRecentFiles:=False, PasswordDocument:="", PasswordTemplate:="", _
  WritePasswordDocument:="", WritePasswordTemplate:="", Revert:=False, _
  Format:=wdOpenFormatAuto, Connection:= _
  "Provider=MSDASQL.1;Persist Security Info=True;Extended Properties=""DSN=LabGest;UID=Labgest;APP=2007 Microsoft Office system;WSID=GORBY02;DATABASE=LabGest;Trusted_Connection=Yes"";Initial Catalog=LabGest" _
  , SQLStatement:="SELECT * FROM ""ImpressoEtiquetasMC""", SQLStatement1 _
  :="", SubType:=wdMergeSubTypeOther
  abrir.ActiveDocument.MailMerge.DataSource.QueryString = _
  "SELECT * FROM [ImpressoEtiquetasMC]"
  With abrir.ActiveDocument.MailMerge
    .Destination = wdSendToNewDocument
    .SuppressBlankLines = True
    With .DataSource
```

```
            .FirstRecord = wdDefaultFirstRecord
            .LastRecord = wdDefaultLastRecord
        End With
        .Execute pause:=False
    End With
    abrir.Windows("Etiquetas_MC.docx").Activate
    abrir.Windows("Etiquetas_MC.docx").Close (no)
    abrir.ActiveDocument.Activate
    abrir.Visible = True
    'abrir.ActivePrinter = "\\cpc01\Zebra TLP2844"
    'abrir.Application.PrintOut FileName:="", Range:=wdPrintAllDocument, Item:= _
    wdPrintDocumentContent, Copies:=1, Pages:="", PageType:=wdPrintAllPages, _
    ManualDuplexPrint:=False, Collate:=True, Background:=True, PrintToFile:= _
    False, PrintZoomColumn:=0, PrintZoomRow:=0, PrintZoomPaperWidth:=0, _
    PrintZoomPaperHeight:=0
    'abrir.ActiveDocument.Close (0)
    Set abrir = Nothing
erro:
    ErrosGerais
End Sub

Private Sub Command15_Click()
    Set VerPerm = New ADODB.Recordset
    VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador
& "", LabDB, adOpenStatic
    If VerPerm.Fields("Check69").Value = 0 Then
        MsgBox "You do not have permissions to access the requested. Please contact System
Administrator!", vbInformation, App.Title
        Exit Sub
    End If
    frmConsultaProducao.Enabled = False
    frmConsultaProducaoImpressao.Show
End Sub
```

```
Private Sub Command16_Click()
On Error GoTo erro
   Set VerPerm = New ADODB.Recordset
   VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador & "", LabDB, adOpenStatic
   If VerPerm.Fields("Check71").Value = 0 Then
      MsgBox "You do not have permissions to access the requested. Please contact System Administrator!", vbInformation, App.Title
      Exit Sub
   End If

   Set imprime = New ADODB.Recordset
   imprime.Open "update tabela_MeiosCultura_Producao set aimprimir = 0 ", LabDB, adOpenStatic
   Set imprime = New ADODB.Recordset
   imprime.Open "update tabela_Meioscultura_Producao set aimprimir = 1 where lote = '" & Tabela3.TextMatrix(Tabela3.RowSel, 3) & "'", LabDB, adOpenStatic
   Set abrir = New Word.Application
   abrir.Documents.Open caminhoprincipal & "\Modelos Word\Etiquetas_MCConfirmado.docx"
   abrir.Visible = False
   abrir.Windows("Etiquetas_MCConfirmado.docx").Activate
   abrir.ActiveDocument.MailMerge.MainDocumentType = wdFormLetters

   abrir.Windows("Etiquetas_MCConfirmado.docx").Activate
   abrir.ActiveDocument.MailMerge.OpenDataSource Name:= _
   caminhoprincipal & "\Modelos Word\ImpressoEtiquetasMC.odc" _
   , ConfirmConversions:=False, ReadOnly:=False, LinkToSource:=True, _
   AddToRecentFiles:=False, PasswordDocument:="", PasswordTemplate:="", _
   WritePasswordDocument:="", WritePasswordTemplate:="", Revert:=False, _
   Format:=wdOpenFormatAuto, Connection:= _
   "Provider=MSDASQL.1;Persist Security Info=True;Extended Properties=""DSN=LabGest;UID=Labgest;APP=2007 Microsoft Office
```

```
system;WSID=GORBY02;DATABASE=LabGest;Trusted_Connection=Yes"";Initial
Catalog=LabGest" _
   , SQLStatement:="SELECT * FROM ""ImpressoEtiquetasMC""", SQLStatement1 _
   :="", SubType:=wdMergeSubTypeOther
   abrir.ActiveDocument.MailMerge.DataSource.QueryString = _
   "SELECT * FROM [ImpressoEtiquetasMC]"
   With abrir.ActiveDocument.MailMerge
      .Destination = wdSendToNewDocument
      .SuppressBlankLines = True
      With .DataSource
         .FirstRecord = wdDefaultFirstRecord
         .LastRecord = wdDefaultLastRecord
      End With
      .Execute pause:=False
   End With
   abrir.Windows("Etiquetas_MCConfirmado.docx").Activate
   abrir.Windows("Etiquetas_MCConfirmado.docx").Close (no)
   abrir.ActiveDocument.Activate
   abrir.Visible = True
   'abrir.ActivePrinter = "\\cpc01\Zebra TLP2844"
   'abrir.Application.PrintOut FileName:="", Range:=wdPrintAllDocument, Item:= _
   wdPrintDocumentContent, Copies:=1, Pages:="", PageType:=wdPrintAllPages, _
   ManualDuplexPrint:=False, Collate:=True, Background:=True, PrintToFile:= _
   False, PrintZoomColumn:=0, PrintZoomRow:=0, PrintZoomPaperWidth:=0, _
   PrintZoomPaperHeight:=0
   'abrir.ActiveDocument.Close (0)
   Set abrir = Nothing
erro:
   ErrosGerais
End Sub

Private Sub Command17_Click()
On Error GoTo erro
   Tabela3.Rows = 1
```

```
    Set getstock = New ADODB.Recordset
    getstock.Open "EXEC GET_MeiosCultura_Stocks", LabDB, adOpenStatic
    If getstock.RecordCount = 0 Then
        Exit Sub
    End If
    getstock.MoveFirst
    linha = 1
    Tabela3.Rows = getstock.RecordCount + 1
    Do While Not getstock.EOF
        Tabela3.TextMatrix(linha, 1) = getstock.Fields("referencia").Value
        Tabela3.TextMatrix(linha, 2) = getstock.Fields("descricao").Value
        Tabela3.TextMatrix(linha, 3) = getstock.Fields("lote").Value
        Tabela3.TextMatrix(linha, 10) = getstock.Fields("lote").Value
        If getstock.Fields("dataproducao").ActualSize <> 0 Then
            Tabela3.TextMatrix(linha, 4) = Format(getstock.Fields("dataproducao").Value,
"dd/mm/yyyy")
        Else
            Tabela3.TextMatrix(linha, 4) = Format(getstock.Fields("data").Value,
"dd/mm/yyyy")
        End If
        Tabela3.TextMatrix(linha, 5) = getstock.Fields("qdeproduzida").Value
        Tabela3.TextMatrix(linha, 6) = getstock.Fields("stockpp").Value
        Tabela3.TextMatrix(linha, 7) = Format(getstock.Fields("datavalidade").Value,
"dd/mm/yyyy")
        If getstock.Fields("datavalidade").Value < Date Then
            FValidade.Rows = FValidade.Rows + 1
            FValidade.TextMatrix(FValidade.Rows - 1, 1) = getstock.Fields("referencia").Value
            FValidade.TextMatrix(FValidade.Rows - 1, 2) = getstock.Fields("descricao").Value
            FValidade.TextMatrix(FValidade.Rows - 1, 3) = getstock.Fields("lote").Value
            If getstock.Fields("dataproducao").ActualSize <> 0 Then
                FValidade.TextMatrix(FValidade.Rows - 1, 4) =
Format(getstock.Fields("dataproducao").Value, "dd/mm/yyyy")
            Else
```

```
            FValidade.TextMatrix(FValidade.Rows - 1, 4) = Format(getstock.Fields("data").Value, "dd/mm/yyyy")
        End If
        FValidade.TextMatrix(FValidade.Rows - 1, 5) = getstock.Fields("qdeproduzida").Value
        FValidade.TextMatrix(FValidade.Rows - 1, 6) = getstock.Fields("stockpp").Value
        FValidade.TextMatrix(FValidade.Rows - 1, 7) = Format(getstock.Fields("datavalidade").Value, "dd/mm/yyyy")
        FValidade.TextMatrix(FValidade.Rows - 1, 8) = getstock.Fields("usercpc").Value
    End If
    Tabela3.TextMatrix(linha, 8) = getstock.Fields("usercpc").Value
    linha = linha + 1
    getstock.MoveNext
  Loop
  m_Fmt = "(#,##0.00)"
  Dim r%, c%
  r = Tabela3.Row
  c = Tabela3.Col
  Tabela3.Col = 0
  Tabela3.ColSel = 2
  Tabela3.Subtotal flexSTClear
  A = Tabela3.ColSel
  Tabela3.Subtotal flexSTSum, 1, 5, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True
  Tabela3.Subtotal flexSTSum, 1, 6, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True
  Tabela3.Row = r
  Tabela3.Col = c
  Tabela3.OutlineBar = flexOutlineBarComplete
  Tabela3.AllowUserResizing = flexResizeColumns
  Tabela3.MergeCol(1) = True
  Tabela3.MergeCol(2) = True
  Tabela3.MergeCol(3) = True
  Tabela3.MergeCol(0) = True
  Tabela3.MergeCells = flexMergeFree
  Tabela3.Outline 2
```

