A Structured Approach toward Standardized Specifications for the Flavor Industry

A structured approach to improving efficiencies, quality and safety

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The process of establishing global specifications for flavoring raw materials and flavorings faces three basic challenges: the enormous range of chemical classes to which the compounds belong, the complexity of their physical and chemical properties, and last but not least, the history of the individual local and regional regulatory approaches. The modern food industry, however, is characterized by global development projects and cross-regional technology transfers. In this scenario, standardization is a task that benefits everyone in one way or another. In an economic context, standards contribute to the development of the free market and permit the free trade of goods and services, minimizing additional modification costs.[1] Standardization organizations support specific industries by the provision of standards in this respect. For instance, the International Standards Organization (ISO) Technical Committee 54 issues standards for essential oils with commercial relevance and thereby supports the flavor industry.*

The portfolios of all flavor companies comprise a huge variety of ingredients in the form of both raw materials and finished flavors. All of these materials need to be defined by certain parameters and respective limits. These definitions of materials are called "specifications." Specifications set the standards for quality control, allow free trading and assure compliance with regulatory attributes. The requirements and specifications for flavorings and their raw materials are mainly derived from food legislation. Specifications for flavoring raw materials are laid down in the Codex Alimentarius and by the Joint FAO/WHO Expert Committee on Food Additives (JECFA).** For some flavoring raw materials, distinct organizations issue specifications, e.g. many essential oils are specified in ISO by Technical Committee 54.

Companies in the flavor industry interact with each other on three levels simultaneously—as customers, suppliers and competitors. Each company creates specifications to support an agreement on product qualities for shipments and for the trading of goods. The industry expends much effort to create and maintain specifications and simultaneously generates huge internal workloads that tend to be counterproductive in terms of facilitation of trade. This paper suggests a structured approach to achieve standardized specifications for flavorings and their respective raw materials. Carriers, additives and other materials produced in the industry, such as fragrances, are not in the focus of this paper, but a comparable structured approach is also practiced in the authors' company. The classification of materials, the selection of test methods and a decision tree (**F-1; Pages 38–39**) for a systematic assignment of specifications are the key elements to this approach.[2]

Definitions

According to the Codex Alimentarius definition, flavorings are:

> *"… products that are added to food to impart, modify, or enhance the flavor of food (with the exception of flavor enhancers considered as food additives under the Codex Class Names and the International Numbering System for Food Additives – CAC/GL 36-1989). Flavorings do not include substances that have an exclusively sweet, sour, or salty taste (e.g. sugar, vinegar, and table salt)."*[3]

In the International Organization of the Flavor Industry (IOFI) Code of Practice, the term "flavoring" is defined as:

> *"Concentrated preparations, with or without flavor adjuncts, used to impart flavor, with the exception of only salty, sweet or acidic tastes. They are not intended to be consumed as such."*

At a Glance

Global specifications for flavoring raw materials and flavorings represent a major challenge for all involved parties, including manufacturers, suppliers, flavorists, regulators and traders. The main objective of the work presented here is the standardization of analytical, microbiological and sensorial test scopes on a global basis. This publication describes a decision tree-based approach (**see F-1; Pages 38–39**) to such globally standardized specifications for the flavor industry.

*www.iso.org/iso/en/stdsdevelopment/tc/tclist/TechnicalCommitteeDetailPage.TechnicalCommittee 54, COMMID=1855

**www.codexalimentarius.net/web/index_en.jsp; www.fao.org/ag/agn/jecfa-flav/search.html?lang=en

In the context of this paper, "flavoring raw materials" shall mean all ingredients in solid, liquid or pastelike form, which are used to formulate flavorings.

In the Codex Alimentarius, the definition of a food additive is:

> *"Food additive means any substance not normally consumed as a food by itself and not normally used as a typical ingredient of the food, whether or not it has nutritive value, the intentional addition of which to food for a technological (including organoleptic) purpose in the manufacture, processing, preparation, treatment, packing, packaging, transport or holding of such food results, or may be reasonably expected to result (directly or indirectly), in it or its by-products becoming a component of or otherwise affecting the characteristics of such foods. The term does not include contaminants or substances added to food for maintaining or improving nutritional qualities."*[3]

The term "flavor adjuncts" is defined by the IOFI Code of Practice as:

> *"Food additives and food ingredients necessary for the production, storage and application of flavorings as far as they are nonfunctional in the finished food."*

Food additives are not in the scope of this paper. However, just like flavorings and flavoring raw materials, food additives and adjuncts must have specifications.

Maintaining Quality and Safety

Quality considerations are important for the reliable creation and production of flavorings and flavoring raw materials. Meanwhile, the product safety aspect has received more and more public attention in the wake of such public health scares as those involving food contaminants. These two aspects address the microbiological properties of raw materials and flavorings, i.e. low rates of total plate count, or the absence of pathogens, are indicators for the hygiene standard in the production of raw materials (including harvesting) and flavorings. Reliable production steps, such as pasteurization, are also required to guarantee a high quality level in production.[4] This influences the selection of the microbiological methods and the structure of the decision tree.

For specific applications, scale-ups, innovative techniques, new creative raw materials and in the case of unusual findings, an individual evaluation by a specialist is required to allocate the suitable parameters and limits. The test scope should cover the necessary information for quality control purposes suited for the routine testing of materials. Additional aspects, such as tests to obtain nutritional information or figures needed for transportation, are not considered to be part of a test plan. These data can be determined once, and then again occasionally when there is evidence that there is a compositional change.

