Sampling: the weak link in the sanitary quality control system of agricultural products

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To ensure a high level of consumer protection, the European Union has in the past years published several regulations setting very low limits for a given number of food contaminants (pesticides, mycotoxins, heavy metals) in many agricultural products (cereals, oilseeds, dry fruits, coffee, spices, etc). These new regulations regarding the sanitary quality of agricultural products, compel both economic operators and officials of different EU member states to set up sampling plans and rigorous analyses aimed at checking whether a product lot complies with the required standards prior to its release on the market. While the laboratory analysis management today is outstanding thanks to the validated and efficient detection methods and procedures available for quality assurance in laboratories (accreditation), this is not necessarily true of the sampling operation, which seems to be the weak link in the sanitary control system for agricultural products. The sampling operation is often the main source of error when assessing the sanitary quality of a lot of agricultural commodities, with both commercial (downgrading of the product) and sanitary (marketing of a product which poses a health risk for the consumer) consequences. Therefore, it is essential for the operators involved to be aware of the significance and difficulties of the sampling operation, which requires important equipment and human resources. Furthermore, drawing up specific standards and guidelines, as well as setting up quality assurance procedures, at the level in charge of carrying out this delicate and important operation, are necessary.

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1 Introduction

The different food crises that have occurred in the last decade have led European authorities to considerably strengthen food safety regulations. To ensure a high level of consumer protection, the European Union has recently published several regulations setting very low limits for many food contaminants (pesticides, mycotoxins, heavy metals, dioxins etc.). These regulations currently affect many agricultural products (cereals, oilseeds, dry fruits, coffee, spices etc.). Conformity control of many commodities in relation to the requirements of these new regulations includes two distinct operations, namely, (i) sampling, aimed at preparing a reduced quantity (the sample) from a more or less important product lot (some quintals to thousands of tons) that is representative of the lot and whose size ranges from hundreds of grams to some kilograms (laboratory sample) and (ii) laboratory test, aimed at determining the content of contaminants by applying analytical means on the assay portion of the ultimate. In terms of results, these two operations are inseparable, because the final error on analysis data consists of the combined errors of all the sampling steps and analysis operations. In many cases, the error induced by the sampling operation is much greater than what occurs at the analysis step (Fig. 1).

This shows that any plan for the sanitary quality control of agricultural products must, in addition to the in-laboratory analysis method, define the sampling procedures. This will strictly limit errors induced by these two operations. Similarly, any regulation directives fixing food contaminants limits are bound to specify the sampling plan and method of analysis. While laboratory analysis is considered a delicate science that only uses performing laboratories with recognized expertise and qualified personnel, this is not true of the sampling operation that is frequently considered of secondary importance.

That is why sampling is often allocated to non-qualified people who work with unsuitable tools and use procedures that cannot ensure the necessary representativeness of a

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Abbreviation: **GMO**, genetically modified organisms

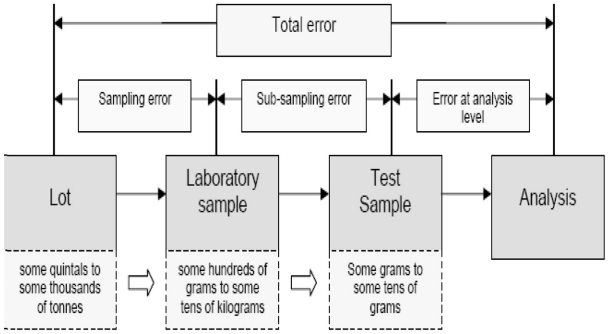

Figure 1. Source of variability associated with the valuation of contaminant.

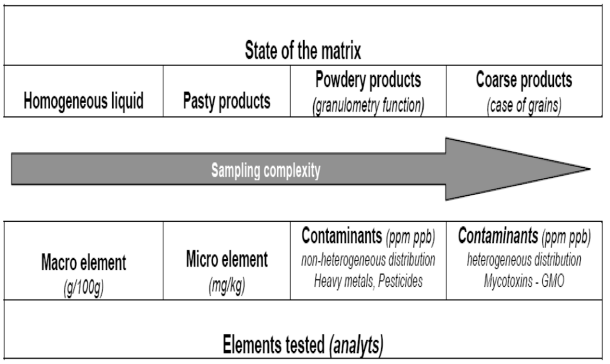

Figure 2. Size of sampling in relation to complexity of the matrix.

sample for sanitary quality evaluation. This lack of interest in the sampling link in the sanitary quality control chain is also reflected in the very low participation in various standardization commissions (CEN, ISO) in charge of establishing standards. In some cases, only two or three persons show up at a commission meeting to discuss regulation directives, which in the end will affect a great number of operators. This situation is due to a lack of knowledge about the significance and impact of sampling operations on the safety conformity of agricultural products, and also due to ignorance of the severe financial consequences as a result of inadequate sampling procedures.

