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Food Control by Government Laboratories: Innovation, Flexibility, and No Restrictions by Reglementation

Konrad Grob*, Hans-Peter Neukom, Rolf Etter, and Ernst Romann

Abstract. Part of the work carried out by the government laboratories is devoted to permanent control of some critical foods and involves well-established and standardized methods. Another part, however, particularly the detection of frauds or poor manufacturing practices, presupposes advanced analytical techniques and flexible politics: an agile sense for hot subjects must be combined with good contacts providing the important information and innovative method development to find ways to obtain the evidence required. As shown for examples, ever new methods and approaches are needed, because the fraud and the negligent worker rapidly adjust to the methods applied for the control – in the end, the analysis may even protect a well arranged fraud. The swindler needs certainty about what the government chemists analyze and what methods they apply, and is, therefore, interested in paralyzing the work of the control, e.g. by requiring that only methods approved by time-consuming procedures are accepted by the court. The control must try to surprise and to create commotion, keeping everyone alert.

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A good portion of the work performed by the government laboratories for food control is routine work with established, often reglemented methods. Numerous controls must be carried out permanently: from the control of milk (water addition,

skimming, microorganisms), drinking water, frying fats, or mycotoxins in nuts, up to whether a 40% liquor really contains 40% ethanol or egg noodles contain the prescribed amount of egg. Such routine analysis is considered necessary for food safety as well as to keep up certain standards. If, for instance, the alcohol content of distillates were not constantly controlled,

the ethanol concentration in certain beverages would decrease in a short time – water is cheaper than the distillate. It may sound strange that a government laboratory helps to keep up the alcohol content of beverages, but this is part of the work performed to enforce that a product corresponds to what the label promises.

What Should Be Analyzed?

The number of subjects requiring control seems nearly unlimited; the government laboratories can analyze a small selection of them only. This selection is based on an evaluation of the importance, first priority, of course, being given to possibly toxic compounds or microorganisms. Food adulteration, inadequate (usually exaggerated) labeling, or poor manufacturing practices provide, however, an at least equal work load. Inevitably, the selection is also determined by knowledge about problems and technical feasibility of analyses: the laboratories cannot be blamed for the fact that numerous ways of deceiving the customer are unknown to the government chemist or cannot be checked analytically.

There is, however, also the danger that the same analyses are performed over and over again. Falling into routine is, in fact, the easiest way of doing the job: well-established methods can be applied, and there is no arguing about the interpretation of the results. Routine analysis may also be the result of lacking new ideas, which is

*Correspondence: Dr. K. Grob
Kantonales Labor
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Kantonales Labor
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CH-8030 Zürich

a problem of government chemists locked into their laboratory. Further, missing contacts easily cause that laboratories continue performing certain controls, although the situation has changed and the adulteration is no longer of interest anyway. In Switzerland, *e.g.*, it is illegal to use cocoa butter substitutes for the production of chocolate. The price of cocoa butter, however, has dropped substantially below that of the substitutes, rendering the search for substitutes unreasonable.

Stereotype analysis may also have another effect: alcoholic beverages are frequently checked for their alcohol content, at the end of the year giving the laboratory the satisfaction of having analyzed a great number of samples. Even a swindler might be happy with this: he has to accept that the ethanol content must be accurate, but he also knows that other aspects are not controlled, *e.g.* whether or not all the ethanol is really from the source shown on the label of the bottle.

The Spiral of Depressed Price and Deteriorated Quality Driving towards Adulteration

Adulteration may be deliberate, but can also be the result of pressure on prices. In a free market, a well-known mechanism may cause degradation of quality and easily ends up in adulteration; it could correspond to the following story: producer 1 sells a perfect product at a good price. One day, however, a competitor enters the market with a comparable product sold at a far lower price. The directors of producer 1 are upset and, of course, come to the conclusion that it is impossible to make and sell the product at such a low price. They assume that the competitor uses a trick, decide to follow the competitor, and also lower their price. The competitor, in turn, is forced to react and, if his original product was still 'real', now feels compelled to exploit some 'possibilities' in the gray area. This starts a development which may easily proceed to a fraud.

