
Spices: quality control and standards

H. van den Dool, International Flavors & Fragrances (Europe), Hilversum, The Netherlands

The opening lecture of the London symposium on spices of the Phytochemical Society of Europe opened a real Pandora's box by its review of problems and challenges.* The contents of the box may be characterized by the titles of two books shown in the window of a London bookshop, one being *Herbs and Spices. Guide to 60 of These and Explaining their Identification, Cultivation And Uses*, and the other *The World's Last Mysteries*.

Also belonging in Pandora's box are the problems and challenges connected with quality control and standards in the field of spices. The problems encountered here are everyday problems in the trade for the analyst, over which standardisation committees have sometimes stumbled. In addition, however, workers in this field face problems of definition.

A look at the symposium program shows titles mentioning mustard, dill, and onions, as well as pepper, capsicum, and ginger. Moreover, one title asks: "Herbs and spices, what is it all about?,"* and another reads "Herbs and spices in medicinal preparations."*

It appears from this that herbs as well as spices are involved and that their end-use is not restricted to food but also includes pharmaceutical products. Both food uses and pharmaceutical uses require control over the quality used. (It should be recalled here that the quality of a product must be defined relative to the purpose for which it is intended.)

Quality specifications may be laid down in standards that reflect the common opinion about how the quality of a product or one of its grades should be defined. Hence standards have to be generally accepted—this common acceptance will facilitate commerce. General acceptance also requires that the tests used to determine the standards have to be reproducible and repeatable.

Pharmacy has laid down its standards in its pharmacopoeias, whereas the spices and herbs trade over the centuries has developed traditional standards. Based on these pharmacopoeial and traditional standards, legal quality requirements for spices and herbs have been drafted. This all looks relatively simple: historical development of the spices and herbs field nicely translated into standards. Taking, a nearer look

at it, however, opens the Pandora's box mentioned earlier. Grading systems may differ by country of export or import; analytical methods for the determination of one specified characteristic may differ and lead to widely spread results.

It is here that the technical spices and condiments subcommittee of the International Standardization Organisation (ISO) tries to arrive at a less complicated situation. It is understandable that success is easier in the less traditional field of analytical methods than in that of the grading systems. In many cases the ISO standards can only reflect the state of the art at a certain date.

The problems arising here stem from the fact that many spices originate from less developed countries. The argument raised against many of the more modern analytical methods was that these less-developed countries could not afford the necessary instrumentation and for that reason were opposed to including more modern specific analytical methods in spice standards.

Another problem with the same origin is the trend in some of the less-developed countries to try to lower requirements and have the present standards changed accordingly. They argue that it is not possible for them to produce spices satisfying present standards. Instead of trying to improve their production methods they want to lower standards. This trend was also noticeable in the discussions in ISO's subcommittee. However, in trade the consumer industry will continue to adhere to present standards, and will select only those batches which satisfy their requirements.

To point to such apparently commercial, and to a certain degree even political, problems may seem out-of-the-way in a symposium of the Phytochemical Society. They indicate, however, a borderland between socioeconomic matters and chemistry in which standards may provide the points of contact between both fields.

Back to the standards themselves. One of the problems that may arise is the complexity of grading systems. In the early days of the spice trade, external characteristics generally determined the quality of a spice. Grading systems based on these external properties were developed over the years, the final system often depending on the producing country as well as on the importing one. Many of these grading systems

*These three papers presented at the symposium will be published in upcoming issues of **Perfumer & Flavorist**.

are still in full operation and apparently will continue to do so as they constitute reference frames for producer, middle men, broker and buyer.

A typical example of such a complex grading system is provided by cinnamon. Cinnamon, in a number of countries, is confounded with cassia, which has a similar flavour. Historically, cassia is the oldest spice of this type known in Europe. Cinnamon comes from Sri Lanka, the Seychelles and Madagascar, whereas cassia comes from China, Viet Nam and Indonesia.