2 Results and discussion

2.1 Sampling, a difficult operation

2.1.1 General observations

Sampling can be defined as all the operations that, applied to a lot of an agricultural product (some quintal to several thousand of tons), lead to a so-called laboratory sample of a workable size (some hundreds of grams to some kilograms). It is this laboratory sample which is in turn sampled down to the assay portion that will be used in laboratory to determine whether the lot of commodity under consideration meets the sanitary quality control criteria or not. It is, therefore, crucial that the final sample from which the assay portion is sampled be truly representative of the initial lot. Preparing a representative sample (not a specimen without any scientific value) is a delicate task, the complexity of which depends on several factors, namely, (i) the nature and state of the product; (ii) the nature of the quality factor to be tested; (iii) the type of packaging of the product; and (iv) the size of the lot to be sampled.

The difficulty of the sampling operation is linked to its nature and to the state of the product to be sampled (matrix). A homogeneous liquid product such as milk or wine is much easier to sample than a lot of maize grains or coffee that present a greater heterogeneity due to the nature of the product. Content and distribution of the quality factor to be tested in the matrix are other important points that need to be addressed when the sampling plan is considered. The lower the amount of the quality factor to be tested in the matrix; the more heterogeneous will be its distribution in the matrix. Thus, the preparation of a representative sample is even more difficult. One of the best illustrations of this situation is the case of mycotoxin discussed below (Section 2.1.2).

Other factors affecting the difficulty of the sampling operation are the type of packaging of the product (packaging unit, bags or bulk) and the size of the lot under sampling, which can range from hundreds of kilograms to several thousand of tons. This means that the sampling operation of a lot of an agricultural product needs careful thinking and analysis to map out a sampling plan that best matches the problem at stake.

2.1.2 The particular case of mycotoxins

To illustrate all the difficulties about the sampling operation, we consider here the case of mycotoxins that has recently been the subject of several regulations and directives in Europe, and that affect today a good number of agricultural products (cereals, dry fruits, coffee, spices *etc*.). Let us remember that mycotoxins are toxic metabolites produced by certain species of moulds (Aspergillus, Penicillium, Fusarium) that grow on a great number of agricultural products (cereals, oilseeds, coffee, grape) (for a review see [1]). Given their mode of contamination, the distribution of mycotoxins in a given lot of agricultural produce is particularly heterogeneous. Therefore, it only takes some contaminated grains to have a significant level of mycotoxins in the lot. Several studies on the heterogeneity of mycotoxins contamination have allowed the development of different statistical distribution models, *e.g.* the distribution of aflatoxins in peanuts [2, 3]. Furthermore, the same studies have shown quite a broad disparity in aflatoxin contents recorded in the

Table 1. Distribution of aflatoxin B1 in different lots of peanuts based on tests of 200 increments of 100 g each.

Lot size	Number of 100 g samples taken	Number of contaminated samples	Average rate of contamination µg/kg	Contamination extreme values µg/kg
15 T	200	3/200 (*1.5%*)	1.0	ND – 109
15 T	200	2/200 (*1.0%*)	3.0	ND – 609
20 T	200	7/200 (*3.5%*)	10.0	ND – 1938

Table 2. Distribution of aflatoxin B1 in different lots of peanuts, maize and cottonseeds.

Product	Number of laboratory samples tested	Size of laboratory samples	Average rate of contamination µg/kg	Contamination extreme values µg/kg
Peanuts (*oil mill*)	154	100 g	192	ND – 3233
Peanuts (*il mill*)	84	100 g	209	ND – 4235
Peanuts (*il mill*)	200	100 g	859	ND – 13 748
Maize (*ain*)	48	100 g	123	21 – 385
Cottonseeds	88	100 g	48	ND – 473

same lot of peanuts with values, recorded from one seed to another, ranging from 0 µg/kg to more than 1 000 000 µg/kg. Therefore, a single peanut seed heavily contaminated by aflatoxin in a 10-kg sample (20 000 seeds) is sufficient to have an aflatoxin content higher than the European regulation limits allowed for peanuts. The same heterogeneity of aflatoxin contamination has been reported in cottonseeds and maize [4–6]. A non-exhaustive summary of the findings of studies carried out on aflatoxins' distribution in peanuts, maize and cottonseeds is given in Tables 1 and 2.