One aspect that also contributes to safety is the risk originating from contaminants. The Codex Alimentarius defines a contaminant as follows:

> *"Any substance not intentionally added to food, which is present in such food as a result of the production (including operations carried out in crop husbandry, animal husbandry and veterinary medicine), manufacture, processing, preparation, treatment, packing, packaging, transport or holding of such food or as a result of environmental contamination. The term does not include insect fragments, rodent hairs and other extraneous matter."*[3]

In a wider context, contaminants can be seen as any undesirable compound or group of compounds possibly occurring in a flavoring raw material at trace levels. Based on this definition, the contaminant risk assessment also includes agricultural residues. The assessment of the risk of a contaminant can be carried out by considering the class of flavoring raw materials in relation to the contaminant class, using the principles of a failure mode and effect analysis (FMEA), as described in EN 60812.[5] A special risk assessment matrix considering the materials used in the flavor industry and the contaminants concerned is applied by the authors to assess the risk of a contaminant for a group of raw materials. The risk assessment is derived from the multiplicative combination of the severity of a contaminant, its detectability and probability of its occurrence. The calculated risk numbers are linked to action levels to allow for adequate measures. The risk assessment has to be updated regularly with respect to recent incident reports, as well as state-of-the-art analytical possibilities.

Structure and Generation of Specifications

A specification can technically be set up by the use of three building blocks: header, a set of parameters, and respective limits or targets and additional information. Header information is very important to describe the subject by a common name, but also by giving information such as synonyms, CAS number and inventory numbers. The set of parameters and limits build the core of the specification stating the physical, chemical, sensory and microbiological properties of the material concerned.

The generation of a specification is the process of selecting suitable parameters and assigning reasonable limits or target values. This process can be structured in three steps:

- Classification of the raw material
- Linkage of the raw material or raw material class to a test scope
- Application of limits or target values to the raw material

The test methods applied should provide a maximum of information for a minimum of effort. The selection of test methods is called the test scope. The appropriate test scope has to be selected to cover the following aspects:

- Sensory properties (e.g. odor, taste, color, clarity, physical aspect)
- Microbiological properties (e.g. total plate count, yeasts and molds, coliforms, *E. coli*)
- Identity (e.g. specific gravity, refractive index, IR spectroscopy)
- Purity (GC or HPLC)

- Stability/degradation (e.g. peroxide value, acid value)
- Authenticity (e.g. optical rotation, isotope ratio mass spectrometry, deuterium NMR, HPLC)
- Contaminants (specific trace analysis for compounds such as solvents or pesticides)
- Safety aspects (e.g. *Bacillus cereus*)
- Regulatory aspects (admissibility in different regions, etc.)

From Test Scope to Specification

Once the test scope is defined for a class of materials, the limits and targets have to be set for each individual material. Any test method has its limitations with regard to accuracy, precision, limit of quantification, linearity, reproducibility and specificity. Many analytical methods can be described by these attributes, whereas microbiological and sensory methods cannot be validated in the same way. Reasonable limits can be obtained by the review of existing data or based on material that represents the desired quality. Tolerances should be chosen with respect to the inherent variation and uncertainty of the test methods. Ideally the tolerances of an analytical parameter should either not fall below six times the standard deviation of the method, or the method capability index should be greater than 1.33.[6] A method capability index of ≥ 1 as suggested for instance by Jenny means that the whole specification range is consumed by the analytical

Tests for flavorings **T-1**

Test	Unit	Target	Tolerances/limits	Decimal places; specification/ COA
Relative density (specific gravity)	-	Mean value, rounded to third decimal place	± 0.01	4*
Refractive index	-	Mean value, rounded to third decimal place	± 0.01	4*
Color value L*, a*, b*	-	Minimum/maximum value, rounded to integer number	± 5 only if slight variation, individual setting as alternative	2
pH value	-	Mean value	± 0.5	1
Ethanol	% Vol.	Mean value	± 2	1
Water content, low (< 4%)	%	Mean value	max. 5	1
Water content, medium (≥ 4% to < 10%)	%	Mean value, rounded to nearest 0.5	maximum value = mean value + 2	1
Water content, high (≥ 10%)	%	Mean value, rounded to nearest 0.5	± 2	1
Sodium chloride content, medium (< 10%)	%	Mean value rounded to nearest 0.5	maximum value = mean value ± 2	1
Sodium chloride content, high (≥ 10%)	%	Mean value rounded to nearest 0.5	± 3	1
Brix	°Brix	Mean value, rounded to nearest 0.5	± 3	1
Turbidity	FNU (NTU)	Minimum/maximum value	Individual setting	1
Particle size distribution	-	Minimum/maximum value	Individual setting	1
Total plate count	cfu/g	Maximum value	Individual setting	0
Yeasts and molds	cfu/g	Maximum value	Individual setting	0
Coliforms	cfu/g	Maximum value	Individual setting	0
E. coli	cfu/g	Maximum value	Individual setting	0
Coagulase positive *Staphylococci*	cfu/g	Maximum value	Individual setting	0
Sulfite reducing *Clostridia*	cfu/g	Maximum value	Individual setting	0
Bacillus cereus	cfu/g	Maximum value	Individual setting	0
Salmonella	negative/x g (e.g. 25 g)	Maximum value per defined mass of sample	Individual setting	0
Clostridium perfringens	cfu/g	Maximum value	Individual setting	0
Acid-tolerant bacteria	cfu/g or cfu/mL	Maximum value	Individual setting	0
Acid-tolerant yeasts and molds	cfu/g or cfu/mL	Maximum value	Individual setting	0

*can be reduced to 3 decimal places on the specification

Selection of associations and programs on analytical and microbiological methods **T-2**

Association	Regional orientation	Raw material group
International Standard Organization (ISO) Technical Committee 54	International	Essential oils
American Spice Trade Association (ASTA)	United States	Spices and oleoresins
International Organization of the Flavor Industry (IOFI)	International	Flavorings
Association of Official Analytical Chemists (AOAC)	United States	Food and many other categories of materials; one chapter, "flavors," dealing with individual tests
Leatherhead Food International	United Kingdom	Food
Deutsche Gesellschaft für Hygiene und Mikrobiologie (DGHM)	Germany	Food
International Federation of Fruit Juice Producers (IFU)	International	Fruit juices
Food Chemical Codex (FCC)	United States	Food chemicals

variability which leaves no margin for the manufacturing process.[7] Therefore, a method capability index of ≥ 1.33 is regarded as adequate. Otherwise, either the specification limit or the analytical procedure must be adjusted.