```
erro:
   ErrosGerais
End Sub


Private Sub Command18_Click()
On Error GoTo erro
   Combo1.Clear
   Combo2.Clear
   Combo3.Clear
   leuguias = 0
   leumeios = 0
   leulotes = 0
   Tree.Nodes.Clear
   Set producao = New ADODB.Recordset
   producao.Open "EXEC Get_Meioscultura_Producao @confirmado = 1", LabDB, adOpenStatic
   If producao.RecordCount = 0 Then
      Exit Sub
   End If
   producao.MoveFirst
   Do While Not producao.EOF
      Set nodX = Tree.Nodes.Add(, , "T" & producao.Bookmark, "Rastreabilidade da ordem de fabrico " & producao.Fields("lote").Value)
      nodX.Image = 8
      nodX.Bold = True
      Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6G" & producao.Fields("pproducao").Value & producao.Bookmark, "Dados Gerais")
      nodX.Image = 13
      Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6R" & producao.Fields("pproducao").Value & producao.Fields("idmeio").Value & producao.Bookmark, "Reagentes (Rastreabilidade)")
      nodX.Image = 13
      Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6C" & producao.Fields("pproducao").Value & producao.Bookmark, "Controlo da Qualidade")
```

```
nodX.Image = 13
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6B" & producao.Fields("pproducao").Value & producao.Bookmark, "Certificados de Análise associados")
nodX.Image = 13
Set getdadosproduto = New ADODB.Recordset
getdadosproduto.Open "select * from tabela_Meioscultura_producao where lote = '" & producao.Fields("lote").Value & "'", LabDB, adOpenStatic
If getdadosproduto.RecordCount <> 0 Then
  Set getdadosproduto2 = New ADODB.Recordset
  getdadosproduto2.Open "EXEC GET_MeiosCultura_Producao_Confirma @pp = '" & getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
  If getdadosproduto2.RecordCount <> 0 Then
    Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6C" & getdadosproduto2.Bookmark & producao.Bookmark & getdadosproduto2.Fields("pproducao").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Produzido em " & getdadosproduto2.Fields("data").Value & " com validade até " & getdadosproduto2.Fields("datavalidade").Value)
    nodX.Image = 14
    Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6C1" & getdadosproduto2.Bookmark & producao.Bookmark & getdadosproduto2.Fields("pproducao").Value & producao.Fields("pproducao").Value, "Quantidade produzida: " & getdadosproduto2.Fields("qdeprevista").Value)
    nodX.Image = 14
  End If
End If
Set rastre = New ADODB.Recordset
rastre.Open "EXEC GET_MeiosCultura_Producao_Confirma_2 @pp = '" & getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
If rastre.RecordCount <> 0 Then
  rastre.MoveFirst
  Do While Not rastre.EOF
```

```
If rastre.Fields("tipo").Value = 1 Then
   unidade = "Litros"
Else
   unidade = "Grama"
End If
If externo = 1 Then
   Set getdados = New ADODB.Recordset
   getdados.Open "SELECT dbo.Item.ItemID, dbo.Item.BarCode, dbo.Item.familyid, dbo.ItemNames.ShortDescription, dbo.ItemNames.Description " & _
   " FROM dbo.Item INNER JOIN " & _
   " dbo.ItemNames ON dbo.Item.ItemID = dbo.ItemNames.ItemID " & _
   " where dbo.item.itemid = '" & rastre.Fields("idreagente").Value & "'", GesPos, adOpenStatic
Else
   getdados.Open "SELECT * from tabela_reagentes where dbo.item.itemid = '" & rastre.Fields("idreagente").Value & "'", LabDB, adOpenStatic
End If
If getdados.RecordCount <> 0 Then
   Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value & producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" & getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: " & getdados.Fields("description").Value & " (" & rastre.Fields("idreagente").Value & ") - Guia de Entrada: " & rastre.Fields("GE").Value & ". Quantidade consumida: " & rastre.Fields("Quantidadereal").Value & " " & unidade)
   nodX.Image = 14
Else
   Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value & producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" & getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: não detectado na base de dados GesPOS (" & rastre.Fields("idreagente").Value & ") - Guia de Entrada: " & rastre.Fields("GE").Value & ". Quantidade consumida: " & rastre.Fields("Quantidadereal").Value & " " & unidade)
```

```
            nodX.Image = 14
          End If
          rastre.MoveNext
        Loop
    End If
    Set getplano = New ADODB.Recordset
    getplano.Open "SELECT TOP (100) PERCENT
dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo,
dbo.Tabela_ControloQualidade_PlanoFinal.IDControloPlano,
dbo.Tabela_ControloQualidade_PlanoFinal.Produto,
dbo.Tabela_ControloQualidade_PlanoFinal.resultado,
dbo.Tabela_ControloQualidade_PlanoFinal.resultadonumerico, " & _
    " dbo.Tabela_ControloQualidade_PlanoFinal.Lote,
dbo.Tabela_ControloQualidade_PlanoFinal.Efectuado,
dbo.Tabela_ControloQualidade_Controlos.Descricao, " & _
    " dbo.Tabela_ControloQualidade_Controlos.Valor ,
dbo.Tabela_ControloQualidade_Controlos.vmin,
dbo.Tabela_ControloQualidade_Controlos.vmax,
dbo.Tabela_ControloQualidade_planofinal.idcontroloplano,
dbo.Tabela_ControloQualidade_planofinal.idcontrolo " & _
    " FROM dbo.Tabela_ControloQualidade_PlanoFinal INNER JOIN " & _
    " dbo.Tabela_ControloQualidade_Controlos ON
dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo =
dbo.Tabela_ControloQualidade_Controlos.IDControlo " & _
    " WHERE (dbo.Tabela_ControloQualidade_PlanoFinal.Lote = '" &
getdadosproduto.Fields("lote").Value & "') " & _
    " ORDER BY dbo.Tabela_ControloQualidade_Controlos.Descricao", LabDB,
adOpenStatic
    If getplano.RecordCount <> 0 Then
      getplano.MoveFirst
      Do While Not getplano.EOF
        Set nodX = Tree.Nodes.Add("R6C" & producao.Fields("pproducao").Value &
producao.Bookmark, tvwChild, "R6R2" & getplano.Bookmark &
```

```
getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Controlo Qualitativo " & getplano.Fields("descricao").Value)
            nodX.Image = 14
            Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R3" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor mínimo especificado: " & getplano.Fields("vmin").Value)
            nodX.Image = 12
            Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R4" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor máximo especificado: " & getplano.Fields("vmax").Value)
            nodX.Image = 12
            Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R5" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Resultado numérico obtido: " & getplano.Fields("resultadonumerico").Value)
            nodX.Image = 12
            Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R6" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Resultado alfanumérico obtido: " & getplano.Fields("resultado").Value)
            nodX.Image = 12
            getplano.MoveNext
        Loop
    End If
    Set getb = New ADODB.Recordset
    getb.Open "SELECT TOP (100) PERCENT dbo.Tabela_Boletins_Parametros_Incubacao.IDIncubacao, dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, " & _
```

```
    "                    dbo.Tabela_Boletins_Parametros_Incubacao.LoteMeioCultura,
dbo.Tabela_Boletins.IDBoletim AS Expr1, dbo.Tabela_Boletins.CodigoFinal " & _
    " FROM      dbo.Tabela_Boletins_Parametros_Incubacao INNER JOIN " & _
    " dbo.Tabela_Boletins ON dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim =
dbo.Tabela_Boletins.IDBoletim " & _
    " where lotemeiocultura = '" & producao.Fields("lote").Value & "' ORDER BY
dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, dbo.Tabela_Boletins.CodigoFinal"
& _
    " ", LabDB, adOpenStatic
    If getb.RecordCount <> 0 Then
      getb.MoveFirst
      Do While Not getb.EOF
        Set nodX = Tree.Nodes.Add("R6B" & producao.Fields("pproducao").Value &
producao.Bookmark, tvwChild, "R6B2" & getb.Fields("idincubacao").Value &
getb.Bookmark & Rnd(0.123456789123457), "Certificado de Análise " &
getb.Fields("codigofinal").Value)
        nodX.Image = 14
        getb.MoveNext
      Loop
    End If
    producao.MoveNext
  Loop
erro:
  ErrosGerais
End Sub