Although only few data on the distribution of other mycotoxins (Ochratoxin A, fumonisin, vomitoxins) exist today [7, 8], there is every reason to believe that the problem of contamination heterogeneity is similar to the case of aflatoxins. Notably, a very recent study by IRTAC (Institut de Recherches Technologiques Agroalimentoires des Céréales) on the distribution of vomitoxin in different lots of French wheat revealed strong heterogeneity in the distribution of this fusarium toxin.

2.1.3 Other contaminants

While the sampling operation for other food contaminants (pesticides, heavy metals, dioxins) seems less problematic than for mycotoxins, it remains an important source of error that depends on the sampling plan to be followed and on the distribution of the contaminant in the product under consideration. The case of genetically modified organisms (GMO) does not really belong to the field of sanitary quality, but they present the same problem of contamination heterogeneity as observed with mycotoxins. However, the problem here is slightly different inasmuch as the required level of detection is clearly higher than in the case of mycotoxins.

2.2 Sampling standards and directives inappropriate to the sanitary control

Methodologies and procedures to be followed to obtain a representative laboratory sample from a given product lot are called the sampling plan. In the case of sanitary quality control of agricultural products, a rigorous sampling plan must at least define the following characteristics: (i) the size of the lot from which the sample will be prepared; (ii) the size and number of increments to be taken; (iii) the sampling points where increments will be taken from; (iv) the tools and equipment to be used to take the increments; (v) the size of bulk sample; (vi) the procedures for bulk sample homogenization and division; (vii) the tools and equipment to be used for homogenization and division; (viii) the number and size of reduced samples; (ix) the number and size of laboratory samples; and (x) the packaging and labeling of laboratory samples. This plan must also take into account the type of packaging (bag or bulk) and storage of the product to be sampled (lorry, wagon, barge, silos) (Fig. 3).

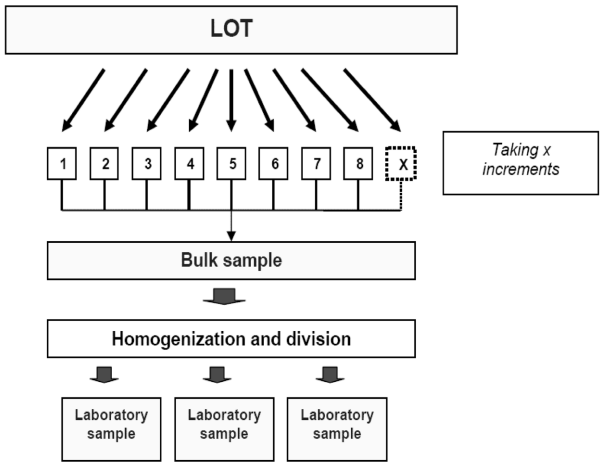

Figure 3. Scheme of preparation of sample.

In the field of sanitary quality control of agricultural products, implementation of such a sampling plan must take into account the technical and economic feasibility in order to make a sound choice between the two following risks: consumer risk, induced by the fact that the selected sampling plan can lead to the acceptance of non-conform product lots (dangerous to health) and producer risk, induced by the fact that the selected sampling plan can lead to the rejection of lots that do not conform with the standards. It is therefore crucial to assess the efficiency in relation to these two risks (consumer and producer) prior to the implementation of a sampling plan. An ideal sampling plan would lead to the acceptance of all the lots with a contaminant content below the allowed limits and to the rejection of those with contaminant content higher than these limits, thus reducing the risk to consumer and producer to zero. In reality how-