In some (probably few) cases, the government chemist stops the above spiral by establishing rules (often supported by the food industry involved). For instance, the amount of egg to be added to egg noodles was regulated. Otherwise some producers would probably have reduced it over maybe many steps to the point of a hen running through the park in front of the factory (shown in color in advertisements) and the director enjoying the eggs for his breakfast. Some dye might have imitated the eggs in the product. The story is, of course, not real for the egg noodles, but maybe for some other products. It should be unnecessary to stress that many producers would never consider frauds.

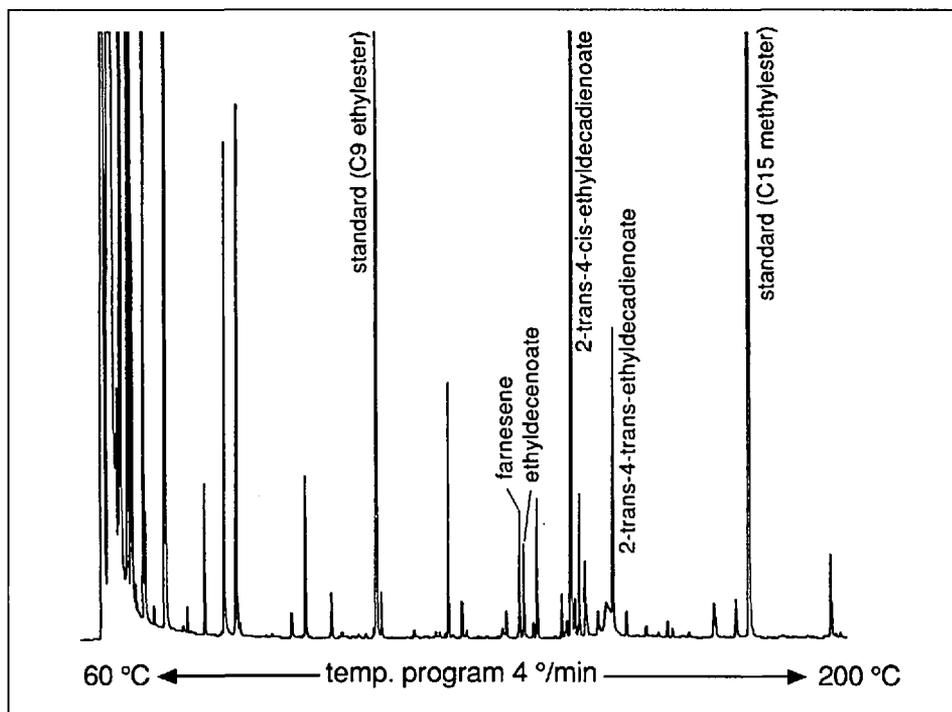


Fig. 1. GC-FID Analysis of a Williams distillate: 10 ml of distillate extracted with 10 ml of hexane; two internal standards. 25 m x 0.25 mm i.d. column coated with Superox 0.6 (a polyethyleneglycol) of 0.17- μ m film thickness; 0.65-bar inlet pressure (H_2); 0.2- μ l on-column injection.

Food control obviously deals with the negative exceptions.

Fraud Adjusted to Control

If government chemists always carry out the same analyses, producers learn about which aspects to care, and they adjust their priorities. If really the key aspects are analyzed, this has indeed the desired effect, but it may also have the side effect that other problems, which may not even be known to the government chemists, are neglected: manufacturing practices may deteriorate as a result of insufficient attention, methods and raw products are debased in the interest of lowering prices, or foods are adulterated with the certainty of not being detected. Some examples from our laboratory should illustrate such problems.