Private Sub Command19_Click()
  Unload Me
End Sub

Private Sub Command20_Click()
  Unload Me
End Sub
```

```
Private Sub Command3_Click()
   Set VerPerm = New ADODB.Recordset
   VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador & "", LabDB, adOpenStatic
   If VerPerm.Fields("Check52").Value = 1 Then
      frmConsultaProducao.Enabled = False
      frmNovaProducaoMeioCultura.Show
   Else
      MsgBox "You do not have permissions to access the requested. Please contact System Administrator!", vbInformation, App.Title
      Exit Sub
   End If
End Sub

Private Sub Command2_Click()
   Unload Me
End Sub

Private Sub Command4_Click()
On Error GoTo erro
   Set VerPerm = New ADODB.Recordset
   VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador & "", LabDB, adOpenStatic
   If VerPerm.Fields("Check53").Value = 1 Then
      If TabelaA.TextMatrix(TabelaA.RowSel, 2) = "" Then
         Exit Sub
      End If
      Set anula = New ADODB.Recordset
      anula.Open "update tabela_Meioscultura_Producao set confirmado = 2, qdeproduzida=0 where pproducao = " & TabelaA.TextMatrix(TabelaA.RowSel, 2) & "", LabDB, adOpenStatic
      MsgBox "Pedido de Produção de Meio de Cultura Anulado! Não foram movimentados reagentes!", vbInformation, App.Title
      Form_Load
```

```
   Else
      MsgBox "You do not have permissions to access the requested. Please contact System
Administrator!", vbInformation, App.Title
      Exit Sub
   End If
erro:
   ErrosGerais
End Sub

Private Sub Command5_Click()
On Error GoTo erro
   Tabela3.Rows = 1
   Set getstock = New ADODB.Recordset
   getstock.Open "EXEC GET_MeiosCultura_Stocks", LabDB, adOpenStatic
   If getstock.RecordCount = 0 Then
      Exit Sub
   End If
   getstock.MoveFirst
   linha = 1
   Tabela3.Rows = getstock.RecordCount + 1
   Do While Not getstock.EOF
      Tabela3.TextMatrix(linha, 1) = getstock.Fields("referencia").Value
      Tabela3.TextMatrix(linha, 2) = getstock.Fields("descricao").Value
      Tabela3.TextMatrix(linha, 3) = getstock.Fields("lote").Value
      Tabela3.TextMatrix(linha, 10) = getstock.Fields("lote").Value
      If getstock.Fields("dataproducao").ActualSize <> 0 Then
         Tabela3.TextMatrix(linha, 4) = Format(getstock.Fields("dataproducao").Value,
"dd/mm/yyyy")
      Else
         Tabela3.TextMatrix(linha, 4) = Format(getstock.Fields("data").Value,
"dd/mm/yyyy")
      End If
      Tabela3.TextMatrix(linha, 5) = getstock.Fields("qdeproduzida").Value
      Tabela3.TextMatrix(linha, 6) = getstock.Fields("stockpp").Value
```

```
        Tabela3.TextMatrix(linha, 7) = Format(getstock.Fields("datavalidade").Value,
"dd/mm/yyyy")
        If getstock.Fields("datavalidade").Value < Date Then
          FValidade.Rows = FValidade.Rows + 1
          FValidade.TextMatrix(FValidade.Rows - 1, 1) = getstock.Fields("referencia").Value
          FValidade.TextMatrix(FValidade.Rows - 1, 2) = getstock.Fields("descricao").Value
          FValidade.TextMatrix(FValidade.Rows - 1, 3) = getstock.Fields("lote").Value
          If getstock.Fields("dataproducao").ActualSize <> 0 Then
            FValidade.TextMatrix(FValidade.Rows - 1, 4) =
Format(getstock.Fields("dataproducao").Value, "dd/mm/yyyy")
          Else
            FValidade.TextMatrix(FValidade.Rows - 1, 4) =
Format(getstock.Fields("data").Value, "dd/mm/yyyy")
          End If
          FValidade.TextMatrix(FValidade.Rows - 1, 5) =
getstock.Fields("qdeproduzida").Value
          FValidade.TextMatrix(FValidade.Rows - 1, 6) = getstock.Fields("stockpp").Value
          FValidade.TextMatrix(FValidade.Rows - 1, 7) =
Format(getstock.Fields("datavalidade").Value, "dd/mm/yyyy")
          FValidade.TextMatrix(FValidade.Rows - 1, 8) = getstock.Fields("usercpc").Value
        End If
        Tabela3.TextMatrix(linha, 8) = getstock.Fields("usercpc").Value
        linha = linha + 1
        getstock.MoveNext
    Loop
    m_Fmt = "(#,##0.00)"
    Dim r%, c%
    r = Tabela3.Row
    c = Tabela3.Col
    Tabela3.Col = 0
    Tabela3.ColSel = 2
    Tabela3.Subtotal flexSTClear
    A = Tabela3.ColSel
    Tabela3.Subtotal flexSTSum, 1, 5, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True
```

```
    Tabela3.Subtotal flexSTSum, 1, 6, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True
    Tabela3.Row = r
    Tabela3.Col = c
    Tabela3.OutlineBar = flexOutlineBarComplete
    Tabela3.AllowUserResizing = flexResizeColumns
    Tabela3.MergeCol(1) = True
    Tabela3.MergeCol(2) = True
    Tabela3.MergeCol(3) = True
    Tabela3.MergeCol(0) = True
    Tabela3.MergeCells = flexMergeFree
    Tabela3.Outline 2
erro:
    ErrosGerais
End Sub

Private Sub Command6_Click()
    Unload Me
End Sub

Private Sub Command7_Click()
On Error GoTo erro
    Set VerPerm = New ADODB.Recordset
    VerPerm.Open "select * from tabela_permissoes where IDutilizador = " & IDUtilizador
& "", LabDB, adOpenStatic
    If VerPerm.Fields("Check54").Value = 1 Then
       If MsgBox("O Stock dos Lotes de Meios de Cultura selecionados serão considerados
Zero. Confirma ?", vbYesNo + vbQuestion, App.Title) = vbYes Then
          If Tabela3.RowSel > 1 Then
             For i = 1 To Tabela3.Rows - 1
                If Tabela3.TextMatrix(i, 9) <> "" Then
                   If Tabela3.ValueMatrix(i, 9) <> 0 Then
                      Set elimina = New ADODB.Recordset
                      elimina.Open "update tabela_meioscultura_producao set Stockpp = 0
where lote = '" & Tabela3.TextMatrix(i, 3) & "'", LabDB, adOpenStatic
```

```
                Set elimina = New ADODB.Recordset
                elimina.Open "insert into tabela_meioscultura_movimentos (IDMeio, TipoMovimento, quantidade, lote, " & _
                " DataSistemaEntrada, HoraSistemaEntrada, UserWindows, UserCPC, Computador) " & _
                " values ('" & Tabela3.TextMatrix(Tabela3.RowSel, 1) & "', " & _
                " 'A', '" & ConverteDecimal(Tabela3.TextMatrix(i, 6) * (-1)) & "', " & _
                " '" & Tabela3.TextMatrix(i, 3) & "', " & _
                " '" & Format(Date, "yyyy/mm/dd") & "', '" & Format(Date, "yyyy/mm/dd") & "', " & _
                " '" & utilizador & "', '" & UserCPC & "', '" & computador & "')", LabDB, adOpenStatic
                End If
            End If
        Next i
      End If
      Form_Load
    Else
      Cancel = True
    End If
  Else
    MsgBox "You do not have permissions to access the requested. Please contact System Administrator!", vbInformation, App.Title
    Exit Sub
  End If
erro:
  ErrosGerais
End Sub

Private Sub Command8_Click()
  Unload Me
End Sub

Private Sub DTP1_Change()
```

```
    fl_Click_Tab = False
    If Weekday(DTP1.Value) = 1 Then
        SSTab2.Tab = 6
    Else
        SSTab2.Tab = Weekday(DTP1.Value) - 2
    End If
    fl_Click_Tab = True
    CalculaIniFimSemana (DTP1.Value)
    Limpa
    carregahistorico
End Sub

Private Sub Form_Activate()
    If origem = 0 Then
        Exit Sub
    ElseIf origem = 1 Then
        lerCQ
    ElseIf origem = 2 Then
        CarregaPP
    ElseIf origem = 3 Then
        CarregaPP
    Else
        Form_Load
    End If
End Sub

Private Sub Form_Load()
On Error GoTo erro
    Me.Top = 10
    Me.Left = 1
    leumeios = 0
    leulotes = 0
    leuguias = 0
    Combo3.Clear
```

```
    Combo1.Clear
    Combo2.Clear
    Set Tree.ImageList = ImageList2
    Tree.Nodes.Clear
    stocksemiactual = 0
    TabelaA.Rows = 1
    Tabela3.Rows = 1
    TabelaCQ.Rows = 1
    Tabela3.Editable = flexEDKbdMouse
    FValidade.Rows = 1
    DTP1.Value = Date
    CalculaIniFimSemana (Date)
    If Weekday(DTP1.Value) = 1 Then
       SSTab2.Tab = 6
    Else
       SSTab2.Tab = Weekday(DTP1.Value) - 2
    End If
    CarregaPP
    Limpa
    carregahistorico
    Movimento.Rows = 1
    Set reagentes1 = New ADODB.Recordset
    reagentes1.Open " Select * from tabela_meioscultura where activo = 1 order by
referencia", LabDB, adOpenStatic
    If reagentes1.RecordCount = 0 Then
       Exit Sub
    End If
    reagentes1.MoveFirst
    Do While Not reagentes1.EOF
       cMP.AddItem reagentes1.Fields("referencia").Value & " - " &
reagentes1.Fields("descricao").Value
       reagentes1.MoveNext
    Loop
erro:
```