Cinnamon and cassia are both dried bark, which comes in rolls, hollow tubes, sometimes filled with pieces of the same bark. The thinner the bark and the lighter its colour the finer the flavour. The grading systems depend on the exporting country. The Sri Lanka system distinguishes between quills, quillings, featherings, and chips. There are 13 grades of quills, the best grade, starting with "alba," and followed in descending order by six continental, three Mexican, and three Hamburg grades. Selection criteria are the length of the quills, their diameter, the number of whole quills of about 1 m in 1 kg, the extent of mottled or foxy cinnamon (that is cinnamon with reddish brown to dark brown patches) and finally the percentage of broken pieces of the same quality. Seychelles and Madagascar cinnamon is graded in four classes, based on whether the bark is scraped or not, on its thickness, and on whether it consists of whole tubes or pieces.

In China cassia is classified in three grades, based on flavour and whether rolls or pieces. Saigon classifies its cassia by three grades according to thickness of the rolls and with a fourth grade, comprising the broken pieces resulting from sorting and handling. The Indonesian classification system is related to the Sri Lanka system for grading cinnamon. However, it is less complicated since it recognizes only three grades of scraped bark rolls with a fourth class for the broken pieces.

It is clear that grading on the basis of these systems has to be carried out by experts. Assessing the degree of foxiness is usually done by visual inspection, as is the inspection of the fillings of the hollow quills. Here fraud with lower quality material, foreign matter, and immature green bark scrapings has to be looked for. To determine the extent of all these characteristics and defects reliably, by visual inspection, requires years of experience.

Luckily, not all grading systems are as complicated as those for cinnamon and cassia. Another tropical spice, cloves, has only three grades, based on the proportions of defects such as fermented cloves, mother cloves, headless cloves, and foreign matter.

To take an example from the temperate zone, Hungarian paprika powder is classified into eight grades according to colour and pungency. The colour may range from pure red to greenish-brown, the pungency from not-pungent to very pungent. Also to be assessed by visual inspection are requirements for freedom from contamination by extraneous matter, moulds, insects, and rodent hairs, the latter not so long ago having played a role in some protectionistic,

import-obstructing actions. A problem arising here was the extent of visual inspection. Should magnifying glasses be used or microscopes? The standard today is that it should be by the "naked eye (corrected, if necessary, for abnormal vision)" and, with "magnification up to 10 times if necessary in particular cases." Further it is obligatory to state in the test report, should any magnification over 10x be used.

Using such detailed wording for what on first sight seems quite simple, may seem to be nit-picking. Those who have some experience with arbitration and the juridical disputes connected with this kind of affairs know that such careful wording is necessary. This, of course, is true also for analytical testing and its results. The more subjective a test is, the more such care is required.

It is understandable that the trade has looked for additional methods to assess the quality and hence the value of spices. And preferably less subjective methods. Such methods became available with the onset of analytical chemistry, in particular the analytical methods developed by pharmacists for their purposes. From these result the oldest analytical methods in the spice trade such as the determination of ash, that of water insoluble ash and acid insoluble ash, and the determination of moisture.

The determination of ash content developed into a kind of art. Nevertheless, it was necessary to standardize the method to obtain comparable results between different laboratories. Ashing temperatures should remain within the $550 \pm 25^\circ\text{C}$ limits to prevent volatilization of alkali salts. The endpoint is determined by the ash being white. However, spices would not be spices if there were not exceptions. Nutmeg, mace, ginger and cloves require $600 \pm 25^\circ$ as the ashing temperature.

The water-insoluble ash does not present any problem, once the total ash is determined. It is mentioned as a requirement in the standards for ginger, nutmeg

and mace. More importance was and is attached to the acid-insoluble ash, as this measures the presence of sand and comparable contaminants, a measure which is of specific importance in ground spices. The determination, again, would not present any problem, if it were not for the concentration of hydrochloric acid to be used. In ISO standards for a number of commodities the hydrochloric acid content for the determination of acid-insoluble ash varied considerably from product to product. The spices subcommittee accepted the same dilution of the acid as did tea committee.