Limits

This section provides some advice on setting limits for flavorings. The corresponding raw materials exist in a huge number of different qualities; due to individual applications, it makes no sense to propose limits for them in this paper. For this we can refer to standards such as pharmacopoeias, which clearly identify qualities by defined limits for special purposes such as active ingredients or excipients in pharmaceuticals. As a consequence, the proposed limits should not be considered to be in competition with known standards (USP, Ph. Eur., JECFA, DGHM, etc.).

In the flavor industry, a particular target is to attain low microbiological results. Therefore the limits for these are always defined as upper limits, without lower limits. In the case of pathogens, the aim is for their absence in a defined amount of product (i.e. negative/25 g).

The limits in **T-1** have been applied for a long time by the authors and have been shown to be effective for all types of materials.

Methods

The methods that are applied to test a material should be defined to ensure reproducible and reliable results. Therefore, many analytical methods have been subject to standardization, primarily driven by the industry concerned. There is no distinct collection of methods that are designed for the flavor industry; however, there are associations linked to this industry which issue methods and work on their standardization (**see T-2**).

The methods recommended in this paper have been selected according to economic criteria and relevance to quality on the one hand, but also with regard to their global applicability. The focus is on methods that are suitable for routine application and which allow for automation. Nevertheless, sensory and microbiological methods—as well as some less economical methods—are indispensable for our industry to ensure high quality and safety. All methods that are mentioned in the **Decision Tree** are given in **T-3** as a guide to standard test scopes.

Decision Tree

The generation of a comprehensive test plan should be highly structured on a logical basis in order to sort materials according to their properties referenced in the corresponding test parameters. A strict performance of this procedure will give clear guidance for all involved parties. Every step in the decision tree (**F-1; Pages 38–39**) poses a different question. The corresponding answer leads to further steps until the basic group with the methods is reached. These methods are important in order to determine the quality of materials. The methods used have their origin in methods already published, as shown in **T-3**. Contaminants such as heavy metals, aflatoxins or pesticides are not considered in the decision tree because these tests are subject to detailed legal frameworks and have to be decided on by an individual risk assessment.

The following description will give a progressive guide to the lowest group until the test scope has been reached in order to demonstrate the functionality of this approach. The names of levels and groups are written in ***bold italics*** for a better orientation. They are numbered according to the level in the explanations below. Every connection point can also lead to the exit ***others***. This option is necessary because of the possibility that there will be some materials that do not fit the categories proposed.

For materials with flavoring application, the first distinction in the decision tree divides materials into ***1.1 flavoring raw materials*** and ***1.2 flavorings***. This needs to be done in order to handle the complexity on the following sublevels. In addition, this approach has the benefit of allowing a better control of the flavoring raw materials, which are the starting point for all flavorings. This concept is based on the fact that a fairly limited number of flavoring raw materials serve as starting materials for a huge number of flavorings.

On the third level, the materials are separated into ***1.1.1 liquid*** and ***1.1.2 solid***. The different physical properties require different handling in the laboratory,

Overview of selected methods for the decision tree approach **T-3**

Method	References	Objective
	Sensory	
Sensory, odor evaluation	ISO 5492 DIN 10954	Identity
Sensory, taste evaluation	DIN 10954 DIN 10973 DIN/ISO 4120	Identity
Visual appearance, color, clarity	ISO 11037 DIN 10954 Ph. Eur. 2.2.1	Identity
	Physical	
Relative density (specific gravity)	ISO 279 FCC (general test) DIN 51757 Ph. Eur. 2.2.5	Identity
Refractive index (Brix scale)	ISO/FDIS 280 FCC (general test) DIN 280 Ph. Eur. 2.2.6	Identity
Optical rotation	ISO/FDIS 592 FCC (general test) Ph. Eur. 2.2.7	Identity
Melting point	FCC (general test) Ph. Eur. 2.2.14	Identity, purity
Dry matter/loss on drying	ISO 4715 FCC (general test) Ph. Eur. 2.2.32 Ph. Eur. 2.8.9	Purity
Determination of color	ISO/WD DIN/ISO 6271 (Pt-Co value) DIN 53403 (iodine value) FCC (Lovibond value) Ph. Eur. 2.2.2	Identity
Hazen-/α-color index	DIN 53409	Identity
Extraction yield (Soxhlet method)	-	Purity
	Chemical	
Carbonyl content	ISO/DIS 1279 ISO 1271 FCC (General Test)	Purity
Salt content (argentometric titration, calculated as sodium chloride)	ISO 5943 (cheese) FCC (monograph sodium chloride)	Purity
Acid number/acid content	ISO/FDIS 1242 FCC (general test) Ph.Eur. 2.5.1 IFU, No. 3	Stability, purity
Essential oil content	ISO 6571 FCC (general test) Ph. Eur. 2.8.12	Purity
Water content	ISO 760 ISO/FDIS 11021 FCC (general test) Ph. Eur. 2.5.32	Identity, purity

T-3 (cont.)