```
    ErrosGerais
End Sub


Private Sub carregahistorico()
    Data1 = datainisem
    Data2 = datafimsem
    Set pp = New ADODB.Recordset
    pp.Open "EXEC Get_Meioscultura_Producao_historico @confirmado = 0, @data1 = ''' & Format(Data1, "yyyy/mm/dd") & ''', @data2 = ''' & Format(Data2, "yyyy/mm/dd") & """, LabDB, adOpenStatic
    linha = 1
    If pp.RecordCount = 0 Then
        Exit Sub
    End If
    pp.MoveFirst
    Do While Not pp.EOF
        If pp.Fields("dataproducao").ActualSize <> 0 Then
            coloca = pp.Fields("dataproducao").Value
        Else
            coloca = pp.Fields("data").Value
        End If
        If Weekday(coloca) = 1 Then
            linha = 6
        Else
            linha = Weekday(coloca) - 2
        End If
        Tabela(linha).Rows = Tabela(linha).Rows + 1
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 2) = pp.Fields("PProducao").Value
        If pp.Fields("dataproducao").ActualSize <> 0 Then
            Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 3) = pp.Fields("dataproducao").Value
        Else
            Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 3) = pp.Fields("data").Value
        End If
```

```
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 1) = pp.Fields("Lote").Value
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 4) = pp.Fields("idmeio").Value
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 5) = pp.Fields("descricao").Value
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 7) =
pp.Fields("qdeproduzida").Value
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 8) =
pp.Fields("DataValidade").Value
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 9) = pp.Fields("usercpc").Value
        Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 10) =
pp.Fields("confirmado").Value
        If Tabela(linha).TextMatrix(Tabela(linha).Rows - 1, 10) = 2 Then
           Tabela(linha).Row = Tabela(linha).Rows - 1
           For i = 1 To Tabela(linha).Cols - 1
              Tabela(linha).Col = i
              Tabela(linha).CellBackColor = &HFFC0C0
           Next i
        End If
        pp.MoveNext
    Loop
    For i = 0 To 6
        Ordertabela (i)
    Next i
End Sub

Private Sub CarregaPP()
On Error GoTo erro
    TabelaA.Rows = 1
    Set pp = New ADODB.Recordset
    pp.Open "EXEC Get_Meioscultura_Producao @confirmado = 0", LabDB, adOpenStatic
    linha = 1
    If pp.RecordCount = 0 Then
        Exit Sub
    End If
    pp.MoveFirst
```

```
    Do While Not pp.EOF
        TabelaA.Rows = TabelaA.Rows + 1
        TabelaA.TextMatrix(linha, 2) = pp.Fields("PProducao").Value
        TabelaA.TextMatrix(linha, 3) = pp.Fields("data").Value
        TabelaA.TextMatrix(linha, 1) = pp.Fields("Lote").Value
        TabelaA.TextMatrix(linha, 4) = pp.Fields("idmeio").Value
        TabelaA.TextMatrix(linha, 5) = pp.Fields("descricao").Value
        TabelaA.TextMatrix(linha, 6) = pp.Fields("qdeprevista").Value
        TabelaA.TextMatrix(linha, 7) = pp.Fields("DataValidade").Value
        linha = linha + 1
        pp.MoveNext
    Loop
    Ordertabelaa
erro:
    ErrosGerais
End Sub

Private Sub Ordertabelaa()
    m_Fmt = "(#,##0.00)"
    Dim r%, c%
    r = TabelaA.Row
    c = TabelaA.Col
    TabelaA.Col = 0
    TabelaA.ColSel = 2
    TabelaA.Subtotal flexSTClear
    A = TabelaA.ColSel
    TabelaA.Subtotal flexSTNone, -2, 3, m_Fmt, &HC0C0FF, RGB(255, 0, 0), True, "Lotes"
    TabelaA.Subtotal flexSTNone, 1, 5, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True, "%s"
    TabelaA.Row = r
    TabelaA.Col = c
    TabelaA.OutlineBar = flexOutlineBarComplete
    TabelaA.AllowUserResizing = flexResizeColumns
    TabelaA.MergeCol(1) = True
    TabelaA.MergeCol(2) = True
```

```
    TabelaA.MergeCol(3) = True
    TabelaA.MergeCol(4) = True
    TabelaA.MergeCol(5) = True
    TabelaA.MergeCol(0) = True
    TabelaA.MergeCol(10) = True
    TabelaA.MergeCells = flexMergeFree
    TabelaA.Outline 2
    For i = 1 To TabelaA.Rows - 1
       TabelaA.Row = i
       TabelaA.Col = 5
       If TabelaA.TextMatrix(i, TabelaA.Col) <> "" Then
          TabelaA.CellBackColor = &HC0E0FF
       End If
       TabelaA.Col = 7
       If TabelaA.TextMatrix(i, TabelaA.Col) <> "" Then
          TabelaA.CellBackColor = &HC0E0FF
       End If
       TabelaA.Col = 9
       If TabelaA.TextMatrix(i, TabelaA.Col) <> "" Then
          TabelaA.CellBackColor = &HC0E0FF
       End If
    Next i
End Sub

Private Sub Ordertabela(Index As Integer)
    m_Fmt = "(#,##0.00)"
    Dim r%, c%
    r = Tabela(i).Row
    c = Tabela(i).Col
    Tabela(i).Col = 0
    Tabela(i).ColSel = 2
    Tabela(i).Subtotal flexSTClear
    A = Tabela(i).ColSel
```

```
    Tabela(i).Subtotal flexSTNone, -2, 3, m_Fmt, &HC0C0FF, RGB(255, 0, 0), True, "Lotes"
    Tabela(i).Subtotal flexSTNone, 1, 5, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True, "%s"
    Tabela(i).Row = r
    Tabela(i).Col = c
    Tabela(i).OutlineBar = flexOutlineBarComplete
    Tabela(i).AllowUserResizing = flexResizeColumns
    Tabela(i).MergeCol(1) = True
    Tabela(i).MergeCol(2) = True
    Tabela(i).MergeCol(3) = True
    Tabela(i).MergeCol(4) = True
    Tabela(i).MergeCol(5) = True
    Tabela(i).MergeCol(0) = True
    Tabela(i).MergeCol(10) = True
    Tabela(i).MergeCells = flexMergeFree
    Tabela(i).Outline 2
    For L = 1 To Tabela(i).Rows - 1
        Tabela(i).Row = L
        Tabela(i).Col = 5
        If Tabela(i).TextMatrix(L, Tabela(i).Col) <> "" Then
            Tabela(i).CellBackColor = &HC0E0FF
        End If
        Tabela(i).Col = 7
        If Tabela(i).TextMatrix(L, Tabela(i).Col) <> "" Then
            Tabela(i).CellBackColor = &HC0E0FF
        End If
        Tabela(i).Col = 9
        If Tabela(i).TextMatrix(L, Tabela(i).Col) <> "" Then
            Tabela(i).CellBackColor = &HC0E0FF
        End If
    Next L
End Sub
```

```
Private Sub Tabela_dbClick(Index As Integer)
   If Tabela(Index).RowSel > 1 Then
      If Tabela(Index).TextMatrix(Tabela(Index).RowSel, 2) <> "" Then
         frmNovaProducaoMeioCulturaConfirma.pedido                    =
Tabela(Index).TextMatrix(Tabela(Index).RowSel, 2)
         frmNovaProducaoMeioCulturaConfirma.origem = 2
         frmNovaProducaoMeioCulturaConfirma.Show
      End If
   Else
      Exit Sub
   End If
End Sub

Private Sub SSTab1_Click(PreviousTab As Integer)
   If SSTab1.Tab = 5 Then
      lerCQ
   End If
End Sub

Private Sub Tabela_DblClick(Index As Integer)
   If Tabela(Index).RowSel > 1 Then
      If Tabela(Index).TextMatrix(Tabela(Index).RowSel, 2) <> "" Then
         frmNovaProducaoMeioCulturaConfirma.pedido                    =
Tabela(Index).TextMatrix(Tabela(Index).RowSel, 2)
         frmNovaProducaoMeioCulturaConfirma.origem = 2
         frmNovaProducaoMeioCulturaConfirma.Show
      End If
   Else
      Exit Sub
   End If
End Sub

Private Sub tabelaa_DblClick()
   If TabelaA.RowSel > 1 Then
```

```
        If TabelaA.TextMatrix(TabelaA.RowSel, 2) <> "" Then
            frmNovaProducaoMeioCulturaConfirma.pedido                    =
TabelaA.TextMatrix(TabelaA.RowSel, 2)
            frmNovaProducaoMeioCulturaConfirma.origem = 1
            frmNovaProducaoMeioCulturaConfirma.Show
        End If
    Else
        Exit Sub
    End If
End Sub