One of the more important determinations is that of moisture content. In practice two methods are available. One is drying at a fixed temperature, the other is entrainment. The first method is not applicable in the presence of volatiles other than water, such as essential oils. As this is the case with a number of spices, oven-drying is seldom seen in standards. Saffron is one of them. In a spice like paprika, where no essential oil content of any importance is present the oven-drying technique is nevertheless not applicable due to the presence of sugars. The oven temperature would lead to caramelisation.

From the foregoing it is understandable that the entrainment method is to be preferred for most spices. This involves azeotropic distillation of the water with a codistillant, such as benzene or toluene.

Generally the method is standardized on toluene and the apparatus to be used is also standard. Problems may arise in the case of high sugar contents. For instance, using different entrainment liquids meant that the moisture content of onion powder depended on the boiling point of the entraining liquid due to the decomposition of sugars present. Such decomposition is particularly important in the case of fructose. Heavy stirring during the azeotropic distillation by means of a magnetic stirrer reduces the decomposition as well as the caramelisation. It may be pointed out that the determination of moisture content is important because it provides a base for calculations, the dry matter, and helps to prevent fraud. One of its principal aims is to avoid moisture contents that would enable moulds and other microbiological contaminants to flourish.

The next step toward better quality control and standardisation was the use of analytical methods that gave indications for processing yields and by this, indicated also the quality and value of the spice or herb. In the meantime spices had departed from their purely household or pharmaceutical applications. The preparation of essential oils, oleoresins, extracts, and tinctures became important as it made possible more rational applications. Rational in the sense of better application forms, such as better solubility and easier handling, in particular with regard to dosage. Rational

above all with regard to better standardisation of the derived product, leading to products of more homogeneous strength.

For analytical determinations it is necessary to prepare a test sample of the spice by grinding. For most analytical purposes a particle size of about 1 mm is sufficient. The grinding of the sample, however, presents the analyst with the problem of the breakdown of the structure of the plant materials, which in combination with the heat developed by the grinding process, causes losses of volatile components. Also, air-oxidation of sensitive components may occur. The Dutch standardisation of this operation reduces these losses considerably by adding an equal amount of diatomaceous earth before grinding, which absorbs the volatile compounds.

Related to this problem are the determination of fineness of grinding and the need to have different standards for whole and ground spices. With regard to the latter problem, examples are the essential oil content of whole and ground cinnamon Sri Lanka, namely 1.0 and 0.7 ml/100 g respectively, and for cloves grade 1 the same figures are 17 and 16 ml/100 g respectively.

As far as the determination of the fineness of grinding is concerned, this test is important in those cases where a limit is set in the standard for ground spices. For instance, for ground chillies min. 95% has to pass a sieve of 500 μ m aperture. For dehydrated garlic the grade indications: "powder," "grit," and "flakes" are based on sieve fractions.

Although the sieves themselves are standardised, carrying out the sieving operation in a reproducible way is difficult. The reference method for spices is the hand-sieving method, whereas machine sieving may be used as a routine. Hand sieving uses shaking and tapping in a special rhythm. Reproducible sieving is not the only problem. Some spices are friable and may become reduced in size during the sieving. Moisture content plays a role. The lower the moisture content the more friable the spice. Ground spices, on the other hand, have a tendency for cohesion leading to *increased* particle size. This tendency increases with volatile oil or fatty oil content, sugar content, and surface moisture. The duration of the sieving operation, and also its results, can be affected considerably by the particle shape. Fibrous, spherical, flaky shapes exert important influence. Reproducible sieving in this way requires real expertise from the analyst. He or she has to be aware that the ground material may absorb or lose moisture readily, depending on the relative humidity of the atmosphere.

Assuming that the test sample has been ground correctly or that the sample to be examined is a ground spice, a number of yield determinations may be carried out. One of the most important yield determinations in most herbs and spices is determination of the essential oil content. The method has been the subject of many discussions in a special working group of ISO's subcommittee on spices, as well as in the Council of Europe's working party on the same subject for the European pharmacopoeia. After many

trials the ISO working group rejected the AOAC and Clevenger apparatus. In the end the Reckitt and Coleman apparatus, the apparatus of the British Pharmacopoeia, and that of the European one, which may be regarded as a hybridisation of the British and Dutch Pharmacopoeial instruments, were compared. The result was rejection of the Reckitt and Coleman version due to consistent inferior results and the final acceptance of the European pharmacopoeial distillation apparatus. Nevertheless there still remain a set of critical factors to be considered, such as the amount of sample, the volume of water to be used, and the duration of the distillation. These factors have to be introduced in the relevant standards when requiring minimum essential oil contents.

