

STANDARDS FOR IDENTITY AND PURITY OF FOOD ADDITIVES

Up till now the legal tolerance of the use of food additives has in most cases been completed in two stages. In the first stage governments laid down which food additives are considered acceptable and in which foodstuffs they can be permitted. It was not until the second stage that standards for purity were considered. It is my pleasure to talk to you on this latter aspect.

Probably the first important, legally demanded standards of purity were the specifications for food colours issued in the U.S.A. (Coal Tar Regulations, 1940). It took a long time for this example to be followed by the European countries, where things were still jogging along in the first stage. Problems set by the demands for purity were consciously pushed into the background, which explains why in the wellknown book by SOUCI and MERGENTHALER ⁽¹⁾, we do find the assay of several food additives, but no methods for the investigation of identity and purity.

The study of specifications of food additives was greatly stimulated by the Joint Committee of FAO and WHO, which discussed this subject in September 1955 and recommended the establishment of standards of purity and uniform methods of physical and chemical examination ⁽²⁾.

This was an auspicious initiative, as this work should indeed be done on a general, international level; if every country were to draw up its own specifications, this would only entail great and unnecessary loss of time and — which would be even worse — would raise new trade barriers.

The initiative taken by FAO and WHO led to the meetings of an Expert Committee on Antimicrobial Preservatives and Antioxidants [December 1958 ⁽³⁾] and of an Expert Committee on Food Colours [December 1959 ⁽⁴⁾]. The next meeting has been planned for February 1963, when emulsifiers and stabilizers will be discussed.

This work by FAO and WHO has already been followed up by national regulations, of which I will mention three instances:

a) Specifications of the British Standard Institution for four food colours ⁽⁵⁾.

b) Drafts of the « Deutsche Forschungsgemeinschaft » for standards of purity of food colours ⁽⁶⁾ and antimicrobials ⁽⁷⁾.

c) Drafts of the « European Economical Community » for standards of purity of antimicrobials ⁽⁸⁾.

It is to be expected that the future will bring a stronger control of food additives. The conviction that the use of chemicals in food entails grave risks, has gained ground after the margarine case in Germany and the Netherlands and certainly after the thalidomide affair. Reactions in the Dutch food processing industry are unmistakable. This tendency will hasten the establishment of standards of purity.

Value of standards of identity and purity.

Legal enactment of purity standards is no doubt of great value in many respects. This has been elaborately explained in the aforementioned Third Report of the Joint FAO/WHO Expert Committee ⁽³⁾, in which the following important points are emphasized:

- 1) protection of the consuming public;
- 2) the importance of standards for regulatory purposes;
- 3) the value to industry;
- 4) the guarantee of safety for public health.

These four subjects have been thoroughly treated in the report and I can only add one new point.

Producers of food additives and food processors must be thankful that governments are prepared to enforce certain standards of purity, as this relieves them of a great responsibility, which is now taken on by the governments.

Toxicologists have often emphasized that impurities or minor constituents may have a significance far greater than their amounts might indicate. Much stress must therefore be laid on the value of a good description of the food additive and its unavoidable impurities with regard to toxicity investigation.

Format.

The format in which the standards for an additive are presented, has developed in the course of years; a good example is the format chosen by the WHO/FAO Expert Committee. It consists essentially of four parts:

- the description of the chemical composition of the substance,
- the identification methods,
- the purity standards, together with the analytical methods and finally the assay.

Each of these parts deserves a closer examination.

Chemical description.

The description of the chemical composition is simple and complete in the case of single chemical substances. However, some food additives are on the contrary, very complex in composition. In this respect I would like to mention:

a) synthetic polymers, as silicones, polyglycols, polyglycerids, polyoxyethylenes;

b) substances of different substitution grade, e.g. sodium carboxymethylcellulose;

c) natural products like tragacanth, guar-gum, annatto-extract;

d) condensation products, if many chemical condensations are possible e.g. condensation of isomerized soya oil with maleinic anhydride and esterification with stearyl alcohol (ME 18 emulgator).

The composition of these substances is usually not exactly known. Complete chemical analysis is very difficult, if not impossible [compare Kröller's interesting analysis of ME 18 (⁹)]. We manage with makeshift delimitations which the complex substances have to meet. I will mention some methods and give some examples derived from USA-legislation:

Principle of limitative description

molecular weight

Example

polythyleneglycol: 400 through 6000

polythyleneglycol: average ranging 6000-7500

<i>Principle of limitative description</i>	<i>Example</i>
	polyvinyl acetate: minimum 2000
	dextrane: below 100.000
chain length	coconut fatty acid: C_{12} - C_{16}
	petroleum sulfonate: C_{12} - C_{18}
specific gravity	polyethylene: 0,83-0,95
solubility	ethylene-butene 1-copolymer: extractibility in xylene <75% (25°C)
	extractibility in ethyl acetate <8% (50°C)
	extractibility in hexane <53% (25°C)
melting range	wax microcrystalline and paraffin: congealing point of 160°F maximum
softening point	asphalt steam-refined: 175°F min
flash point	asphalt steam-refined: 500°F min
boiling range	hydrocarbons aromatic: 362-490°F mineral oil: min. 50% boiling
	below 700°F and min. 95% boiling below 775°F
viscosity	dimethylpolysiloxane: 100-350 centistokes
	sodium carboxymethylcellulose: min. 25 cP for 2% sol
UV-absorption spectrum	modified hop extract
Substitution grade	starch modified with propylene oxide (hydroxypropylstarch): limit 12% propylene oxide
	methylcellulose: methoxy % 27,5-31,5
	sodium carboxymethylcellulose: max. 0,95 carboxymethylgroups per anhydroglucose unit
	ethyl cellulose: max. 2,60 ethoxy groups per anhydroglucose unit
	castor oil, polyoxyethylated: 4-84 mols ethylene oxide

<i>Principle of limitative description</i>	<i>Example</i>
by combination of saponification number + hydroxyl number + oxyethylene content	polyoxyethylene (20) — sorbitan tristearate: 88-98 and 44-60 and 46-50%
by combination of Reichert Meissl and acid values	distilled acetylated monoglycerides: 75-150 and less than 6

It should be clearly explained to the toxicologists, to what degree complex additives are identifiable in the mentioned chemical and physical terms and to what degree the limits of reproducible composition can be defined. It is up to the toxicologist to decide, in close concert with the chemist, whether it is sufficiently probable that the batch of the additive tested in animals is the same as used by food processors and whether his toxicity investigation of one batch is sufficient for the evaluation of the safety of use of all commercial products falling within the limits of the description.

Identification.

The part called identification, contains the methods to determine whether the sample is indeed the substance as labelled. In my experience food processors consider this identification even more important than purity control. They live in constant fear of mistakes by the manufacturer of the additives or mistakes by their own staff. The combination of three groups of tests, namely for identification, for purity control and for assay should together form a sieve which allows the common, commercial poisonous substances to be discovered. This control will be a protection, not only against predictable impurities, but also against unexpected, poisonous substances.

Purity.

Purity control of a substance must be based on a thorough knowledge of its production history. If it is a natural substance of origin, e.g. gelatin, agar-agar, annatto, the methods of extraction, isolation, purification and chemical treatment must be known.

If the substance is a synthetic product, impurities may arise from raw materials, reagents, solvents and equipment. Intermediates or by-products may be present in the commercial product and decomposition products may be formed during storage. A standard of purity, consisting only of a few words, may have a background of longwinded discussions of chemical factory methods just like a concise law text may be a condensation of long hours of study.

It is impossible and it seems unnecessary to set up standards for every possible impurity. However, the purity must be guaranteed in these three aspects:

1) that the use of the additive is acceptable to public health authorities;

2) that the technological effect of the additive is not damaged by impurities;

3) that the food is not contaminated by an impurity in the additive, if that impurity is avoidable in good manufacturing practice.

It is easily said that impurities in an additive must not form a danger for public health, but toxicologists have great difficulty to lay down tolerance limits for impurities, such as arsenic, selenium, amines; this point, I suppose, will be treated in the next reports of this Symposium.

The normal procedure, however, in establishing standards of purity is not to invite toxicologists to indicate their safety limits in the first stage. One should actually investigate which concentration of impurities need not be exceeded in good manufacturing practice. The next stage should be to consult the toxicologist on those concentrations. Only in rare cases normal commercial purity has not been found satisfactory for safe use.

Economical aspects.

It is conceivable that a producer of a very pure additive advocates more stringent specifications than a producer of a less pure product. Stringent specifications may cause a monopoly position. International consultations should not lead to unnecessary monopoly positions of some branch of industry or some country.

Who supplies the necessary data for standards?

A small number of substances used as food additives are also used as drugs and dealt with in pharmacopoeias or related compendia. In

such cases standards of purity are available and they may provide a basis for use in the food field. However, most food additives are not used as drugs or as components of drugs.

The data required for setting up purity standards are only partly supplied by governmental officials and scientific laboratories. An important part of the information has to come from industry itself, because complete knowledge of production methods and of risks of contamination is in most cases only available from a small number of persons connected with the production. Producers generally have specifications and analytical methods for internal use, which, in most cases, have never been published. I have the impression that bonafide industries cooperate without reluctance with the commissions that have to set up standards.

Nature of methods of investigation.

Investigation methods applied in well-equipped industrial laboratories, are often intended for series of analyses, done with precious instruments, such as the infrared spectrophotometer, the emission-spectrophotometer, the gas-chromatograph, the polarograph etc. For the time being these methods are less suitable for public laboratories, at least in the Netherlands. These public laboratories receive samples of all kinds of foodstuffs, of which very different components have to be determined. Therefore they want methods which do not demand a long specialistic experience from the investigator and do not necessitate repeated setting-up and running-in of complicated equipment which has been out of use for some time.