Example 1: Williams Distillates

About ten years ago, we analyzed a Williams (pear) distillate with an extremely weak flavor, obviously intended to enter the market as a low-price product. The flavor of Williams pears is relatively simple: the principal components consist of ethyl *cis/trans*- and *trans/trans*-decadienoate. The concentration of the sum of these two esters was, in fact, *ca.* 4 mg/100 g of abs. EtOH, instead of 20–40 mg found in average good distillates. The producer claimed to have used mediterranean Williams pears (our reference Williams distillates were made from Swiss pears). Regardless of whether this claim correspond-

ed to the facts, the distillate was refused, and a minimum concentration of 10 mg/100 g abs. EtOH was set for the two important esters. After rejecting many more distillates with insufficient flavor, the products all of a sudden improved: ester concentrations were at levels of *ca.* 15 mg. We had, of course, to accept them now.

Shortly later, we obtained an industrial Williams flavor consisting of the two dienoates and *ca.* 25% ethyl decenoate. The latter compound is also present in Williams distillates, but concentrations usually correspond to *ca.* 1% of the dienoates only (Fig. 1). The interesting point: the 'improved' Williams distillates also contained ethyl decenoate at concentrations 3–5 times above normal. This revealed that the products were flavored, which, according to Swiss law, is illegal. Many samples were, therefore, refused again. It was remarkable to note that the producers did not immediately stop adding flavor; they obviously wanted to try the reliability of our method. We did not inform them about our way of detecting the fraud, of course.

Some two years later, Williams distillates from the same sources contained substantially less ethyl decenoate at acceptable concentrations of the flavor components, and we had to accept the product again. Was the product really made of more or better Williams pears? After some more basic studies on various components, we noticed that the product was still artificially flavored, although with a flavor containing less ethyl decenoate. We