One aspect of this determination is that most essential oil components are lighter than water. However, in a number of these oils components heavier than water also occur. To overcome this difficulty xylene is used as a collecting medium in the distillation trap. Since the water circulates in this distillation, it becomes saturated with xylene. Moreover, the volume of the space of the apparatus will contain xylene vapour. These are reasons why it is advisable to distill the xylene and the water first before adding the spice sample for the test itself.

The other yield determination methods are extraction methods. They determine the amount of material extracted from the spice by a specified solvent. The choice of solvent depends on the spice. Cold water extraction is required, for example, in saffron and in ginger. The ginger standard also mentions alcohol soluble extraction. Nonvolatile ether extraction is requested for, among others, caraway, mustard seed, and chillies.

The alcohol soluble and cold water soluble extracts are made by extraction at room temperature, evaporation of the solvent and finally drying of the evaporation residue at 103°C.

The nonvolatile ether extract is obtained by Soxhlet extraction, hence at 35°C, distilling off the ether and drying at 110°C. One of the difficulties encountered here was the presence in the Soxhlet extract of components that were not soluble in ether at room temperature. For this reason the extract obtained by Soxhlet extraction has to be shaken with ether after drying. After evaporation of the solvent the residue is dried again at 110°C.

One of the critical factors in these extraction methods is the difficulty of determining the endpoint of the drying of the extract, due to the presence of volatile components such as the essential oils. Are all the volatiles included or are they removed as in the nonvolatile ether extract? The difference is drying temperatures between alcoholic and cold water extractions on one hand (103°) and the nonvolatile ether method (110°) seems to suggest this. Nevertheless, in the determination of moisture by the drying oven method a temperature of 103°C is used. The result is usually indicated as comprising water as well as some volatiles

In case extraction solvents such as pentane or ether

are used, which are relatively more volatile than most essential oil compounds, the difficulty of endpoint determination can be solved graphically by determining the weight losses at regular time intervals under controlled conditions. This technique, however, is time-consuming and requires much attention.

Although all the quality control methods discussed so far are related to the trade and industrial use of spices, they have one thing in common—their nonspecificity. In fact they do not tell us anything about those characteristics which make a product be a spice. They do not measure any of those specific properties to which the special flavour of spices might be attributed. Although the determination of the essential oil content of a spice was a step in this direction, it was not sufficiently specific.

The same holds for the measuring of pungency by the Scoville index. In this method dilutions of an ethanolic extract solution in sucrose syrup are prepared and the most dilute of these solutions at which the pungent sensation just can be detected is determined. The subjectivity of the method and its dependence on personal sensitivity are evident. Nevertheless, it has served a useful purpose, for lack of a better method. This is true especially with capsicums and chillies.

To be able to carry out more specific determinations, knowledge must be available about those chemical substances that are responsible for the typical flavour of a spice; subsequently methods must be available for their measurement. Phytochemistry has isolated and identified many substances, mostly in the volatiles. New analytical tools have made possible the quantitation of a number of these compounds. Spectrophotometric techniques, sometimes combined with thin layer chromatography, gas chromatography, and high pressure liquid chromatography (now developing rapidly into a technique well-suited for non-volatiles) have made progress possible in this part of the field and are already becoming incorporated in quality control and in standards. The vanillin content of vanilla products and the piperine content in pepper oleoresin are examples of such more specific properties.

It would, however, be unrealistic not to point out that a spice's action cannot be attributed to only one such compound. For instance, the piperine content of a pepper product cannot solely account for the complete pungency sensation of that product. It usually is a combination of compounds, many times closely related, which is responsible for this.