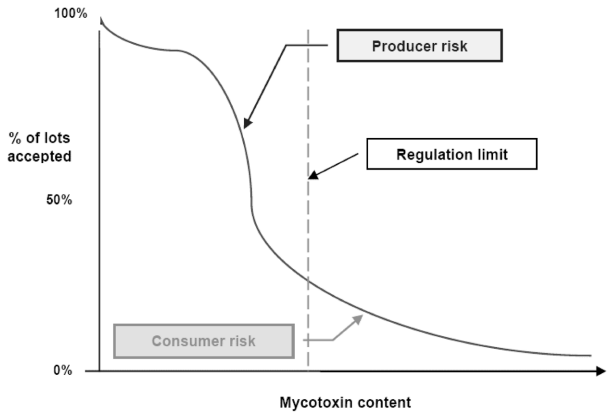

Figure 4. Typical OC curve to predict the portion of lots accepted and to evaluate the producer and consumer risk.

ever, depending on the selected sampling plan, there is always an amplification of one risk against the other. Fig. 4 is a graphic representation of the characteristics of a sampling plan for the detection of mycotoxins in whole grain products.

A certain number of sampling plans are now defined through specific standards published by various standardization organizations (CEN, ISO, GAFTA, Codex Alimentarius). Table 3 gives a non-exhaustive list of major normative texts in relation to the sampling of agricultural products.

Most of these standards were established with the intention of preparing samples to determine the commercial quality of certain products (cereals, oilseeds). This commercial quality is generally determined on the basis of the analytical criteria (proteins, oils, water), the contents of which are quoted in% and distribution of which is relatively homogeneous compared to what happens with mycotoxins (see Fig. 5).

This means that these sampling standards cannot be applied as such to the case of sanitary quality control, particularly

Table 3. List of major normative texts in relation to the sampling of agricultural products

Reference	Title of the standard
NF ISO 6644	Moving cereals and cereal meal Automatic sampling using mechanical samplers
NF ISO 13690	Cereals, vegetables and meals Sampling of static lots
NF EN ISO 542	Oilseeds Sampling
NF ISO 5500	Oilseeds cakes Sampling
pr-EN ISO 21568	Analysis and detection of GMO Sampling
GAFTA N°124	Sampling rules

to the detection of mycotoxins. This is generally mentioned when it comes to the field of application of these standards. In the absence of specific normative texts, it is vital that various commissions in charge of standardization endeavor to establish specific sampling procedures of the sanitary quality control of agricultural products. To this end, the people and structures concerned actively participate in meetings, and the work of these standardization commissions must come up with normative texts that are as consensual as possible. These new sampling standards should be established taking into account their economic feasibility and side-errors in relation to consumer (acceptance of non-conform lots) and producer (rejection of conform lots) risks mentioned above. At this stage, we should acknowledge the recent initiative by IRTAC in France that led to the draft on standards, dealing with this specific case of sampling grain products with a view to the analysis of non-uniformly distributed characteristics. The text is currently being tested in the field and will soon be submitted to CEN and ISO for consideration.

There is also a draft on sampling standards for the detection of GMO that is in the process of being adopted by CEN and ISO (pr EN ISO 21568). The unwieldy sampling plan suggested in the above-mentioned draft is a perfect example of

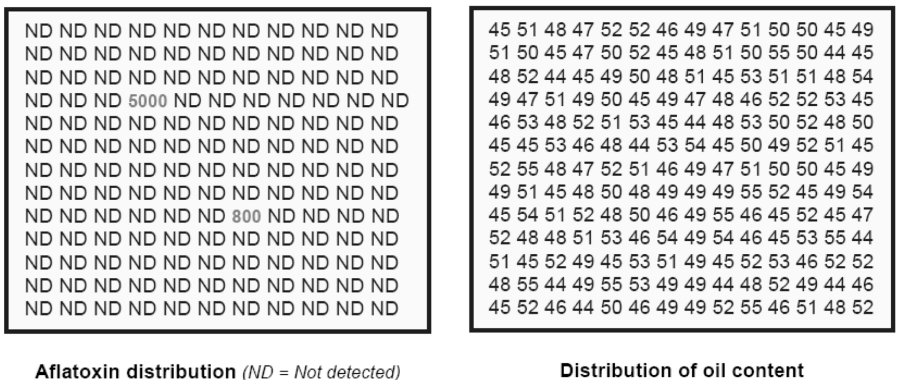

Figure 5. Comparison of the distribution of aflatoxin B1 and oil content in a peanut lot.