Function Limpa()
    For i = 0 To 6
        Tabela(i).Rows = 1
    Next i
End Function

Function CalculaIniFimSemana(mData As Date)
    Select Case Weekday(mData)
        Case 1
            datainisem = CStr(mData - 6)
            datafimsem = CStr(mData)
        Case Else
            datainisem = CStr(mData - (Weekday(mData) - 2))
            datafimsem = CStr(mData + (8 - Weekday(mData)))
    End Select
End Function

Private Sub LerMovimentos()
On Error GoTo erro
    Movimento.Rows = 1
    Set lertodos = New ADODB.Recordset
    If lertodosv = 1 Then
        lertodos.Open "EXEC Get_MeiosCultura_Movimentos", LabDB, adOpenStatic
```

```
   ElseIf lertodosv = 0 Then
      lertodos.Open "EXEC Get_MeiosCultura_Movimentos_Filtro @IDMeio = '" &
IDReagente & "'", LabDB, adOpenStatic
   End If
   If lertodos.RecordCount = 0 Then
      Exit Sub
   End If
   linha = 1
   Movimento.Rows = lertodos.RecordCount + 1
   lertodos.MoveFirst
   Do While Not lertodos.EOF
      Movimento.TextMatrix(linha, 1) = lertodos.Fields("referencia").Value
      Movimento.TextMatrix(linha, 2) = lertodos.Fields("descricao").Value
      Movimento.TextMatrix(linha, 4) = lertodos.Fields("lote").Value
      If lertodos.Fields("tipoMovimento").Value = "P" Then
         Movimento.TextMatrix(linha, 5) = "Produção"
      Else
         Movimento.TextMatrix(linha, 5) = "Acerto"
      End If
      Movimento.TextMatrix(linha, 7) = lertodos.Fields("data").Value
      If lertodos.Fields("quantidade").ActualSize <> 0 Then
         Movimento.TextMatrix(linha, 8) = lertodos.Fields("quantidade").Value
      End If
      lertodos.MoveNext
      linha = linha + 1
   Loop
   m_Fmt = "(#)"
   Dim r%, c%
   r = Movimento.Row
   c = Movimento.Col
   Movimento.Col = 0
   Movimento.ColSel = 2
   Movimento.Subtotal flexSTClear
```

```
    A = Movimento.ColSel
    'Movimento.Subtotal flexSTSum, 0, 7, m_Fmt, &HC0E0FF, RGB(0, 0, 255), True,
"Stock Total"
    Movimento.Subtotal flexSTSum, 1, 8, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True
    Movimento.Row = r
    Movimento.Col = c
    Movimento.OutlineBar = flexOutlineBarComplete
    Movimento.AllowUserResizing = flexResizeColumns
    Movimento.MergeCol(1) = True
    Movimento.MergeCol(2) = True
    Movimento.MergeCells = flexMergeFree
    Movimento.MergeCells = flexMergeFree
    Movimento.Outline 2
    Movimento.Outline 1
erro:
    ErrosGerais
End Sub

Private Sub lerCQ()
On Error GoTo erro
    TabelaCQ.Rows = 1
    Set ppCQ = New ADODB.Recordset
    ppCQ.Open "EXEC Get_Meioscultura_ControloQualidade", LabDB, adOpenStatic
    linha = 1
    If ppCQ.RecordCount = 0 Then
        Exit Sub
    End If
    ppCQ.MoveFirst
    Do While Not ppCQ.EOF
        TabelaCQ.Rows = TabelaCQ.Rows + 1
        TabelaCQ.TextMatrix(linha, 2) = ppCQ.Fields("PProducao").Value
        If ppCQ.Fields("dataproducao").ActualSize <> 0 Then
            TabelaCQ.TextMatrix(linha, 3) = ppCQ.Fields("dataproducao").Value
        Else
```

```
            TabelaCQ.TextMatrix(linha, 3) = ppCQ.Fields("data").Value
        End If
        TabelaCQ.TextMatrix(linha, 1) = ppCQ.Fields("Lote").Value
        TabelaCQ.TextMatrix(linha, 4) = ppCQ.Fields("idmeio").Value
        TabelaCQ.TextMatrix(linha, 5) = ppCQ.Fields("descricao").Value
        TabelaCQ.TextMatrix(linha, 6) = ppCQ.Fields("qdeprevista").Value
        TabelaCQ.TextMatrix(linha, 7) = ppCQ.Fields("DataValidade").Value
        linha = linha + 1
        ppCQ.MoveNext
    Loop
    OrdertabelaCQ
erro:
    ErrosGerais
End Sub

Private Sub OrdertabelaCQ()
    m_Fmt = "(#,##0.00)"
    Dim r%, c%
    r = TabelaCQ.Row
    c = TabelaCQ.Col
    TabelaCQ.Col = 0
    TabelaCQ.ColSel = 2
    TabelaCQ.Subtotal flexSTClear
    A = TabelaCQ.ColSel
    TabelaCQ.Subtotal flexSTNone, -2, 3, m_Fmt, &HC0C0FF, RGB(255, 0, 0), True,
"Lotes"
    TabelaCQ.Subtotal flexSTNone, 1, 5, m_Fmt, &HFFC0C0, RGB(0, 0, 255), True, "%s"
    TabelaCQ.Row = r
    TabelaCQ.Col = c
    TabelaCQ.OutlineBar = flexOutlineBarComplete
    TabelaCQ.AllowUserResizing = flexResizeColumns
    TabelaCQ.MergeCol(1) = True
    TabelaCQ.MergeCol(2) = True
    TabelaCQ.MergeCol(3) = True
```

```
    TabelaCQ.MergeCol(4) = True
    TabelaCQ.MergeCol(5) = True
    TabelaCQ.MergeCol(0) = True
    TabelaCQ.MergeCol(10) = True
    TabelaCQ.MergeCells = flexMergeFree
    TabelaCQ.Outline 2
    For i = 1 To TabelaCQ.Rows - 1
       TabelaCQ.Row = i
       TabelaCQ.Col = 5
       If TabelaCQ.TextMatrix(i, TabelaCQ.Col) <> "" Then
          TabelaCQ.CellBackColor = &HC0E0FF
       End If
       TabelaCQ.Col = 7
       If TabelaCQ.TextMatrix(i, TabelaCQ.Col) <> "" Then
          TabelaCQ.CellBackColor = &HC0E0FF
       End If
       TabelaCQ.Col = 9
       If TabelaCQ.TextMatrix(i, TabelaCQ.Col) <> "" Then
          TabelaCQ.CellBackColor = &HC0E0FF
       End If
    Next i
End Sub

Private Sub TabelaCQ_DblClick()
    If TabelaCQ.RowSel > 1 Then
       If TabelaCQ.TextMatrix(TabelaCQ.RowSel, 2) <> "" Then
          frmNovaProducaoMeioCulturaConfirma.pedido                    =
TabelaCQ.TextMatrix(TabelaCQ.RowSel, 2)
          frmNovaProducaoMeioCulturaConfirma.origem = 3
          frmNovaProducaoMeioCulturaConfirma.Show
       End If
    Else
       Exit Sub
    End If
```

```
End Sub

Private Sub LerFiltro()
On Error GoTo erro
   Combo1.Clear
   Combo3.Clear
   leuguias = 0
   leumeios = 0
   Tree.Nodes.Clear
   Set producao = New ADODB.Recordset
   producao.Open "SELECT TOP (100) PERCENT dbo.Tabela_MeiosCultura_Producao.PProducao, dbo.Tabela_MeiosCultura_Producao.Data, dbo.Tabela_MeiosCultura_Producao.DataProducao, " & _
   " dbo.Tabela_MeiosCultura_Producao.Lote, dbo.Tabela_MeiosCultura_Producao.IDMeio, dbo.Tabela_MeiosCultura.Descricao, " & _
   " dbo.Tabela_MeiosCultura_Producao.QdePrevista, dbo.Tabela_MeiosCultura_Producao.DataValidade, " & _
   " dbo.Tabela_MeiosCultura_Producao.Confirmado " & _
   " FROM dbo.Tabela_MeiosCultura_Producao INNER JOIN " & _
   " dbo.Tabela_MeiosCultura ON dbo.Tabela_MeiosCultura_Producao.IDMeio = dbo.Tabela_MeiosCultura.Referencia " & _
   " WHERE Confirmado = 1 AND lote = '" & Combo2.Text & "' " & _
   " ORDER BY dbo.Tabela_MeiosCultura_Producao.PProducao", LabDB, adOpenStatic
   If producao.RecordCount = 0 Then
      MsgBox "Dados não encontrados. Provavelmente a ordem de fabrico ainda não se encontra fechada!"
      Exit Sub
   End If
   producao.MoveFirst
   Do While Not producao.EOF
      Set nodX = Tree.Nodes.Add(, , "T" & producao.Bookmark, "Rastreabilidade da ordem de fabrico " & producao.Fields("lote").Value)
      nodX.Image = 8
      nodX.Bold = True
```