Method	References	Objective
Sulfate ash	FCC (general test) Ph. Eur. 2.4.14	Purity
Gas chromatography	ISO 7609 ISO 7359 ISO/FDIS 11024 FCC (general test) IOFI recommended method 18 Ph. Eur. 2.2.28	Identity, purity
Solvent residues by gas chromatography	ISO/FDIS 14714 FCC (general test) Ph. Eur. 2.4.24	Contaminants
UV/vis spectroscopy	ISO/FDIS 4735 ISO 5566 FCC (citrus oils) Ph. Eur. 2.2.25	Identity, purity
pH value	FCC (general test) Ph. Eur. 2.2.3	Identity
Peroxide value	ISO 3960 FCC (general test) AOCS CD 8b-90 FMA Ph. Eur. 2.5.5	Stability
Infrared spectroscopy	FCC (4, infrared spectra) Ph. Eur. 2.2.24	Identity
Turbidity	ISO 7027 (turbidity in water FNU) EPA 5.5 (NTU) Ph. Eur. 2.2.1	Property
Particle-size distribution of emulsions	Ph. Eur. 2.9.31	Property
Ethanol content by HPLC with RI detection	Ph. Eur. 2.2.29 (general test) IOFI recommended method 29 (1995)	Identity, purity
HPLC for the quantification of various analytes e.g. vanillin, vanillic acid, capsaicin, piperine, coumarin, safrole, benzaldehyde, glycyrrhizic acid, etc.	ISO 8432 (general test) ISO 7358 (bergaptene) Ph. Eur. 2.2.29 (general test) Ph. Eur. 1859 (monograph capsicum)	Identity, purity
	Microbiological	
Total plate count	ISO 4833	Property
Yeasts and molds	ISO 7954	Property
Coliforms	ISO 16649-2	Property
E. coli	ISO 16649-2	Property
Coagulase positive *Staphylococci*	ISO 6888-1 LFGB 00.00.55	Safety
Sulfite reducing *Clostridia*	DIN 10103 LFGB 06.00.39 LFGB 59.00.4	Safety
Bacillus cereus	ISO 7932	
LFGB 00.00.25	Safety	
Salmonella	ISO 6579 (classical)	
LFGB 00.00.52 (PCR)	Safety	
Clostridium perfringens	ISO 7937	
LFGB 00.00.57	Safety	
Acid-tolerant bacteria	IFU, Microbiol. Methods No. 2 and 7	Property
Acid-tolerant yeasts and molds	IFU, Microbiol. Methods No. 2 and 7	Property

which is a direct consequence of the varying methods and analytical techniques, and also the sample preparation.

The next step forward in the decision tree, the fourth level, takes the different production technologies into consideration. That means that liquid flavoring raw materials can be grouped as ***1.1.1.1 distilled, cold pressed, essence*** **or** ***extracted***. In this row the only alternative is the exit to ***1.1.1.2 others***, which means that in our opinion there is no other group that is big enough to build up its own second category. In the direct line of the group ***1.1.1.2 others*** is a group ***1.1.1.2.1.1 juice*** **and** ***juice concentrates*** which can not be included in ***1.1.1.1***. This group is of utmost importance for beverages, so an individual category was created. The tests for this kind of product should include color and appearance, odor and taste, brix or dry matter, acid content, pH value, and UV/visible absorbance in the case of use as coloring material. The microbiological test scope should include testing for acid-tolerant bacteria and acid-tolerant yeasts and molds, due to the high water content and the presence of fruit acids.

Sorting materials according to the production technologies is not valid for the side of the decision tree dealing with liquid products, but it is important for the branches dealing with solid products of ***1.1.2*** and ***1.2.2***. For example, inorganic impurities such as drying aids (e.g. anhydrous potassium carbonate) that are soluble in small amounts can also crystallize in the crystallization process, or be included in the crystalline state of the material itself, but cannot be present in the distillate of a material. Thus, crystallized solid products need a more advanced test plan than distilled solid products. Further details will be explained in the next paragraph, which explains the section for solid products.

Going down vertically in the decision tree to the fifth level, the "natural" status of the material appears as the next criterion. This has a great impact on the application of the flavoring raw materials, the legal status of the resulting flavors, and the cost structure, which can be important (e.g. vanilla). The alternative to ***1.1.1.1.1 natural materials*** is ***1.1.1.1.2 nature identical/synthetic materials***, which are in general not as expensive. The term natural here is used in the sense of being directly obtained from plants, animals or from fermentation processes, according to European legislation.

Descending as an example to the lowest levels of a natural, distilled/cold pressed/essence/extract liquid flavoring raw material, we reach ***1.1.1.1.1.1 alcohols, ketones, ethers, thiols, heterocycles***, the test plan for which should include color, clarity and appearance, odor and taste and relative density, refractive index, optical rotation, and preferably GC for the determination of the purity. This group does not show degradation to acids, hence the acid value has limited relevance. The next basic group, ***1.1.1.1.1.2 esters, aldehydes***, should have a test scope of color, clarity and appearance, odor and taste and relative density, refractive index, optical rotation, color value, purity determined by GC analysis, and acid value. This very heterogeneous group has different functional groups from a chemical point of view, but the acid value does give good information with regard to degradation processes. This section is completed by ***1.1.1.1.1.4 terpenes***, ***1.1.1.1.1.3 carboxylic acids***, ***1.1.1.1.1.5 distilled citrus oils and terpene containing essential oils***, ***1.1.1.1.1.7 cold-pressed citrus oils***, ***1.1.1.1.1.8 extracts, aqueous extraction***, ***1.1.1.1.1.10 extracts, non-aqueous extraction***, ***1.1.1.1.1.9 ethanol-containing essences*** and ***1.1.1.1.1.11 others***. The details of the test scopes can be reviewed in the decision tree itself. Special interest should be given to the group ***1.1.1.1.1.6 essential oils*** due to their importance as raw materials in this industry. In addition to the tests for color and appearance, clarity, odor and taste, refractive index and relative density, optical rotation and color value, the composition determined by GC plays an important role. The main ingredients and their relation to each other provide information on authenticity and quality. Trace compounds can also indicate the origin and quality of the composition of an essential oil, and for that reason a single GC run gives much more information than a simple test for an assay of the main component.