It often happens, therefore, that new methods have to be worked out which do not need precious equipment. Here we come up against a deficiency: no laboratory feels inclined to devote much time to this. This gap might possibly be filled up by fellowships from FAO and WHO to analytical chemists.

Investigation of certain impurities of a food additive can sometimes be done by simple « limitative reactions ». They yield a negative result, as long as the impurity contents remain below the maximum tolerance. Thus the need for quantitative determination of the impurity is obviated.

The legal demand that food additives may only be kept by food processors in the original packing bearing name, composition and the indication « for use in food » is contributive to the control of identity and purity. U.S.A. regulations for colouring matter still go a lot further by numbering and analysing each lot, only allowing numbered packages to be in stock.

Microbiological standards.

Microbiological standards of food additives have not been discussed during the international meetings on colours and antimicrobials. This question was not topical for these additives, although in principle all additives can be contaminated with bacteria, spores of bacteria or moulds during preparation or storing.

This aspect, however, must not be lost sight of for other additives. Years ago BUTTIAUX ⁽¹⁰⁾ drew attention to infection of meat owing to the adding of gelatine. MOSSEL ⁽¹¹⁾ continued on this subject and dealt with the sometimes very bad bacteriological condition of commercial qualities of spices, common salt, brine salt, sugar, milkpowder and flower. BODROSSY ⁽¹²⁾ investigated the bacterial flora of spices and preservatives, GHYSEN ⁽¹³⁾ of samples of cooking-salt, spices, starch and gelatine and HOEKE ⁽¹⁴⁾ of chicken albumen. I should like to add that it might also be interesting to investigate the microbiological condition of natural products which are not or cannot be heated, such as guar-gum, gum arabic, and enzyme preparations.

Acceptance of FAO/WHO proposals without alterations.

It appears that some countries are prepared to accept the FAO/WHO recommendations, though with certain alterations. As an example, I give a survey of the arsenic, lead, copper and zinc maxima:

	Food Colours				Antimicrobials		
	As	Pb	Cu + Zu	Cu	As	Pb	Cu + Zn
FAO/WHO	3	10	—	—	3	10	—
British Standard							
Inst.	4	10	—	10			
DFG	5	20	—	—	3	10	50
EEG					—	—	—

— means: no specification.

There are also differences in other specifications. DFG for instance lays down more stringent demands for the selenium contents of sulphites, another assay method for para-esters and so on. The EEG-draft mentions no methods at all, it is more stringent on selenium but less for the percentage of propionates.

DFG says in the Introduction on Antimicrobials: « The recommendations agree on important points with those of Joint WHO/FAO Expert Committee on Food Additives » ⁽⁷⁾. Personally I am of opinion that it would be better for countries to accept the FAO/WHO recommendations unaltered.

REFERENCES

- (1) S. W. SOUCI and E. MERGENTHALER - Fremdstoffe in Lebensmitteln, Verlag von J. F. Bergmann, München, 1958.
 - (2) General Principles governing the use of food additives, First Report of the Joint FAO/WHO Expert Committee on Food Additives, WHO Techn. Report Series 129 (1957) and FAO Nutrition Meetings Report Series 15 (1957).
 - (3) Specifications for identity and purity of food additives: antimicrobial preservatives and antioxidants, Third Report of the Joint FAO/WHO Expert Committee on Food Additives (mimeographed 1959).
 - (4) Specifications for identity and purity of food additives: Food Colours, Fourth Report of the Joint FAO/WHO Expert Committee on Food Additives (draft, mimeographed 1960).
 - (5) British Standard Institution, British Standard House, 2 Park Street, London W 4
3340: 1961 Specification for Sunset Yellow FCF
3341: 1961 Specification for Amaranth
3342: 1961 Specification for Ponceau 4 R
3343: 1961 Specification for Carmoisine
3210: Methods for the analysis of water-soluble coal tar dyes permitted for use in foods.
 - (6) Farbstoff-Kommission aus der Deutschen Forschungsgemeinschaft, Mitteilung 10 (1961), Allgemeine Reinheitsanforderungen für Lebensmittelfarbstoffe.
 - (7) Kommission zur Prüfung der Lebensmittelkonservierung aus der Deutschen Forschungsgemeinschaft, Mitteilung VI (1961), Reinheitsanforderungen.
 - (8) European Economic Community, Spezifische Reinheitskriterien (für Konservierungsmittel).
 - (9) E. KRÖLLER - Fette, Seifen, Anstr., 64: 85 and 602 (1962).
 - (10) R. BUTTIAUX - Food Manufacture, 28: 112 and 135 (1953); *ibid*, Ann. Inst. Pasteur Lille, 4: 145 and 7 (1954); 162 (1955).
 - (11) D. A. A. MOSSEL - Ann. Inst. Pasteur Lille, 7: 171 (1955).
 - (12) L. BODROSSY - Húsipar, 9 (1960) 160; refer. Z. Lebensm. Unters. Forsch., 115: 80 (1961).
 - (13) J. GHYSEN - Conserva, 10: 138 (1962).
 - (14) F. HOEKE - Conserva, 10: 186 (1962).
-