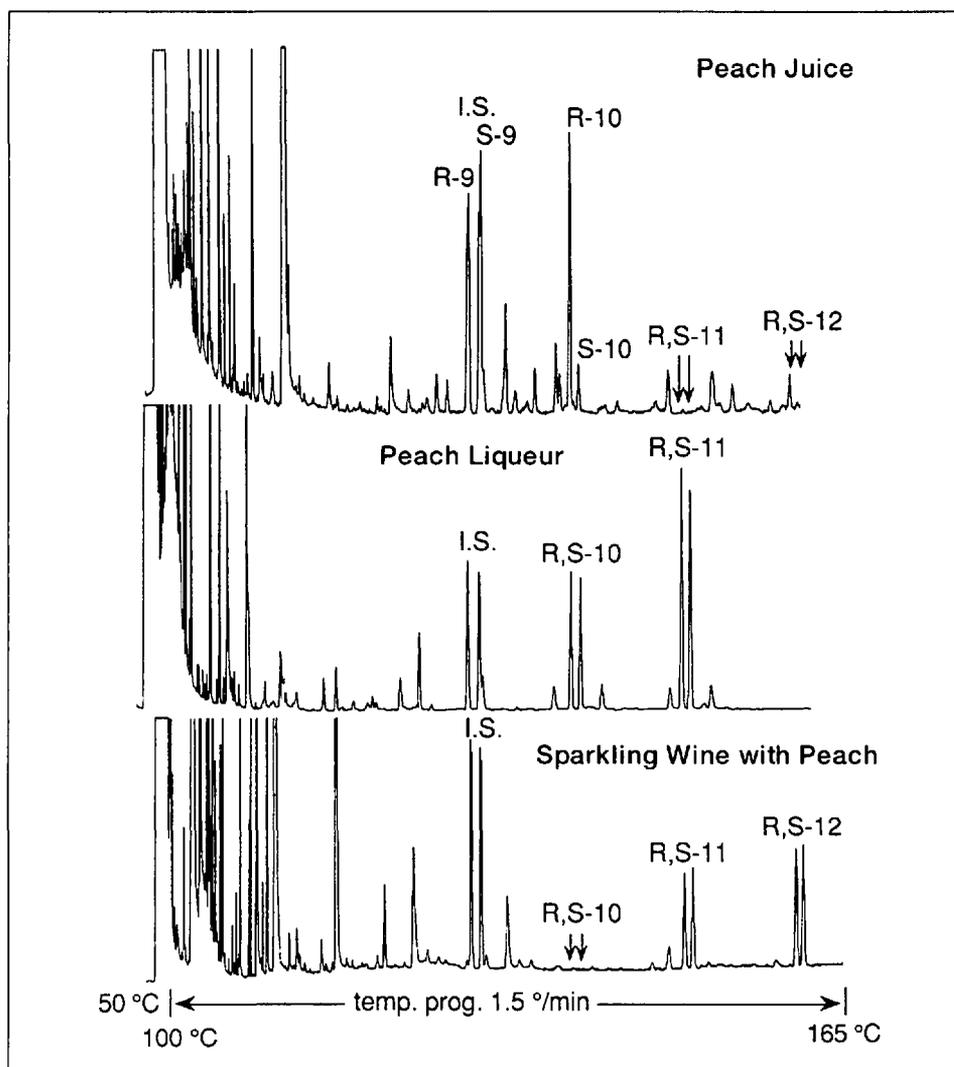


Fig. 2. GC-FID Chromatograms of extracts from peach juice and two alcoholic beverages with peach flavor. Peaks labeled by C number of the γ -lactone and as (*R*)- or (*S*)-enantiomer. Racemic γ -nonalactone was added as internal standard (I.S., 0.2 ppm for the peach juice and 2 ppm for the two other beverages).

prefer not to describe our new method, because this would probably prompt the 'other side' to again better adjust themselves to our methods and compel us to develop another new method.

This example taught us that only a steadily improved control is capable of recognizing adulterations, because the producers learn to 'fulfil' our requirements (even if we do not tell them what they are).

Example 2: Lactones as Flavors in Beverages

The second example illustrates what happens if regulations are not enforced. According to Swiss law [1], distillates labeled by a fruit must be produced from this fruit without addition of flavors or alcohol from other sources. The flavor of liqueurs, however, may be 'slightly reinforced' by 'flavors identical to the natural flavor' (produced synthetically). No control, however, was performed, and the results of the recent analysis of the γ -lactones in various beverages and foods turned out as it seems typical when a control is performed the first time. γ -Lac-

tones are important flavor components in many fruits, such as in strawberries, apricots, peaches, and coconuts. They are chiral, which provides us a relatively easy means of distinguishing natural and synthetic flavors.

Most of the distillates analyzed only contained the natural γ -lactones, *i.e.* primarily decalactone and dodecalactone, of which *ca.* 95% consist of the (*R*)-enantiomer (GC analysis on a chiral cyclodextrin stationary phase). The flavor of liqueurs, however, was not just 'slightly enforced': not even a small amount of natural γ -lactones could be detected. This has been shown previously for beverages based on apricots (or the corresponding synthetic flavors) [2].

Fig. 2 shows similar results for beverages based on peaches. Beverages were extracted with hexane/*tert*-butyl methyl ether; extracts were injected on-column onto a 25 m \times 0.25 mm i.d. capillary column coated with OV-1701 containing 30% of 3-*O*-acetyl-2,6-di-*O*-pentyl- α -cyclodextrin (0.225- μ m film thickness).

As shown for the peach juice (top chro-

matogram), the γ -lactones of peach nearly exclusively consist of decalactone, of which *ca.* 90% is the (*R*)- and 10% the (*S*)-enantiomer (in agreement with [3]).

The center chromatogram was obtained from a peach liqueur. The γ -lactones consisted of decalactone and undecalactone; peak areas of the (*R*)- and (*S*)-enantiomers were identical, *i.e.* no (*R*)- γ -decalactone from peaches was detectable. The flavor of the γ -lactones was, therefore, synthetic to more than 98%, which is certainly not in agreement with the 'slight enforcement' of the natural flavor allowed by law. Furthermore, the identity of the synthetic flavor with the natural γ -lactones only concerned the class of compound; neither the number of C-atoms nor the enantiomer ratio corresponded to the γ -lactones from peaches, *i.e.* the added flavor had a composition which certainly does not deserve the description 'identical to nature'.