```
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6G" & producao.Fields("pproducao").Value & producao.Bookmark, "Dados Gerais")
nodX.Image = 13
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6R" & producao.Fields("pproducao").Value & producao.Fields("idmeio").Value & producao.Bookmark, "Reagentes (Rastreabilidade)")
nodX.Image = 13
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6C" & producao.Fields("pproducao").Value & producao.Bookmark, "Controlo da Qualidade")
nodX.Image = 13
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6B" & producao.Fields("pproducao").Value & producao.Bookmark, "Certificados de Análise associados")
nodX.Image = 13
Set getdadosproduto = New ADODB.Recordset
getdadosproduto.Open "select * from tabela_Meioscultura_producao where lote = '" & producao.Fields("lote").Value & "'", LabDB, adOpenStatic
If getdadosproduto.RecordCount <> 0 Then
   Set getdadosproduto2 = New ADODB.Recordset
   getdadosproduto2.Open "EXEC GET_MeiosCultura_Producao_Confirma @pp = '" & getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
   If getdadosproduto2.RecordCount <> 0 Then
      Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6C" & getdadosproduto2.Bookmark & producao.Bookmark & getdadosproduto2.Fields("pproducao").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Produzido em " & getdadosproduto2.Fields("data").Value & " com validade até " & getdadosproduto2.Fields("datavalidade").Value)
      nodX.Image = 14
      Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6C1" & getdadosproduto2.Bookmark & producao.Bookmark & getdadosproduto2.Fields("pproducao").Value & producao.Fields("pproducao").Value, "Quantidade produzida: " & getdadosproduto2.Fields("qdeprevista").Value)
```

```
            nodX.Image = 14
          End If
        End If
        Set rastre = New ADODB.Recordset
        rastre.Open "EXEC GET_MeiosCultura_Producao_Confirma_2 @pp = '" &
getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
        If rastre.RecordCount <> 0 Then
          rastre.MoveFirst
          Do While Not rastre.EOF
            If rastre.Fields("tipo").Value = 1 Then
              unidade = "Litros"
            Else
              unidade = "Grama"
            End If
            If externo = 1 Then
              Set getdados = New ADODB.Recordset
              getdados.Open "SELECT dbo.Item.ItemID, dbo.Item.BarCode,
dbo.Item.familyid, dbo.ItemNames.ShortDescription, dbo.ItemNames.Description " & _
              " FROM dbo.Item INNER JOIN " & _
              " dbo.ItemNames ON dbo.Item.ItemID = dbo.ItemNames.ItemID " & _
              " where dbo.item.itemid = '" & rastre.Fields("idreagente").Value & "'", GesPos,
adOpenStatic
            Else
              getdados.Open "SELECT * from tabela_reagentes where dbo.item.itemid = '"
& rastre.Fields("idreagente").Value & "'", LabDB, adOpenStatic
            End If
            If getdados.RecordCount <> 0 Then
              Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value &
producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" &
getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value
& producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: " &
getdados.Fields("description").Value & " (" & rastre.Fields("idreagente").Value & ") - Guia
de Entrada: " & rastre.Fields("GE").Value & ". Quantidade consumida: " &
rastre.Fields("Quantidadereal").Value & " " & unidade)
```

```
            nodX.Image = 14
        Else
            Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value &
producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" &
getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value
& producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: não detectado na
base de dados GesPOS (" & rastre.Fields("idreagente").Value & ") - Guia de Entrada: " &
rastre.Fields("GE").Value & ". Quantidade consumida: " &
rastre.Fields("Quantidadereal").Value & " " & unidade)
            nodX.Image = 14
        End If
        rastre.MoveNext
      Loop
    End If
    Set getplano = New ADODB.Recordset
    getplano.Open "SELECT TOP (100) PERCENT
dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo,
dbo.Tabela_ControloQualidade_PlanoFinal.IDControloPlano,
dbo.Tabela_ControloQualidade_PlanoFinal.Produto,
dbo.Tabela_ControloQualidade_PlanoFinal.resultado,
dbo.Tabela_ControloQualidade_PlanoFinal.resultadonumerico, " & _
    " dbo.Tabela_ControloQualidade_PlanoFinal.Lote,
dbo.Tabela_ControloQualidade_PlanoFinal.Efectuado,
dbo.Tabela_ControloQualidade_Controlos.Descricao, " & _
    " dbo.Tabela_ControloQualidade_Controlos.Valor ,
dbo.Tabela_ControloQualidade_Controlos.vmin,
dbo.Tabela_ControloQualidade_Controlos.vmax,
dbo.Tabela_ControloQualidade_planofinal.idcontroloplano,
dbo.Tabela_ControloQualidade_planofinal.idcontrolo " & _
    " FROM dbo.Tabela_ControloQualidade_PlanoFinal INNER JOIN " & _
    " dbo.Tabela_ControloQualidade_Controlos ON
dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo =
dbo.Tabela_ControloQualidade_Controlos.IDControlo " & _
```

```
" WHERE (dbo.Tabela_ControloQualidade_PlanoFinal.Lote = '" & getdadosproduto.Fields("lote").Value & "') " & _
" ORDER BY dbo.Tabela_ControloQualidade_Controlos.Descricao", LabDB, adOpenStatic
If getplano.RecordCount <> 0 Then
  getplano.MoveFirst
  Do While Not getplano.EOF
    Set nodX = Tree.Nodes.Add("R6C" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Controlo Qualitativo " & getplano.Fields("descricao").Value)
    nodX.Image = 14
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R3" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor mínimo especificado: " & getplano.Fields("vmin").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R4" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor máximo especificado: " & getplano.Fields("vmax").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R5" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Resultado numérico obtido: " & getplano.Fields("resultadonumerico").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R6" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value &
```

```
producao.Fields("pproducao").Value, "Resultado alfanumérico obtido: " &
getplano.Fields("resultado").Value)
            nodX.Image = 12
            getplano.MoveNext
         Loop
      End If
      Set getb = New ADODB.Recordset
      getb.Open "SELECT TOP (100) PERCENT
dbo.Tabela_Boletins_Parametros_Incubacao.IDIncubacao,
dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, " & _
      " dbo.Tabela_Boletins_Parametros_Incubacao.LoteMeioCultura,
dbo.Tabela_Boletins.IDBoletim AS Expr1, dbo.Tabela_Boletins.CodigoFinal " & _
      " FROM dbo.Tabela_Boletins_Parametros_Incubacao INNER JOIN " & _
      " dbo.Tabela_Boletins ON dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim =
dbo.Tabela_Boletins.IDBoletim " & _
      " where lotemeiocultura = '" & producao.Fields("lote").Value & "' ORDER BY
dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, dbo.Tabela_Boletins.CodigoFinal"
& _
      " ", LabDB, adOpenStatic
      If getb.RecordCount <> 0 Then
         getb.MoveFirst
         Do While Not getb.EOF
            Set nodX = Tree.Nodes.Add("R6B" & producao.Fields("pproducao").Value &
producao.Bookmark, tvwChild, "R6B2" & getb.Fields("idincubacao").Value &
getb.Bookmark & Rnd(0.123456789123457), "Certificado de Análise " &
getb.Fields("codigofinal").Value)
            nodX.Image = 14
            getb.MoveNext
         Loop
      End If
      producao.MoveNext
   Loop
erro:
   ErrosGerais
```

```
End Sub

Private Sub LerFiltro2()
On Error GoTo erro
   Combo2.Clear
   Combo3.Clear
   leuguias = 0
   leulotes = 0
   Tree.Nodes.Clear
   Set producao = New ADODB.Recordset
   producao.Open "SELECT TOP (100) PERCENT dbo.Tabela_MeiosCultura_Producao.PProducao, dbo.Tabela_MeiosCultura_Producao.Data, dbo.Tabela_MeiosCultura_Producao.DataProducao, " & _
   " dbo.Tabela_MeiosCultura_Producao.Lote, dbo.Tabela_MeiosCultura_Producao.IDMeio, dbo.Tabela_MeiosCultura.Descricao, " & _
   " dbo.Tabela_MeiosCultura_Producao.QdePrevista, dbo.Tabela_MeiosCultura_Producao.DataValidade, " & _
   " dbo.Tabela_MeiosCultura_Producao.Confirmado " & _
   " FROM dbo.Tabela_MeiosCultura_Producao INNER JOIN " & _
   " dbo.Tabela_MeiosCultura ON dbo.Tabela_MeiosCultura_Producao.IDMeio = dbo.Tabela_MeiosCultura.Referencia " & _
   " WHERE Confirmado = 1 AND idmeio = '" & idmeio & "' " & _
   " ORDER BY dbo.Tabela_MeiosCultura_Producao.PProducao", LabDB, adOpenStatic
   If producao.RecordCount = 0 Then
      MsgBox "Dados não encontrados. Provavelmente a ordem de fabrico ainda não se encontra fechada!"
      Exit Sub
   End If
   producao.MoveFirst
   Do While Not producao.EOF
      Set nodX = Tree.Nodes.Add(, , "T" & producao.Bookmark, "Rastreabilidade da ordem de fabrico " & producao.Fields("lote").Value)
      nodX.Image = 8
      nodX.Bold = True
```