Table 4. Main directives in relation to the sampling of agricultural products for detection of a several contaminants (mycotoxins, heavy metals, dioxins, pesticides etc)

Directive	Contaminants
98/53/EC Modified by directives 2002/27/EC & 2003/121/EC	Aflatoxins
2002/26/EC	Ochratoxin A
2003/78/EC	Patulin
2005/38/CE	Fusarium toxins (DON, fumonisins, Zearalenone)
2001/22/EC	Heavy metals (Hg, Pb, Cd)
2002/70/EC	Dioxin and PCB
2002/63/EC	Pesticides

a normative text that is totally unrealistic and that seems to be out of proportion with the issue at stake (labeling of foodstuffs). We shall have the opportunity to go back over this draft for longer as it was unfortunately on the basis of this text that a recommendation on sampling for the detection of GMO was recently (4/10/2004) published by the European Union (Regulation (EC) No 1830/2003 of the European Parliament and of the Council of 22 September 2003 and amending Directive 2001/18/EC).

Table 4 gives references of major directives in relation to the sampling of agricultural products for the detection of a several contaminants (mycotoxins, heavy metals, dioxins, pesticides).

While we are thrilled that European Union has defined sampling plans for official control in the detection of food contaminants, implementation of some of these directives in the field is not without problems today. First, it is unfortunate that these directives are not clear enough about the methodology to be followed, thus leaving to the operator some freedom that can lead to significant errors in the preparation of the final sample and therefore possibly to litigations. The critical analysis of directive 98/53/EC published in 2001 [9] listed major areas that required improved definition to limit errors, namely regarding the: (i) sampling points; (ii) sampling tools; (iii) mixing conditions for bulk sample (homogenization); (iv) conditions and tools for the further division of samples from the initial bulk sample into the laboratory samples and the assay portion for analysis.

It is also worth noting that sampling procedures set out in these directives, particularly those related to the detection of mycotoxins (aflatoxins and Ochratoxin A) are totally inappropriate for the sampling of products that are stored and delivered in bulk.

At this level, directives 98/53/EC (aflatoxins) and 2002/26/EC (Ochratoxin A), which were initially established for sampling products in bags (peanuts, pistachio) have been recently reviewed, and the new directive (401/2006) (Regulation (EC) No 882/2004 of the European Parliament and of The Council of 29 April 2004) will take into account specificities of sampling products stored and shipped in bulk, particularly the sampling of cereals. This new directive will abrogate the directives 98/53/CE; 2002/26/CE and 2005/38/CE. To correct these deficiencies, an easy way would be for these directives to refer to the existing sampling standards (CEN or ISO) that perfectly expose the methodology to be followed and the instruments to be used, depending on the commodity at stake and its physical state. The same remarks apply to the new directive 2005/38/CE for sampling to determine the Fusarium toxins content (deoxynivalenol, Zearalenone, and fumonisins), this directive being identical to the directive 2002/26/EC (Ochratoxin A). It is regrettable that the European Commission was content with only copying the Ochratoxin A directive and did not find it necessary to assess the relevance of this sampling plan for Fusarium toxins. One would have expected that the sampling plan to control Fusarium toxins be less unwieldy than the directive 2002/26/EC, given that the envisaged regulation limits for deoxynivalenol, Zearalenone and fumonisins are fixed at levels of 100 to 300 times higher than the limits for Ochratoxin A.

Another criticism is that, generally speaking, these texts try to minimize the consumer risk, thus increasing the producer or exporter risk (rejection of conform lots) accordingly. In the same publication in 2001 [9] we clearly showed that the sampling plan chosen by the European Union in directive 98/53/EC considerably amplified the severity of regulation limits fixed by the EC 257/2002 regulation for the control of aflatoxins in dried fruits. Note that one of the most disputed measures of this directive 98/53/EC is that the decision on lot conformity is only based on the analysis of three sub-samples of 10 kg each. This means that only one subsample beyond regulation limits is enough to reject the whole lot even if the average of the results of the three tests meets the regulation limits. That is why the sampling operation in the case of the detection of aflatoxins in accordance with directive 98/53/EC appears today to be very often the luck of draw, thus entailing serious financial consequences for operators dealing with risky products. Currently, this is particularly true for pistachio from Iran: 50% of the lots shipped to Europe are rejected on the basis of official controls carried out in accordance with the sampling and analysis plan set out in directive 98/53/EC. Today, only Iranian pistachio lots with almost no aflatoxin contamination (<1 μg/kg) will have a chance to be accepted at the European borders. Note that, according to the regulations enforced by the World Trade Organization (WTO), particularly the SPS agreement on health and phytosanitary measures, if Iran were a WTO member country, they would have every right to initiate proceedings against Europe for a non-tariff obstacle to trade and would have every chance of winning this case.