The bottom chromatogram was obtained from a beverage 'au vin mousseux et aux extraits de pêche' (sparkling wine with peach extract). No (*R*)- γ -decalactone could be detected, however, *i.e.* there was no trace of natural peach flavor of the 'peach extract' promised by the label; the γ -lactones found consisted of racemic undecalactone and dodecalactone and were, therefore, not 'identical to nature'.

Nearly all liqueurs turned out similar to those above; producers showed to be surprised when confronted with the analytical results. The argument that less (concentrated) synthetic flavor was added than peach extract is, of course, not valid, because the two additives cannot be compared. If it is technically impossible to produce a strong peach flavor without the help of synthetic flavors, the label must be adjusted as well as the law.

Example 3: Olive Oils

Adulteration of olive oils has a long tradition. It is highly profitable, because a high-quality olive oil may cost ten times more than another edible oil. If, for instance, a lorry driver transporting the oil from the press to the firm confectioning the oil replaces 10% of his load by a cheaper oil, he may make as much as \$ 20 000 profit.

Some 10–15 years ago, admixture of rape seed oil to olive oil must have been frequent or even almost normal. In an effort to get this fraud under control, the sterol analysis became widely applied: rape seed oil contains brassicasterol, which is not present in olive oil, and nearly hundred times more campesterol than olive oil. At many places, olive oil was unloaded only after the brassicasterol and campesterol concentrations were determined.

Maybe as a result of this campaign, we could not find a trace of rape seed oil in some 300 olive oils from the Swiss market analyzed 1989–1991 (detection limit, 0.5%) [4].

This does not mean that oils are no longer adulterated. Frauds just became more subtle: the oils added are selected such that the government chemists do not detect them by the classical methods [5]. Everybody involved in this business seems to have at least a vague idea of what the government chemists analyze and of the conclusions about which oils can be added up to what concentrations such that it remains undetected.

Even more adulterations involve admixture of a low-quality olive oil to a high-quality oil (different qualities of olive oils were recently described by *Wessels* [6]) – price differences also include a factor of four. The official EC method for determining the cheapest olive oil, the oil extracted by solvent from the press residue, analyzes the triterpenediols erythrodilol and uvaol. These diols have no taste and are not toxic, *i.e.* are just the marker components for the recognition of solvent-extracted oil. To enable addition of such oil to higher-priced press oils, obviously some firms remove these diols at least partially, *e.g.* by oxidation with dichromate [7] – big machinery is kept running just to ‘satisfy’ the analysis of the government chemists – the producers probably smile thinking of the considerable efforts made by the control laboratory for the determination of the triterpenediols and the wrong conclusions it draws.

The official method for determining refined olive oils usually involves the determination of conjugated dienes and trienes by UV spectroscopy. There are, however, refined oils on the market which are prepared in such a way that their UV spectrum corresponds to that of an extra virgin oil [8]. A possibility of eliminating the conjugated dienes and trienes involves maleic anhydride: a *Diels-Alder* adduct is formed, which can be removed in a subsequent neutralization [9]. The costs of the corresponding procedures seem to be no problem compared to the profit made by selling a cheap oil under a better label.

At least for the determination of solvent-extracted oil in a pressed oil or refined olive oils in extra virgin oil, the official methods are obsolete: they only allow the detection of frauds by some uninformed outsiders or some clumsy deceivers. There are new methods for detecting such adulterations (*e.g.* [10][11]), which revealed, in fact, adulterations in large numbers, not only for olive oils. It would not be surprising, however, if they were offset by new tricks in a short time.

If adulteration is profitable, there seem to be acrobats with an astonishing flexibility to adjust to the control methods. If a government chemist continues analyzing edible oils by the fatty-acid composition or by the conventional sterol method only, thinking that he can perform a rapid check of the olive oils after having analyzed chewing gums and whiskies, he risks being laughed at. It approaches arrogance to believe that he (and his text books) are so knowledgeable to catch the silly swindler by the first injection. The ‘other side’ learns rapidly and probably knows more about the oil than the government chemist.