```
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6G" & producao.Fields("pproducao").Value & producao.Bookmark, "Dados Gerais")
nodX.Image = 13
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6R" & producao.Fields("pproducao").Value & producao.Fields("idmeio").Value & producao.Bookmark, "Reagentes (Rastreabilidade)")
nodX.Image = 13
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6C" & producao.Fields("pproducao").Value & producao.Bookmark, "Controlo da Qualidade")
nodX.Image = 13
Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6B" & producao.Fields("pproducao").Value & producao.Bookmark, "Certificados de Análise associados")
nodX.Image = 13
Set getdadosproduto = New ADODB.Recordset
getdadosproduto.Open "select * from tabela_Meioscultura_producao where lote = '" & producao.Fields("lote").Value & "'", LabDB, adOpenStatic
If getdadosproduto.RecordCount <> 0 Then
   Set getdadosproduto2 = New ADODB.Recordset
   getdadosproduto2.Open "EXEC GET_MeiosCultura_Producao_Confirma @pp = '" & getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
   If getdadosproduto2.RecordCount <> 0 Then
      Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6C" & getdadosproduto2.Bookmark & producao.Bookmark & getdadosproduto2.Fields("pproducao").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Produzido em " & getdadosproduto2.Fields("data").Value & " com validade até " & getdadosproduto2.Fields("datavalidade").Value)
      nodX.Image = 14
      Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6C1" & getdadosproduto2.Bookmark & producao.Bookmark & getdadosproduto2.Fields("pproducao").Value & producao.Fields("pproducao").Value, "Quantidade produzida: " & getdadosproduto2.Fields("qdeprevista").Value)
```

```
            nodX.Image = 14
          End If
        End If
        Set rastre = New ADODB.Recordset
        rastre.Open "EXEC GET_MeiosCultura_Producao_Confirma_2 @pp = '" &
getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
        If rastre.RecordCount <> 0 Then
          rastre.MoveFirst
          Do While Not rastre.EOF
            If rastre.Fields("tipo").Value = 1 Then
              unidade = "Litros"
            Else
              unidade = "Grama"
            End If
            If externo = 1 Then
              Set getdados = New ADODB.Recordset
              getdados.Open "SELECT dbo.Item.ItemID, dbo.Item.BarCode,
dbo.Item.familyid, dbo.ItemNames.ShortDescription, dbo.ItemNames.Description " & _
              " FROM dbo.Item INNER JOIN " & _
              " dbo.ItemNames ON dbo.Item.ItemID = dbo.ItemNames.ItemID " & _
              " where dbo.item.itemid = '" & rastre.Fields("idreagente").Value & "'", GesPos,
adOpenStatic
            Else
              getdados.Open "SELECT * from tabela_reagentes where dbo.item.itemid = '"
& rastre.Fields("idreagente").Value & "'", LabDB, adOpenStatic
            End If
            If getdados.RecordCount <> 0 Then
              Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value &
producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" &
getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value
& producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: " &
getdados.Fields("description").Value & " (" & rastre.Fields("idreagente").Value & ") - Guia
de Entrada: " & rastre.Fields("GE").Value & ". Quantidade consumida: " &
rastre.Fields("Quantidadereal").Value & " " & unidade)
```

```
            nodX.Image = 14
        Else
            Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value &
producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" &
getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value
& producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: não detectado na
base de dados GesPOS (" & rastre.Fields("idreagente").Value & ") - Guia de Entrada: " &
rastre.Fields("GE").Value & ". Quantidade consumida: " &
rastre.Fields("Quantidadereal").Value & " " & unidade)
            nodX.Image = 14
        End If
        rastre.MoveNext
      Loop
    End If
    Set getplano = New ADODB.Recordset
    getplano.Open "SELECT TOP (100) PERCENT
dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo,
dbo.Tabela_ControloQualidade_PlanoFinal.IDControloPlano,
dbo.Tabela_ControloQualidade_PlanoFinal.Produto,
dbo.Tabela_ControloQualidade_PlanoFinal.resultado,
dbo.Tabela_ControloQualidade_PlanoFinal.resultadonumerico, " & _
    " dbo.Tabela_ControloQualidade_PlanoFinal.Lote,
dbo.Tabela_ControloQualidade_PlanoFinal.Efectuado,
dbo.Tabela_ControloQualidade_Controlos.Descricao, " & _
    " dbo.Tabela_ControloQualidade_Controlos.Valor ,
dbo.Tabela_ControloQualidade_Controlos.vmin,
dbo.Tabela_ControloQualidade_Controlos.vmax,
dbo.Tabela_ControloQualidade_planofinal.idcontroloplano,
dbo.Tabela_ControloQualidade_planofinal.idcontrolo " & _
    " FROM dbo.Tabela_ControloQualidade_PlanoFinal INNER JOIN " & _
    " dbo.Tabela_ControloQualidade_Controlos ON
dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo =
dbo.Tabela_ControloQualidade_Controlos.IDControlo " & _
```

```
" WHERE (dbo.Tabela_ControloQualidade_PlanoFinal.Lote = '" & getdadosproduto.Fields("lote").Value & "') " & _
" ORDER BY dbo.Tabela_ControloQualidade_Controlos.Descricao", LabDB, adOpenStatic
If getplano.RecordCount <> 0 Then
  getplano.MoveFirst
  Do While Not getplano.EOF
    Set nodX = Tree.Nodes.Add("R6C" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Controlo Qualitativo " & getplano.Fields("descricao").Value)
    nodX.Image = 14
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R3" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor mínimo especificado: " & getplano.Fields("vmin").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R4" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor máximo especificado: " & getplano.Fields("vmax").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R5" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Resultado numérico obtido: " & getplano.Fields("resultadonumerico").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R6" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value &
```

```
producao.Fields("pproducao").Value, "Resultado alfanumérico obtido: " &
getplano.Fields("resultado").Value)
            nodX.Image = 12
            getplano.MoveNext
         Loop
      End If
      Set getb = New ADODB.Recordset
      getb.Open "SELECT TOP (100) PERCENT
dbo.Tabela_Boletins_Parametros_Incubacao.IDIncubacao,
dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, " & _
      " dbo.Tabela_Boletins_Parametros_Incubacao.LoteMeioCultura,
dbo.Tabela_Boletins.IDBoletim AS Expr1, dbo.Tabela_Boletins.CodigoFinal " & _
      " FROM dbo.Tabela_Boletins_Parametros_Incubacao INNER JOIN " & _
      " dbo.Tabela_Boletins ON dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim =
dbo.Tabela_Boletins.IDBoletim " & _
      " where lotemeiocultura = '" & producao.Fields("lote").Value & "' ORDER BY
dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, dbo.Tabela_Boletins.CodigoFinal"
& _
      " ", LabDB, adOpenStatic
      If getb.RecordCount <> 0 Then
         getb.MoveFirst
         Do While Not getb.EOF
            Set nodX = Tree.Nodes.Add("R6B" & producao.Fields("pproducao").Value &
producao.Bookmark, tvwChild, "R6B2" & getb.Fields("idincubacao").Value &
getb.Bookmark & Rnd(0.123456789123457), "Certificado de Análise " &
getb.Fields("codigofinal").Value)
            nodX.Image = 14
            getb.MoveNext
         Loop
      End If
      producao.MoveNext
   Loop
erro:
   ErrosGerais
```

```
End Sub

Private Sub LerFiltro3()
On Error GoTo erro
   Combo2.Clear
   Combo1.Clear
   leulotes = 0
   leulotes = 0
   Tree.Nodes.Clear
   Set GE = New ADODB.Recordset
   GE.Open "select * from Tabela_MeiosCultura_Rastreabilidade where ge = '" & Combo3.Text & "'", LabDB, adOpenStatic
   If GE.RecordCount <> 0 Then
      Set producao = New ADODB.Recordset
      producao.Open "SELECT TOP (100) PERCENT dbo.Tabela_MeiosCultura_Producao.PProducao, dbo.Tabela_MeiosCultura_Producao.Data, dbo.Tabela_MeiosCultura_Producao.DataProducao, " & _
      " dbo.Tabela_MeiosCultura_Producao.Lote, dbo.Tabela_MeiosCultura_Producao.IDMeio, dbo.Tabela_MeiosCultura.Descricao, " & _
      " dbo.Tabela_MeiosCultura_Producao.QdePrevista, dbo.Tabela_MeiosCultura_Producao.DataValidade, " & _
      " dbo.Tabela_MeiosCultura_Producao.Confirmado " & _
      " FROM dbo.Tabela_MeiosCultura_Producao INNER JOIN " & _
      " dbo.Tabela_MeiosCultura ON dbo.Tabela_MeiosCultura_Producao.IDMeio = dbo.Tabela_MeiosCultura.Referencia " & _
      " WHERE Confirmado = 1 AND pproducao = '" & GE.Fields("pproducao").Value & "' " & _
      " ORDER BY dbo.Tabela_MeiosCultura_Producao.PProducao", LabDB, adOpenStatic
      If producao.RecordCount = 0 Then
         MsgBox "Dados não encontrados. Provavelmente a ordem de fabrico ainda não se encontra fechada!"
         Exit Sub
      End If
      producao.MoveFirst
```