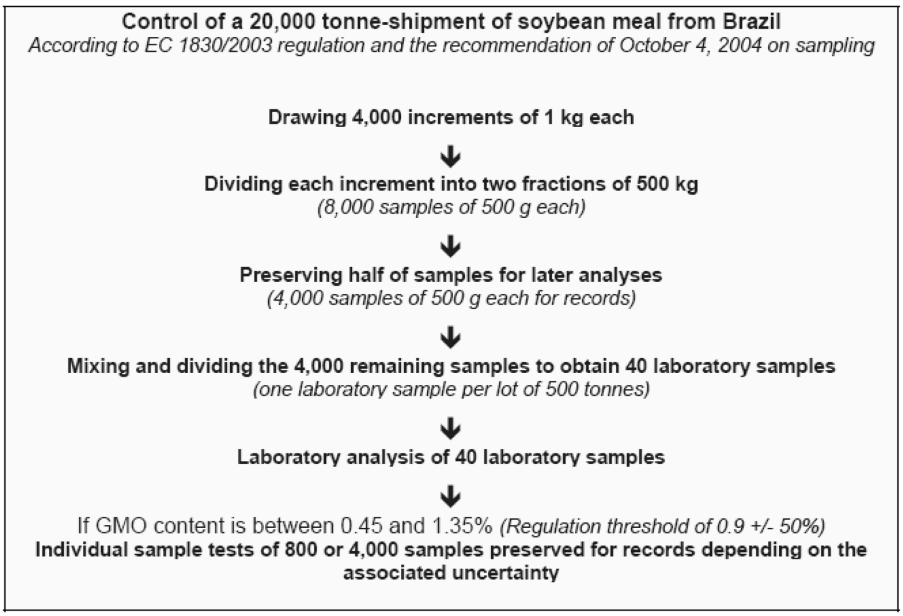

Figure 6. Scheme of control of soybean according EC 1830/2003 regulation.

To once again illustrate the severity of the European texts as regards the drawing of increments, we refer to the recommendation of October 4, 2004 by the Commission defining guidelines in relation to the sampling for the detection of GMO in the framework of the EC/1830/2003 regulation (Regulation (EC) No 1830/2003 of the European Parliament and of the Council of 22 September 2003 concerning the traceability and labeling of genetically modified organisms and the traceability of food and feed products produced from genetically modified organisms and amending Directive 2001/18/EC). This recommendation, which is widely inspired by the draft of the pr-EN ISO 21568 standards mentioned earlier, is based on a very heavy protocol of statistical evaluation meant to assess the associated uncertainty. This text indicates that for a lot of 500 tonnes, it is required to draw not less than 100 increments of 1 kg each, which will be divided into two fractions of 500 g each, one to be used in the mixing to obtain the bulk sample from which laboratory samples will be obtained, while the other will be preserved as an increment for records. In case the test results are close to the regulation limit (+50% of its value), it is advisable to assess the associated uncertainty by individually testing 20 to 100 increments depending on the dispersion of the results. Implementation of this recommendation seems totally unrealistic for operators, particularly in terms of economic feasibility as clearly shown by the case of a shipment of soybean meal (Fig. 6). Assuming 100 € per sample as an average cost for measuring GMO, it is clear that the cost of analytical control ranges from 4000 € (testing 40 samples) to 400 000 € (testing 4000 samples), and one must add to this amount the costs of the sampling operation and storage of increments meant for records. One wonders about the motives that led the legislator to draft such a sampling plan on GMO, which does not concern food safety but rather deals with product labeling (consumer information) (Fig. 6).

2.3 No result requirement

As mentioned in the Introduction, unlike laboratory analysis, the sampling operation is not subject to any exigency on quality and results, and this means that today anybody can offer sampling services. Appellations such as "first class company" or "market recognized company" on which many companies prize themselves are in fact just marketing ploys that are not based on any precise reference that can assure the quality of their services, particularly in the case of sampling for sanitary quality control.