At least for the analysis of olive oils, the government chemist has two options: either he gets himself well informed and proves the new methods of fraud with the necessary expertise and appropriate analytical methods or he stops performing oil analysis, leaving it to colleagues specialized in this field. The easy go with the simple analysis of the classical type, which was successful 10–20 years ago, is a waste of time today – no longer, every small control laboratory can invest enough efforts to be capable to analyze all the foods of the market.

Further Examples

It is easy to elongate the list of examples showing that only innovative and flexible government chemists catch more than just some small ignorants who adulterate foods or apply other illegal practices in an easily detectable way.

A story which happened 70 years ago was recently mentioned by *Oeser* [12]: As artificial lemon juice (citric acid in water) was detected by analyzing the ashes, the fraud was improved by potash. When the government chemist improved his method by adding a determination of nitrogen, this was also added to the product, and so it went on with the extract, sugar, and glycerol.

As too many laboratories determined additions of glycerol to wine, diethylene glycol was added – and was only found because insiders blabbed out. It is, of course, no longer used, which does not rule out, however, that again other substances are added.

Diethylstilbestrol was applied rather widely to accelerate the growth of calves and to reduce their feed consumption, but it seems that it was replaced by ‘better’ means rapidly after some cases became public (an issue of interest, of course, only for countries disallowing the use of these hormones).

Many ‘natural’ vegetable oils are advertised as ‘cold pressed’, ‘non-refined’, or similar, and sold at a correspondingly high price. After having developed a new

method for the control of such claims [10], nearly half of the products tested (but only few of the olive oils) turned out to consist of or contain oil treated more intensively than declared, some of them even being refined rather brutally. With the conventional UV-detection method, raffination could have been unambiguously determined just in a few cases. When confronted with these results, many producers had to admit the application of high temperatures during pression, steaming, or other steps, and excused themselves citing professors having said that pression at 90° could be taxed as ‘cold pressed’, and that an oil steamed at 160° would still be unrefined. Such practices are no problem, of course, as long as nobody performs an efficient control!

Apparently, government chemists were not aware of the large amounts of mineral oil which can often be found in foods, originating from release agents, packaging materials, or lubricating oils [13][14]. Release agents consisting of refined mineral oil are delivered by tank lorries (and leave it again a few days later with the foods). As nobody performed a control, some used them rather carelessly. The technical names given to them, pleasing to the ear, supported the idea that they could be applied without hardly any restriction. In the end, concentrations in the foodstuffs reached thousands of ppm.

Innovative Controls and Analysis Methods

There is no doubt that certain subjects require a permanent control by the government chemists. This may involve a method applied over decades, especially if the legal limits include a definition of the analytical method. Frying oils, for instance, are often used for too long even after many warnings, and in winter times excessively high concentrations of fungicides and nitrates are found in salad even after a ten-years campaign. Water is expected to be added to milk as soon as controls are stopped. In other fields, however, innovation of subjects and methods is a prerequisite. Illegal veterinary drugs and sterilizing agents in wines are replaced as soon as their use became public. Usually, it does not make sense to continue analyses for more than a few months – maybe a check some ten years later is more rewarding.

It is impossible to control all aspects of foods, nor is the government laboratory directly responsible for food safety, good manufacturing practices, or correct identity of the foodstuff; the producers and vendors carry this responsibility and have to ensure the complete control. The

government laboratory carries out spot checks, making sure that the control of the foodstuffs is performed properly.

Most important is its presence as an authority, convincing the reluctant about their responsibility, and its leadership in setting standards and criterias. It certainly has an impact on quality control in a factory, if those responsible know that the product might get into trouble when analyzed by the government laboratory.

The government laboratory must try to get a maximum effect out of the limited resources available. This means doing well-respected work in ever changing subjects, particularly in those areas where the control of the producers and vendors is unsatisfactory or insufficient. This requires intensive studies on such subjects, good contacts with leaders in the field, and permanent investment into the development of new methods. A government chemist who applies the same old methods for always the same range of products, maybe with a detailed statistical evaluation and beautiful graphic presentation of the results, is not efficient in this respect.