```
Do While Not producao.EOF
    Set nodX = Tree.Nodes.Add(, , "T" & producao.Bookmark, "Rastreabilidade da ordem de fabrico " & producao.Fields("lote").Value)
    nodX.Image = 8
    nodX.Bold = True
    Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6G" & producao.Fields("pproducao").Value & producao.Bookmark, "Dados Gerais")
    nodX.Image = 13
    Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6R" & producao.Fields("pproducao").Value & producao.Fields("idmeio").Value & producao.Bookmark, "Reagentes (Rastreabilidade)")
    nodX.Image = 13
    Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6C" & producao.Fields("pproducao").Value & producao.Bookmark, "Controlo da Qualidade")
    nodX.Image = 13
    Set nodX = Tree.Nodes.Add("T" & producao.Bookmark, tvwChild, "R6B" & producao.Fields("pproducao").Value & producao.Bookmark, "Certificados de Análise associados")
    nodX.Image = 13
    Set getdadosproduto = New ADODB.Recordset
    getdadosproduto.Open "select * from tabela_Meioscultura_producao where lote = '" & producao.Fields("lote").Value & "'", LabDB, adOpenStatic
    If getdadosproduto.RecordCount <> 0 Then
      Set getdadosproduto2 = New ADODB.Recordset
      getdadosproduto2.Open "EXEC GET_MeiosCultura_Producao_Confirma @pp = '" & getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
      If getdadosproduto2.RecordCount <> 0 Then
        Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6C" & getdadosproduto2.Bookmark & producao.Bookmark & getdadosproduto2.Fields("pproducao").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Produzido em " & getdadosproduto2.Fields("data").Value & " com validade até " & getdadosproduto2.Fields("datavalidade").Value)
        nodX.Image = 14
```

```
            Set nodX = Tree.Nodes.Add("R6G" & producao.Fields("pproducao").Value &
producao.Bookmark, tvwChild, "R6C1" & getdadosproduto2.Bookmark &
producao.Bookmark & getdadosproduto2.Fields("pproducao").Value &
producao.Fields("pproducao").Value, "Quantidade produzida: " &
getdadosproduto2.Fields("qdeprevista").Value)
               nodX.Image = 14
            End If
         End If
         Set rastre = New ADODB.Recordset
         rastre.Open "EXEC GET_MeiosCultura_Producao_Confirma_2 @pp = '" &
getdadosproduto.Fields("pproducao").Value & "'", LabDB, adOpenStatic
         If rastre.RecordCount <> 0 Then
            rastre.MoveFirst
            Do While Not rastre.EOF
               If rastre.Fields("tipo").Value = 1 Then
                  unidade = "Litros"
               Else
                  unidade = "Grama"
               End If
               If externo = 1 Then
                  Set getdados = New ADODB.Recordset
                  getdados.Open "SELECT dbo.Item.ItemID, dbo.Item.BarCode,
dbo.Item.familyid, dbo.ItemNames.ShortDescription, dbo.ItemNames.Description " & _
                  " FROM dbo.Item INNER JOIN " & _
                  " dbo.ItemNames ON dbo.Item.ItemID = dbo.ItemNames.ItemID " & _
                  " where dbo.item.itemid = '" & rastre.Fields("idreagente").Value & "'",
GesPos, adOpenStatic
               Else
                  getdados.Open "SELECT * from tabela_reagentes where dbo.item.itemid = '"
& rastre.Fields("idreagente").Value & "'", LabDB, adOpenStatic
               End If
               If getdados.RecordCount <> 0 Then
                  Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value
& producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" &
```

```
getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: " & getdados.Fields("description").Value & " (" & rastre.Fields("idreagente").Value & ") - Guia de Entrada: " & rastre.Fields("GE").Value & ". Quantidade consumida: " & rastre.Fields("Quantidadereal").Value & " " & unidade)
                nodX.Image = 14
            Else
                Set nodX = Tree.Nodes.Add("R6R" & producao.Fields("pproducao").Value & producao.Fields("idmeio").Value & producao.Bookmark, tvwChild, "R6R2" & getdadosproduto2.Bookmark & rastre.Bookmark & rastre.Fields("nummovimento").Value & producao.Fields("pproducao").Value & Rnd(0.123456789), "Reagente: não detectado na base de dados GesPOS (" & rastre.Fields("idreagente").Value & ") - Guia de Entrada: " & rastre.Fields("GE").Value & ". Quantidade consumida: " & rastre.Fields("Quantidadereal").Value & " " & unidade)
                nodX.Image = 14
            End If
            rastre.MoveNext
          Loop
        End If
        Set getplano = New ADODB.Recordset
        getplano.Open "SELECT TOP (100) PERCENT
dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo,
dbo.Tabela_ControloQualidade_PlanoFinal.IDControloPlano,
dbo.Tabela_ControloQualidade_PlanoFinal.Produto,
dbo.Tabela_ControloQualidade_PlanoFinal.resultado,
dbo.Tabela_ControloQualidade_PlanoFinal.resultadonumerico, " & _
        " dbo.Tabela_ControloQualidade_PlanoFinal.Lote,
dbo.Tabela_ControloQualidade_PlanoFinal.Efectuado,
dbo.Tabela_ControloQualidade_Controlos.Descricao, " & _
        " dbo.Tabela_ControloQualidade_Controlos.Valor ,
dbo.Tabela_ControloQualidade_Controlos.vmin,
dbo.Tabela_ControloQualidade_Controlos.vmax,
dbo.Tabela_ControloQualidade_planofinal.idcontroloplano,
dbo.Tabela_ControloQualidade_planofinal.idcontrolo " & _
```

```
" FROM dbo.Tabela_ControloQualidade_PlanoFinal INNER JOIN " & _
" dbo.Tabela_ControloQualidade_Controlos ON dbo.Tabela_ControloQualidade_PlanoFinal.IDControlo = dbo.Tabela_ControloQualidade_Controlos.IDControlo " & _
" WHERE (dbo.Tabela_ControloQualidade_PlanoFinal.Lote = '" & getdadosproduto.Fields("lote").Value & "') " & _
" ORDER BY dbo.Tabela_ControloQualidade_Controlos.Descricao", LabDB, adOpenStatic
If getplano.RecordCount <> 0 Then
  getplano.MoveFirst
  Do While Not getplano.EOF
    Set nodX = Tree.Nodes.Add("R6C" & producao.Fields("pproducao").Value & producao.Bookmark, tvwChild, "R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Controlo Qualitativo " & getplano.Fields("descricao").Value)
    nodX.Image = 14
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R3" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor mínimo especificado: " & getplano.Fields("vmin").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R4" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Valor máximo especificado: " & getplano.Fields("vmax").Value)
    nodX.Image = 12
    Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, tvwChild, "R6R5" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value, "Resultado numérico obtido: " & getplano.Fields("resultadonumerico").Value)
    nodX.Image = 12
```

```
            Set nodX = Tree.Nodes.Add("R6R2" & getplano.Bookmark &
getplano.Fields("idcontroloplano").Value & producao.Fields("pproducao").Value,
tvwChild, "R6R6" & getplano.Bookmark & getplano.Fields("idcontroloplano").Value &
producao.Fields("pproducao").Value, "Resultado alfanumérico obtido: " &
getplano.Fields("resultado").Value)
               nodX.Image = 12
               getplano.MoveNext
            Loop
         End If
         Set getb = New ADODB.Recordset
         getb.Open "SELECT TOP (100) PERCENT
dbo.Tabela_Boletins_Parametros_Incubacao.IDIncubacao,
dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, " & _
         "                dbo.Tabela_Boletins_Parametros_Incubacao.LoteMeioCultura,
dbo.Tabela_Boletins.IDBoletim AS Expr1, dbo.Tabela_Boletins.CodigoFinal " & _
         " FROM dbo.Tabela_Boletins_Parametros_Incubacao INNER JOIN " & _
         " dbo.Tabela_Boletins ON dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim
= dbo.Tabela_Boletins.IDBoletim " & _
         " where lotemeiocultura = '" & producao.Fields("lote").Value & "' ORDER BY
dbo.Tabela_Boletins_Parametros_Incubacao.IDBoletim, dbo.Tabela_Boletins.CodigoFinal"
& _
         " ", LabDB, adOpenStatic
         If getb.RecordCount <> 0 Then
            getb.MoveFirst
            Do While Not getb.EOF
               Set nodX = Tree.Nodes.Add("R6B" & producao.Fields("pproducao").Value &
producao.Bookmark, tvwChild, "R6B2" & getb.Fields("idincubacao").Value &
getb.Bookmark & Rnd(0.123456789123457), "Certificado de Análise " &
getb.Fields("codigofinal").Value)
               nodX.Image = 14
               getb.MoveNext
            Loop
         End If
         producao.MoveNext
```

```
        Loop
    End If
erro:
    ErrosGerais
End Sub
```