The decision to form the group for natural liquid flavoring ***1.1.1.1.1.3 carboxylic acids*** derives from the fact that the purity is easier to determine by titration than by GC, which is preferred for the other groups. The optical rotation should be omitted if no asymmetrical carbon atom is included in the structure of the compound. ***1.1.1.1.1.4 terpenes*** are separated from the others because of their sensitivity towards oxidation, which can be detected by the peroxide value, the latter giving an indication of incorrect storage or handling of the product. These lowest groups were chosen not only on account of their chemical behavior, which reflects the chemical functional groups such as aldehydes or alcohols, or similar sensitivity towards degradation, but also due to the process details such as the separation of extracts obtained with water or organic solvents. Therefore, the test plan of ***1.1.1.1.1.8 extracts, aqueous extraction*** contains only appearance, color, odor, taste, brix or dry matter, and microbiology, and in the case of a coloring material UV/visible absorbance. The extraction solvent water permits growth of microorganisms and therefore an advanced testing of total plate count, yeasts and molds (quality indicators), coliforms (hygiene indicators), *E. coli*, and *Salmonella* are necessary to ensure microbiological quality and safety. The test plan for ***1.1.1.1.1.10 extracts, non-aqueous extraction*** includes additional determination of residual solvents and essential oil content. Due to the lack of water, microbiological testing is not necessary for this group. For many raw materials the optical rotation is a test that has proven to be an excellent indication of raw material origin, since many plants are characterized by the enrichment of individual enantiomers. More sophisticated methods such as isotope ratio mass spectrometry (IRMS) coupled to gas chromatography are not appropriate as a routine method for the great majority of natural materials.

1.1.1.1.2 nature-identical/synthetic materials is organized according to chemical functional groups that are also present and already described on the natural materials level, as in the groups ***1.1.1.1.2.1*** to ***1.1.1.1.2.5***. The test scope of the groups that are found under ***1.1.1.1.1 natural materials*** and ***1.1.1.1.2 nature-identical materials*** are identical, with the exception of optical rotation, which is not necessary for the latter groups since they are by definition not natural.

Decision tree for the standardization of analytical, microbiological and sensorial test scopes for flavors

1 MATERIALS WITH

1.1 flavoring raw materials

1.1.1 liquid

1.1.1.1 distilled, cold pressed, essence, extracted

1.1.1.1.1 natural materials

1.1.1.1.1.1 alcohols, ketones, ethers, thiols, heterocycles
appearance, color, clarity, odor, taste, relative density, refractive index, optical rotation, color value, purity by GC

1.1.1.1.1.2 esters, aldehydes
appearance, color, clarity, odor, taste, relative density, refractive index, optical rotation, color value, acid value, purity by GC

1.1.1.1.1.3 carboxylic acids
appearance, color, clarity, odor, taste, relative density, refractive index, optical rotation, color value, purity by acid value or GC

1.1.1.1.1.4 terpenes
appearance, color, clarity, odor, taste, relative density, refractive index, optical rotation, color value, peroxide value, purity by GC

1.1.1.1.1.5 dist. citrus oils and terpene containing essential oils
appearance, color, clarity, odor, taste, relative density, refractive index, optical rotation, color value, peroxide value, composition by GC

1.1.1.1.1.6 essential oils
appearance, color, clarity, odor, taste, relative density, refractive index, optical rotation, color value, composition by GC

1.1.1.1.1.7 cold pressed citrus oils
appearance color, clarity, odor, taste, relative density, refractive index, optical rotation, color value, carbonyl content, peroxide value, residue on evaporation, composition by GC

1.1.1.1.1.8 extracts, aqueous extraction
appearance, color, odor, taste, brix or dry matter product individual: substances by HPLC or UV/vis absorbance for coloring products, total plate count, coliforms, *E. coli*, yeasts/moulds, *Salmonella*

1.1.1.1.1.9 ethanol containing essences
appearance, color, clarity, odor, taste, relative density, refractive index, color value, ethanol content

1.1.1.1.1.10 extracts, non aqueous extraction
appearance, color, odor, taste, residual solvents, total plate count individual: essential oil content or substances by HPLC or GC or UV/vis absorbance for coloring materials

1.1.1.1.1.11 others
individual

1.1.1.1.2 nature identical/ synthetic materials

1.1.1.1.2.1 esters, aldehydes
appearance, color, clarity, odor, taste, relative density, refractive index, color value, acid value, purity by GC

1.1.1.1.2.2 alcohols, ketones, ethers, thiols, heterocycles
appearance, color, clarity, odor, taste, relative density, refractive index, color value, purity by GC

1.1.1.1.2.3 terpenes
appearance, color, clarity, odor, taste, relative density, refractive index, color value, peroxide value, purity by GC

1.1.1.1.2.4 carboxylic acids
appearance, color, clarity, odor, taste, relative density, refractive index, color value, purity by acid value or GC

1.1.1.1.2.5 others
individual

1.1.1.2 others

1.1.1.2.1 natural materials

1.1.1.2.1.1 juice, juice concentrates
appearance, color, odor, taste, dry matter or brix, acid content, pH value, UV/vis absorbance for coloring products, acid tolerant bacteria, acid tolerant yeasts/moulds

1.1.1.2.1.2 others
individual

1.1.1.2.2 nature identical/ synthetic materials

individual

1.1.2 solid

1.1.2.1 distilled

1.1.2.1.1 natural materials

1.1.2.1.1.1 esters, aldehydes
appearance, color, odor, taste, specific rotation, acid value, melting point or IR spectra, purity by GC

1.1.2.1.1.2 alcohols, ketones, ethers, thiols, heterocycles
appearance, color, odor, taste, specific rotation, melting point or IR spectra, purity by GC

1.1.2.1.1.3 carboxylic acids
appearance, color, odor, taste, specific rotation, melting point or IR spectra, purity by acid value or GC