The swindler is threatened by an agile control. He wants to be sure that raffination of the oil is controlled by UV spectroscopy, such that he can adjust his method to this control; he wants to have control over the government laboratories' control. A good fraud requires considerable development work and maybe substantial investments, which must be paid off before a new control method is introduced. For such reasons, innovative control generating surprise effects has a much broader (psychological) impact than the control which is really performed; it deters possible swindlers and creates unease among those applying poor manufacturing practices.

The Controlled Government Chemist

Do the government chemists primarily control producers, or do the producers control the government laboratories? In several countries, it rather seems to be the second. Usually, the control of the government laboratories occurs *via* analytical methods: only results obtained by certain methods are accepted. If there is no method for detecting something, the producers can feel safe that a fraud remains undetected (at least the control cannot take legal actions against it).

Ever new analytical methods are required to provide information on subjects not studied before, to enable the analysis of more samples by the few people available, but also to replace older methods which became ineffective because, *e.g.*,

the marker components analyzed by the official method for detecting solvent-extracted or refined olive oils has been removed by the clever swindlers. To promote the efficiency of the government laboratory, innovation must be encouraged, and freedom must be given to act rapidly on the basis of such new methods. Legislation also must be agile to support such activity.

Control only by Certified Methods?

At a time the economic leaders loudly call for more liberal laws, liberalism seems to be in great danger for analytical chemistry; it seems that some food industry wants to fetter the government laboratories in the course of the new European legislation. In some countries, the government chemist is allowed to do his work with some reglemented methods only, which are nearly invariably old and technically outdated. If evidence on adulteration is obtained by a new method, lawyers block the government chemist. It is not asked whether the analysis is correct (which could be proved by independent experts), but whether he applied a method given to him in a recipe book. This paralyzes control work and, of course, stops innovative people working in the field.

If methods must be certified by a time-consuming procedure, many of them can be applied only after the subject is no longer of interest. The subjects, *e.g.*, of diethylstilbestrol applied to calves or diethylenglycol and (more recently) methyl isothiocyanate in wine were of interest for at most one year and, of course, the methods applied had to be created instantly. Certification of the methods years later would have been a waste of resources. There is no doubt that methods for the more or less permanent use should be well-defined and controlled, but there must remain the possibility of obtaining evidence by completely new ways. In some cases, it is even important to act on the basis of methods which are not fully disclosed – the artificially flavored *Williams* was an example for this.

People having an interest in hindering government laboratories might (mis-)use the certification procedures for new methods as an excellent tool to delay the application of new analytical procedures with an inconspicuous justification.

Reglemented methods also create the danger that legal limits get interpreted in a wrong manner. Returning to the admixture of solvent-extracted olive oil to a pressed oil: erythrodiol and uvaol are the markers officially used for solvent-extracted oil; in a pressed oil, they must not exceed 4.5% of the total sterol content. Some re-interpreted this law, saying that

an oil containing less than 4.5% of these components must be accepted under the label of a pressed oil. As no alternative method for determining solvent-extracted oil is accepted, there is no chance to prove on adulteration. This ends in the absurd situation that a 'de-erythrolied' extraction oil becomes a pressed oil, and that a method conceived for preventing adulteration with extraction oil sanctions it.

Peaceful Finale?

Some of the European proposals to reglement analytical chemistry in government laboratories sound frightening. To paint it drastically, a peaceful scenario with everybody being relaxed and laboratory staff manipulating at the burettes occurs. The government laboratories only apply new methods after a time-consuming certification procedure. Occasionally, somebody takes the effort to get a method through this procedure, but most analysts lost their impetus in doing innovative work. Chemists no longer work on analytical methods, but sit at the computer, making statistics and beautiful graphics on the results obtained by analyzing the same product over twenty years. Producers and vendors are also happy, because they feel safe and reduce costs in their analytical division. Since the government laboratories lost their capability of developing new techniques and methods, a bad surprise is quite impossible.

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