We have the same problem with official controls carried out in Europe to guarantee that foodstuffs meet regulation standards insofar as regulation texts seldom state specific requirements for sampling whereas those related to laboratory analysis are widely specified. EC 178/2002 and EC 882/2004 regulations, which are today the basis of European food legislation, are a good illustration of this problem (Regulation (EC) No 882/2004 of the European Parliament and of The Council of 29 April 2004; Regulation (EC) No 178/2002 of the European parliament and of the Council of 28 January 2002). Whereas exigencies for laboratories and methods of analysis are covered by several specific articles in these two texts (method validation, accreditation, inter comparisons, reference materials), the sampling operation is not regulated by any requirement or any definite obligation. There is a clear inconsistency between what is required, the quality of laboratories in

charge of carrying out analyses and the near absence of obligation in this field for people and structures in charge of sampling. This is shocking when, as it has been shown at the beginning of this publication, one knows the significance and difficulty of the sampling operation in the detection of certain food contaminants.

It is, therefore, urgent that regulation provisions be taken to control the sampling operation and to subject structures in charge of sampling for sanitary control to the same exigencies of quality assurance as the ones required today in analytical laboratories. While the EN ISO/CEI 17025 standard, which is the reference for the accreditation of analytical laboratories (tests), deals well with sampling issues (point 5.7), these provisions only apply to laboratory structures that carry out the sampling themselves; and in fact this is very rare, be they public or private laboratories. The sampling operation is generally carried out by structures that are totally independent from laboratories.

Currently in France there is no food control laboratory that is accredited by the COFRAC for this sampling component of the EN ISO/CEI 17025 standard. Still on the EN ISO/CEI 17025 standard, all test reports by accredited laboratories that do not carry out the sampling operation themselves, must be marked with "Test results only relate to samples submitted for analysis". This precaution currently poses a difficult question inasmuch as it restricts the results to the sample submitted for analysis only and does not offer any possibility to go back from the test results to the initial lot from which the sample was taken. That means that an operator who would have been penalized for importing or trading a lot of foodstuffs that does not meet safety standards on the basis of analysis carried out by an official accredited laboratory, would have every right to quash this penalty before tribunals. This would also apply to a commercial dispute arising between a buyer and a seller.

This example shows how abnormal the current situation is, since a certificate issued under accreditation may not be recognized by tribunals due to the precautionary clause about the sample representativeness of the lot, whereas a certificate issued out of accreditation may be accepted as a result of the absence of a precautionary mark. The only way to correct this situation is to put in place a reference that sets out all the instructions to be followed to assure quality of the sampling operation. The text would serve as a basis for the accreditation of structures that are in charge of sampling as is the case for laboratories. Similarly, each sampling operation should be systematically subjected to a sampling report exposing the protocol followed for a given lot of product to the laboratory sample. All the sampling steps should also be traceable.

While there is an inspection standard (NF EN ISO/CEI 17020) that could serve as a reference for the accreditation of structures carrying out the sampling operation, this text covers all the fields of inspection and its contents are too general and do not deal with sampling alone. There should be either a review of this standard on inspection to bring in technical requirements specific to the sampling operation, or a draft of additional documents showing the protocol to be followed specifically for each area of sampling, as has been put in place by the COFRAC in France through analytical programs for the ISO/CEI 17025 standard.

In conclusion, sampling appears today to be the weak link in the sanitary control system in the field of agricultural products. This means that many European regulations established in recent years to ensure a high level of consumer protection can be challenged today on the basis of non-representativeness of samples on which controls are performed. This requires that all the people and structures concerned (public or private) be absolutely aware of the problem and significance of this sampling operation in the sanitary quality control system, which requires important material and human resources. It is also advisable that steps necessary to fill all the gaps currently noticed in the normative and regulation supervision of this sampling operation be quickly taken. These include (i) drafting and publishing sampling standards and directives specific to the detection of food contaminants in agricultural products. Texts must be sufficiently precise on protocol (methodology) to avoid all attempts at adaptation by operators in charge of the sampling operation. Accepted sampling plans must be as balanced as possible between the consumer and producer risks and take into account the practical and economical feasibility. And (ii), establishing an accreditation reference for people and structures that are in charge of sampling to ensure quality of services as is already in place for laboratories carrying out analyses on agro-food products. Without quick implementation of these measures, given the current restriction on exigencies in relation to food safety in the trade of agricultural products, we run the risk of highly increased regulation and/or commercial litigations in which sampling will be the major component of disputes.

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