1.1.2.1.1.4 others
individual

1.1.2.1.2 nature identical/ synthetic materials

1.1.2.1.2.1 esters, aldehydes
appearance, color, odor, taste, acid value, melting point or IR spectra, purity by GC

1.1.2.1.2.2 alcohols, ketones, ethers, thiols, heterocycles
appearance, color, odor, taste, melting point or IR spectra, purity by GC

1.1.2.1.2.3 carboxylic acids
appearance, color, odor, taste, melting point or IR spectra, purity by acid value or GC

1.1.2.1.2.4 others
individual

1.1.2.2 crystal-lized

1.1.2.2.1 natural materials

1.1.2.2.1.1 esters, aldehydes
appearance, color, odor, taste, specific rotation, acid value, sulfate ash, dry matter, melting point or IR spectra, purity by GC

1.1.2.2.1.2 alcohols, ketones, ethers, thiols, heterocycles
appearance, color, odor, taste, specific rotation, sulfate ash, dry matter, melting point or IR spectra, purity by GC

1.1.2.2.1.3 carboxylic acids
appearance, color, odor, taste, specific rotation, sulfate ash, dry matter, melting point or IR spectra, purity by acid value or GC

1.1.2.2.1.4 others
individual

1.1.2.2.2 nature identical/ synthetic materials

1.1.2.2.2.1 esters, aldehydes
appearance, color, odor, taste, acid value, sulfate ash, dry matter, melting point or IR spectra, purity by GC

1.1.2.2.2.2 alcohols, ketones, ethers, thiols, heterocycles
appearance, color, odor, taste, sulfate ash, dry matter, melting point or IR spectra, purity by GC

1.1.2.2.2.3 carboxylic acids
appearance, color, odor, taste, sulfate ash, dry matter, melting point or IR spectra, purity by acid value or GC

1.1.2.2.2.4 others
individual

1.1.2.3 others

1.1.2.3.1 natural materials

1.1.2.3.1.1 fruits
appearance, color, odor, taste, dry matter or brix, pH value, acid content, UV/vis absorbance for coloring materials

1.1.2.3.1.2 herbs, spices
appearance, color, odor, taste, total plate count, coliforms, *E. coli*, yeasts/moulds, *Salmonella*, coagulase positive Staphylococci, *Bacillus cereus*, sulphite reducing Clostridia, materials used for extraction = extraction yield

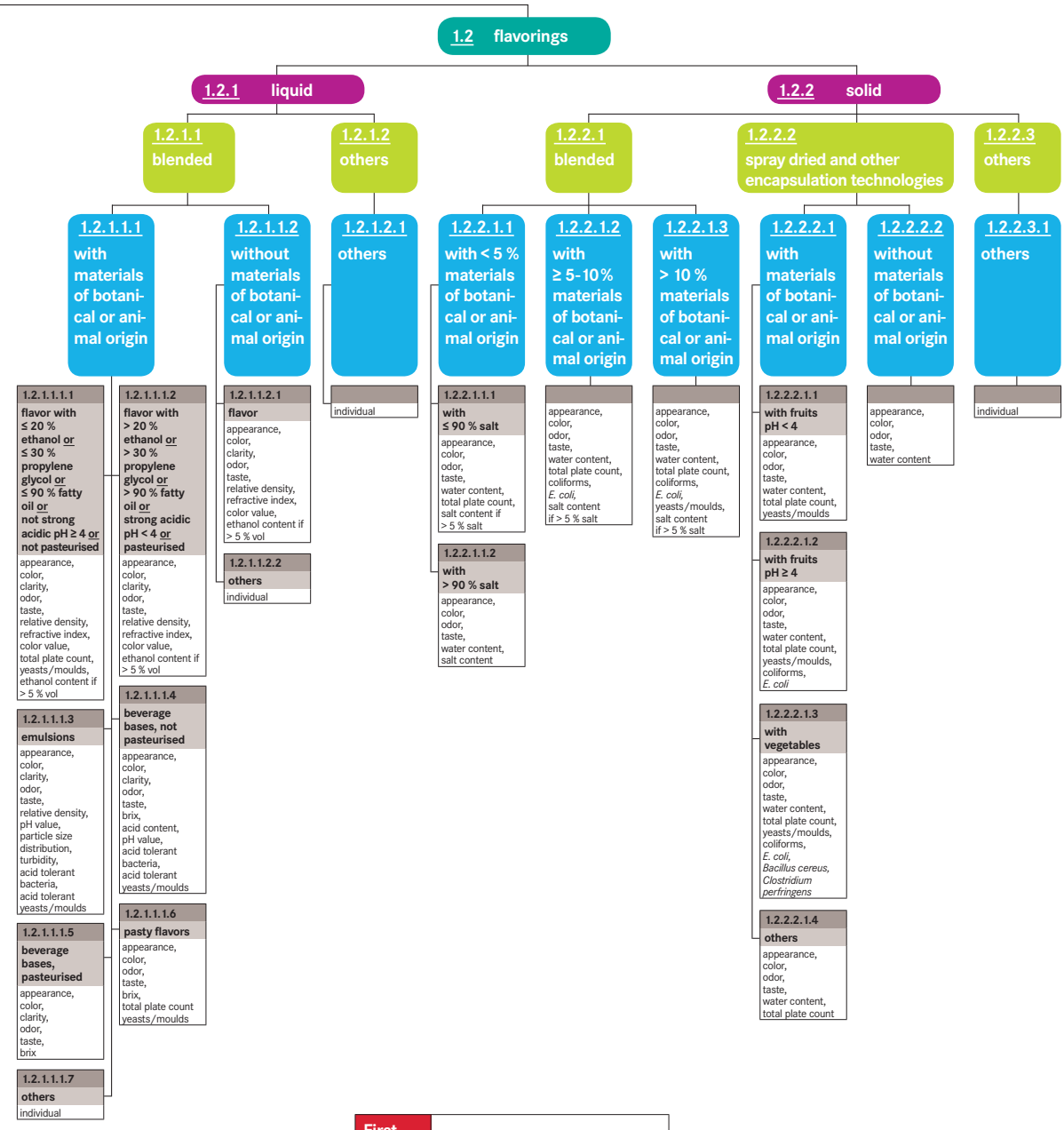
1.1.2.3.1.3 yeast extracts (aqueous extraction)
appearance, color, odor, taste, dry matter or brix, salt content, total plate count