Although such a complete picture would be desirable, the determination of the main, more specific compound is nevertheless a big step forward. Speaking of a complete picture, a pictorial insight into the composition of the spice may be obtained by thin layer chromatography. Formerly the action of spices was attributed to essential oils. If this was not sufficient to account for a spice's flavour the nonvolatile resinous matter was suggested, without any further detail. Some more details in these matters can be made visible by TLC. The state of knowledge about

the most often-used drugs, including spices and their thin layer fingerprints, is to be found in Stahl's book on the microscopic and chromatographic analysis of drugs.

In the Netherlands a standard for a general thin layer chromatographic method has been issued, together with a proposal for stabilised R_F values, based on the work of Dhont and coworkers at CIVO. The concept of stabilised R_F values is rather important, as it makes the thin layer chromatograms obtained in different laboratories comparable.

Apart from the well-known critical factors of TLC in general, collaborative experience has shown that the amount of sample applied is important, as otherwise spots that should be visible may get lost.

One of the best known applications of thin layer chromatography is that to vanilla products. The presence of anisic aldehyde shows the product to be derived from *Vanilla tahitensis*, that of piperonal its derivation from *Vanilla pompona*. Both constituents fail in the most common type: *Vanilla fragrans*. Moreover, TLC allows us to detect fraud. Certain components have to be present, such as para-hydroxybenzaldehyde. On the other hand, the presence of ethylvanillin, an artificial flavouring substance, is easily detectable. The latter has proved a problem in the case of vanilla extracts. In these extracts, if they are alcoholic extracts or in case they should contain poly-

valent alcohols such as glycerol, acetals are formed, which in the thin layer chromatogram give a spot at the same place as ethyl vanillin does. This confusion can be avoided by adding acid in the extraction step in order to destroy these acetals. The same phenomenon could be demonstrated also by gas chromatography. In alcoholic solutions of vanillin, or in solutions in glycerol, an extra peak occurs on standing that could be identified as the acetal.

Another interesting example of the application of TLC is in dehydrated mint. This herb may be contaminated with *Mentha* species or *Mentha* hybrids containing relatively high amounts of carvone, which may spoil the flavour. The presence of this compound is easily detected by TLC.

Saffron is a very expensive spice and for that reason subject to much fraud. Apart from its characteristic flavour, saffron also has colouring properties and the chromatography of a saffron extract may give indications on the purity of the spice on the basis of the presence or absence of the saffron pigment spots, of which crocin is one of the most important.

The presence of colouring components in some spices, together with their amount, is of great interest in determining the quality of such spices. The presence of crocin compounds in saffron has already been mentioned. Others of importance are curcumin in turmeric and capsanthin and other carotenoids in chillies and capsicum.

The method of choice for determining the colour strength of these spices is the spectrophotometric method. As applied in these cases in the visible region and by measuring at one wave length only, the method is not specific and purity has to be checked by a chromatographic technique. Difficulties are not to be expected by this kind of determination; the fact that in some cases the specific extinction coefficients are not known can be avoided by giving extinction coefficients $E1\% 1\text{ cm}$.

In a number of cases spectrophotometry has been used also to determine what might be called the active principles of spices. A few wellknown cases may be put forward; they include vanillin in vanilla, piperine in pepper, and capsaicin in capsicum species. Measurement here is mostly in the ultraviolet region of the spectrum.

The standard method for determining vanillin in vanilla products is based on a base-line technique that eliminates background absorption. Variants on this technique are those methods in which vanillin is isolated chromatographically and then measured spectrophotometrically. This isolation may be carried out by thin layer chromatography. The vanillin band is then either scraped off and extracted or, by using aluminum foil plates, the band is cut off and extracted.

With piperine in pepper and pepper oleoresins the pungent principles of spices enter the picture. Apart from piperine, there is capsaicin, and a number of others amongst which are the sharp principles of ginger. All have in common that the pungency of the spice involved is not due to one, sole, compound. In

most cases, a complex of compounds is responsible, of which compounds that mentioned earlier may be the most prominent one. In the case of ginger two components are of interest, gingerol and shogaol; both very pungent and both consisting of the reaction product of the also pungent ketone zingerone, with heptanal in the case of gingerol or with hexanal in that of shogaol. The quantitative determination of these sharp principles in ginger is still a problem to be solved.