1.1.2.3.1.4 others
individual

1.1.2.3.2 nature identical/ synthetic materials

individual

FLAVOR APPLICATION



First Level	flavor application
Second Level	raw material or flavoring
Third Level	appearance (liquid/solid)
Fourth Level	production technology (e.g. distilled, crystallized, blended)
Fifth Level	source of material (natural, nature identical/synthetic)
Sixth Level	details (e. g. terpenes, alcohols, ketones)

Additives (carriers, solvents ...) are not included

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The branch ***1.1.2 solid*** of flavoring raw materials shows the same structure as the liquid branch and can be separated into three groups, ***1.1.2.1 distilled*** materials, ***1.1.2.2 crystallized*** materials and ***1.1.2.3 others***. The next identical step down in the decision tree is the separation into natural and nature-identical/synthetic materials.

The first group in this row is ***1.1.2.1.1.1 esters, aldehydes***, which should be tested for appearance, color, odor, taste, specific rotation, acid value, melting point (or as an alternative IR spectra) and purity by GC. The difference from the equivalent group ***1.1.1.1.2.1*** in the branch for liquids is that the clarity, refractive index and relative density are no longer relevant test criteria, though melting point or IR spectra are performed. This test scope can also be found in group ***1.1.2.1.1.3 carboxylic acids*** in this row, but again the optical rotation for carboxylic acids should only be determined if appropriate. The same is true for ***1.1.2.1.1.1 alcohols, ketones, ethers, thiols, heterocycles***, though determination of the acid value is not necessary for these.

The test scope of the basic groups ***1.1.2.1.2.1*** to ***1.1.2.1.2.4*** shows the same structure as for ***1.1.2.1.1.1*** to ***1.1.2.1.1.4***; for detailed information on the test scope, reference should be made to the decision tree.

Crystallized natural and nature-identical/synthetic materials require, as previously described, additional testing for inorganic non-volatile residues. This can be checked via dry matter and sulfate ash. All other test parameters are comparable to solid distilled products.

There is a completely new section for natural materials such as ***1.1.2.3.1.1 fruits***, ***1.1.2.3.1.2 herbs and spices***, and ***1.1.2.3.1.3 yeast extracts***. Fruits should be tested for color, appearance, odor, taste, brix or dry matter, acid content, pH value, and in the case of a coloring material, the UV/visible absorbance. In general fruits are processed and not used as such in flavorings. For that reason the test plan should cover degradation of the resulting products. Depending on the processing steps used, the storage conditions, and the further usage of the intermediate product, the microbiological examination as described in ***1.1.1.2.1.1 juice, juice concentrates*** might be necessary.

The test scope for ***1.1.2.3.1.2 herbs and spices*** must cover all aspects of raw material testing for further processing and also for direct usage in flavorings such as seasonings for snacks. For that reason the microbiology is mandatory for herbs and spices if used in such applications. The countries of origin, the expected harvesting conditions, the character of the raw materials and the difficulties of applying germ-reducing procedures provide good reasons for the comprehensive microbiological test scope. The extraction yield should be determined if they are used as raw materials for extraction.

1.1.2.3.1.3 yeast extracts, which result from aqueous extraction, should be tested for appearance, color, odor, taste, brix or dry matter, and sodium chloride, which can contribute a considerable amount to the total salt content of a flavoring. From a microbiological point of view determination of total plate count is an appropriate test. Because of the presence of water and nutrient ingredients in the production process of yeast extracts, the growth of microorganisms has to be expected in general. On the other hand, the yeasts are a strong competitive flora for microbial contaminations that may occur, and the production of yeast extracts is highly processed. Therefore total plate count is a compromise to monitor the quality of this raw material group.

Switching to the second level in the decision tree of ***1.2 flavorings***, there is a branch dealing with ***1.2.1 liquid***, which is divided into ***1.2.1.1 blended*** and ***1.2.1.2 others***. In the authors' opinion, there is no notable production technique for liquid flavorings other than blending. The liquid flavorings are separated into groups due to the presence of materials of botanical or animal origin, which can cause presence of germs in the flavoring; hence there is a risk assessment according to microbiological stability.

The groups ***1.2.1.1.1.2*** and ***1.2.1.1.1.1*** could be considered mirror images of one another. The flavoring group ***1.2.1.1.1.2*** is characterized by an ethanol content above 20%, or a propylene glycol content above 30%, or more than 90% fatty oil, or a pH value below 4, or pasteurized, and has an identical analytical and sensorial test plan to ***1.2.1.1.1.1***, but microbiological testing is not necessary because this kind of contamination is unlikely.

The percentages given should be considered as guidelines and not as limits. Cumulative effects can also lead to reduced microbiological testing for ***1.2.1.1.1.1***. The test scope is completed by determination of the ethanol content if this exceeds 5%, and the details can be reviewed in the decision tree itself.

In addition, there are ***1.2.1.1.1.3 emulsions, beverage bases***—which can be pasteurized (***1.2.1.1.1.5***) or not (***1.2.1.1.1.4***) and consequently lead to two different groups— and ***1.2.1.1.1.6 pasty flavors***. The next group, ***1.2.1.1.1.3 emulsions***, should be checked for pH value and particle-size distribution, which are important parameters for stability. Turbidity, relative density and sensorial testing complete the test scope. A refractive index determination is not part of the test plan, due to difficulties in the analysis. Microbiological testing includes acid-tolerant bacteria and acid-tolerant yeasts and molds, comparable to ***1.1.1.2.1.1 juice and juice concentrates***. ***1.2.1.1.1.4 beverage bases, not pasteurized*** have differences in the analytical part. Turbidity and particle size are no longer included, but brix is an important parameter. Testing the pH value is necessary because certain stabilizing systems only show functionality in a defined pH interval. ***1.2.1.1.1.5 beverage bases, pasteurized*** are tested accordingly, with pH determination and microbiological testing being no longer necessary. For ***1.2.1.1.1.6 pasty flavors*** the analytical test scope is reduced to brix. Sensorial description of the quality can also be reduced to appearance, color, odor and taste. The acidity of these products is no longer of importance; thus, microbiological testing is only necessary for total plate count and yeasts and molds, to monitor the quality of the processed raw materials, and the process itself.

1.2.1.1.2 liquid flavors without botanical or animal origin material were chosen as the next group. Due to the use of materials without germ-containing potential, the tests of group ***1.2.1.1.2.1*** are identical to those of ***1.2.1.1.1.1***, and consequently are without microbiological testing.

The branch ***1.2.2 solid*** flavorings is divided into the production techniques ***1.2.2.1 blended*** and ***1.2.2.2***

spray dried and other encapsulation technologies, in addition to ***1.2.2.3 others***.

Below ***blended 1.2.2.1***, the group ***1.2.2.1.1 with < 5% materials of botanical or animal origin*** is found. The basic groups in this category differ as to whether their salt content is greater or less than 90%. A material with a salt content of up to 90% ***1.2.2.1.1.1*** should be tested for appearance, color, odor, taste, water content and total plate count. Due to nutritional and sensorial reasons sodium chloride should be determined if the content exceeds 5%. For ***1.2.2.1.1.2 with > 90% salt***, microbiological testing is no longer required, but salt testing becomes mandatory due to its high content.

Due to the increasing use of materials of botanical or animal origin, the groups ***1.2.2.1.2 with ≥ 5 – 10%*** and ***1.2.2.1.3 with > 10% of materials of botanical or animal origin***, were formed. The test plans differ from ***1.2.2.1.1.1*** only by advanced microbiological testing. So, for groups ***1.2.2.1.2*** and ***1.2.2.1.3*** the microbiological test scope is represented by total plate count, coliforms, *E. coli*, yeasts and molds. These methods are suitable for monitoring the quality of the raw materials used and the hygiene aspects.

Group ***1.2.2.2*** in this branch can also be divided according to its content of materials of botanical or animal origin. Under ***1.2.2.2.1*** are four groups of spray dried or other encapsulated solid flavors with materials of botanical or animal origin such as fruits, vegetables or others. Starting flavorings with ***1.2.2.2.1.1 with fruits with a pH value lower than 4***, in addition to tests for appearance, color, odor, taste and water content, the microbiological test scope includes total plate count, yeasts and molds. In the next group ***1.2.2.2.1.2 with fruits pH value above 4***, coliforms and *E. coli* are added to the microbiological test scope. The higher pH value does not inhibit the growth of coliforms and *E. coli,* and these should consequently be test points. Even more advanced is the test plan for ***1.2.2.2.1.3 with vegetables*** as ingredients, leading to additional testing. The last group, ***1.2.2.2.1.4 others***, is adequately tested by appearance, color, odor, taste, water content and total plate count. The only alternative in this group is ***1.2.2.2.2 without materials of botanical or animal origin*** in the formulation. Here, the growth of microorganisms is unlikely and the associated testing is not necessary. Hence, water content is the only test parameter required besides the sensorial testing, which is mandatory for all materials.

Working Examples

At the end of the description of the decision tree, two examples should show how the logical sorting process is performed. Natural ethyl butyrate is taken as the first example.

Example 1: Ethyl butyrate is a flavoring raw material, and so is classified as ***1.1 flavoring raw materials***. It is a liquid and is distilled, classifying it further under ***1.1.1.1***. In the example, ethyl butyrate is a natural material, hence finally ***1.1.1.1.1.2 esters, aldehydes*** is the correct group to locate the test scope, which includes appearance, color, clarity, odor, taste, relative density, refractive index, color value, acid value and purity by GC. In the example chosen, the optical rotation, which is part of ***1.1.1.1.1.2***, should be omitted because the molecule does not have a chiral center, and for that reason the optical rotation is zero.

Example 2: The second example is a blended liquid strawberry flavoring that contains materials of botanical origin. This example contains more than 30% propylene glycol and 10% ethanol. To find the corresponding test plan, the classification proceeds via ***1.2*** and ***1.2.1*** to ***1.2.1.1***. Due to the presence of ingredients of botanical origin, classification continues via ***1.2.1.1.1*** to ***1.2.1.1.1.2***; as a result, the flavoring has a test scope of appearance, color, odor, clarity, taste, relative density, refractive index, color value and ethanol content.

Summary

The selected approach for the setting-up of global specifications is based on the differentiation between flavoring raw materials and flavorings. The main group criteria are the physical form (liquid or solid), the manufacturing process and the product characterization (e.g. chemical group, essential oil, or botanical or animal origin). The selected methods need to be fast and robust, inexpensive, and widely accepted based on industry and normative standards.

Since there is currently no comprehensive and systematic framework available, the input of the different flavor associations such as FEMA, IOFI, EFFA, JFFMA has been thoroughly reviewed, in order to ensure applicability and acceptance of this first global concept for flavoring raw materials and flavorings.

Acknowledgements

The authors would like to thank Heike Gruber for her inexhaustible patience during all the amendments made after numerous reviews. Special thanks to Peter Liddle for the review of the manuscript.

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