The determination of the pungent principles of pepper has a long history. One of the earlier methods (ASTA) was based on splitting off formaldehyde, which subsequently was determined with chromotropic acid. Another early method was a Dutch one where alkaline hydrolysis was applied, the resulting volatile amines were distilled off and determined by titration. Both methods have in common that they are unspecific. The more modern methods are based on spectrophotometry. One, proposed by the U.K., used acetone for extraction, followed by dissolving of the extract in ethanol, whereas a second one, proposed by the Dutch and based on an American study, employed ethanol only.

The four methods were subjected to a collaborative study in France. The ASTA and the hydrolysis method gave inconsistent results. Of the spectrophotometric methods the Dutch-American one turned out to be the best, although its results also were the lowest. It was shown also that the most critical factor in this determination was that the extraction and all following steps had to be carried out in the dark, as otherwise piperine is isomerised. This is absolutely vital.

Another point which many spectrophotometric determinations have in common is the problem of the exact extinction coefficient of the compound to be measured. For piperine this has long been a matter of discussion. In about 130 determinations the Dutch standardisation committee arrived at a value of 1238 ± 40 at 343 nm. Such values are of commercial importance, as they are converted into percentages of the relevant compounds in the spices which are better understood by the trade. In such cases it might make sense to present in the test result also the E1% 1 cm value. In the case of piperine it was further of interest that from the spectrum recorded, it also was possible to estimate the amount of piperettine present.

The determination of capsaicin in capsicums and their oleoresins is a problem which has not yet been solved completely, although much time and effort has been devoted to this. From the point of view of quality control and standardisation the Kubeka Jurenitsch method is under test in the Netherlands. These methods use a first separation by thin layer chromatography, followed by a gas chromatographic determination of the derivatised capsinoids. The application of gas chromatography improves the specificity of the determination considerably, as it enables one to estimate the proportion of the different capsinoids present in addition to capsaicin itself.

Especially of interest is the possibility of estimating

the proportion of nonanoylvanillylamide present, because sometimes the synthesised compound is added, in addition to the amount present naturally. This, if not declared properly, constitutes fraud. In principle it would be possible to demonstrate such an addition by the C 12/C 13 method, as in the case of synthetic vanillin added to vanilla products. This method was developed by Bridoux to prove the presence of synthetic ethanol in fermentation alcohol, and was shown to be applicable to vanillin also. However, the necessary basic data are not yet available for nonanoylvanillylamide. To solve the problem of the determination of nonvolatile characteristics, such as capsaicin or piperine, derivatisation followed by gas chromatography may not be the optimal method to be used. Better solutions might be found by studying the possibilities of the application of high pressure liquid chromatography.

Another group of highly interesting and characteristic substances, which may serve for standardisation of spices, are the sulphur compounds such as the isothiocyanates in mustard seeds and the organic sulphides in onions and garlic. Of interest for mustard seed evaluation are the determination of allyl isothiocyanate in the Brassica mustards (black and Indian mustard) and of p-hydroxybenzyl isothiocyanate in Sinapsis (white) mustard.

The problem with the determination of both

isothiocyanates is that they occur in the plant as glucosides, namely sinigrin and sinalbin respectively. These have to be split by enzymatic action in the first step of the test procedure. Usually the enzyme activity of the seed itself is sufficient. However, in some older samples this might not be so, at least not in black mustard. In that case low results are obtained and enzymes have to be added. In most cases white mustard free of sulphur-containing volatiles will be used for this purpose. After the enzymatic reaction the allyl isothiocyanate is steam distilled into alcoholic ammonia solution and the isothiocyanate ion is determined argentometrically. For the white mustard determination of p-hydroxybenzyl isothiocyanate consists also of an enzymatic reaction, however, followed by hydrolysis of the ester set free and determination of the isothiocyanate either argentometrically or colorimetrically.

The organic sulfides, such as diallyl disulfide and allylpropyldisulfide in garlic follow the same pattern in their determination. Enzymatic decomposition of the alliin present into the unstable alliin in the steam distillation following on the enzymatic reaction breaks down into diallyl disulfide and other sulfides. The sulfides are determined argentometrically in the distillate. Although the method is an improvement, a more specific method would be desirable. Gas chromatography might be the answer to this problem.

As stated before, the volatile constituents of spices, their essential oils, constitute a very important factor for their appreciation, next to the nonvolatile aromatic components. The complexity of essential oils is well known. The method of choice for their evaluation is gas chromatography. This analytical tool has been well described and is standardised. It does not seem necessary to spend much time here on it. One example from the spice field may be used here to show some aspects which deserve attention. Oil of clove buds contains as the most important constituent eugenol; eugenyl acetate is also present, as are caryophyllenes. Naves has shown that caryophyllene is not present as such in the clove buds. It is formed, in the course of the distillation, from epoxy-dihydrocaryophyllene.

The value of oil of clove buds as a spice essential oil, however, is not determined only by the main compounds mentioned; trace compounds like methyl amyl ketone are responsible for the peculiar by-odor of the bud oil. Indirectly, these trace components also determine the value of the spice itself. The question may be put forward whether they are present originally or due to fermentation during drying of the buds. If the oil is to be used for industrial purposes as a raw material it is the total eugenol content, the sum of bound and free eugenol, which is of interest.

Although many essential oils have bacteriostatic and fungistatic properties, and hence microbiological contamination of spices might seem less of a problem, especially if attention is paid to their moisture content, microbiological examinations have shown the importance of this aspect of quality control. Spices such as paprika powder and onion and garlic powder

are notorious for their microbiological contamination. Since gas treatment by ethylene oxide is prohibited more and more often, irradiation-sterilisation has become an interesting alternative for sterilisation purposes. Although methods exist for the detection of ethylene oxide residues and of its breakdown products, such as ethylenechlorohydrin, the detection of irradiation, if not declared on the label, is still an unsolved problem.

The mention of residues brings up the aspect of legal standards for spices. Where such regulations exist they usually follow the existing trade practice closely. This situation changes when their derived products are taken into consideration. From the regulatory point of view two aspects require attention here.

The first aspect is that of undesirable components in the derived products, which are naturally present in the spice itself. Examples of such components are thujone and safrole. Thujone occurs, among others, in sage, which is widely used in food flavors. Safrole is a constituent of nutmeg and mace. Their determination does not present any technical problem. Steam distillation, extraction of the distillate, concentration of the extract, and gas chromatography with an internal standard is the method of choice. Safrole presents a problem in that the reference solution for the preparation of the calibration curve is not stable. Both, however, present a rather general problem. This is the availability of the reference materials, such as pure thujone, pure safrole, and pure capsaicin, required for the preparation of standard curves or calibration graphs. This is one of the main problems in quality control and standardisation of analytical methods for spices.

The second aspect is also one of undesirable compounds in derived products, however, not one of compounds naturally present. What is meant here is the problem of the determination of solvent residues in spice extracts and oleoresins. Although receiving attention for many years, the perfection of gas chromatographic techniques has brought us nearer to the solution of these problems. At present the method of choice is the gas chromatographic headspace technique. The main drawbacks are interference by low boiling volatile spice constituents and the matrix influence of the substrate. The first drawback, that of the low boiling volatiles, may be overcome by selection of the appropriate column packing such as Porapak, or (for example for benzene), Chromosorb 102. The use of specific detectors, for instance in the case of halogenated solvents, may be of assistance. The matrix influence, which is very important in this kind of determination, based on the equilibrium between phases, may be eliminated by dilution with an equal amount of diethyl phthalate.

Opening Pandora's box is always a surprise. Some of Pandora's surprises, however, by their very nature, have been shown to be highly interesting problems. The cooperation of science with the flavor trade and industry may lead to their solution. Let us hope this will be to the satisfaction of every one involved.