COUNCIL OF EUROPE
COMMITTEE OF MINISTERS

(PARTIAL AGREEMENT IN THE SOCIAL AND PUBLIC HEALTH FIELD)

RESOLUTION AP (89) 1

ON THE USE OF COLOURANTS IN PLASTIC MATERIALS COMING INTO CONTACT WITH FOOD

(Adopted by the Committee of Ministers on 13 September 1989
at the 428th meeting of the Ministers' Deputies)

The Representatives on the Committee of Ministers of Belgium, France, the Federal Republic of Germany, Italy, Luxembourg, the Netherlands and the United Kingdom of Great Britain and Northern Ireland, these states being parties to the Partial Agreement in the social and public health field, and the Representatives of Austria, Denmark, Ireland, Norway, Spain, Sweden and Switzerland, states which have participated in the public health activities pursued within the above-mentioned Partial Agreement since 1 October 1974, 2 April 1968, 23 September 1969, 11 July 1979, 21 April 1988, 10 June 1975 and 5 May 1964 respectively,

Considering that the aim of the Council of Europe is to achieve a greater unity between its members and that this aim may be pursued, amongst others, by common action in the social and public health field;

Having regard to the provisions of the Brussels Treaty signed on 17 March 1948, by virtue of which Belgium, France, Luxembourg, the Netherlands and the United Kingdom of Great Britain and Northern Ireland declared themselves resolved to strengthen the social ties by which they were already united;

Having regard to the protocol modifying and completing the Brussels Treaty, signed on 23 October 1954 by the signatory states of the Brussels Treaty, on the one hand, and the Federal Republic of Germany and Italy, on the other hand;

Observing that the seven states parties to the Partial Agreement which have continued, within the Council of Europe, the social work hitherto undertaken by the Brussels Treaty Organisation and then by Western European Union, which derived from the Brussels Treaty as modified by the protocol mentioned in the fourth paragraph above, as well as Austria, Denmark, Ireland, Norway, Spain, Sweden and Switzerland, who participate in Partial Agreement activities in the field of public health, have always endeavoured to be in the forefront of progress in social matters and also in the associated field of public health, and have for many years undertaken action towards harmonisation of their legislation;

Having regard to the fact that colourants are used to impart a colour to plastic materials coming into contact with food;

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Forty years
Council of Europe

Considering that plastic materials coming into contact with food may, by reason of their colouration, pose a risk to human health if not used under normal conditions or if the colourants used do not meet purity criteria based on good manufacturing practice;

Taking the view that each member state faced with the need to introduce regulations governing this matter would find it beneficial to harmonise such regulations at European level,

Recommend that the governments of the states parties to the Partial Agreement as well as the governments of Austria, Denmark, Ireland, Norway, Spain, Sweden and Switzerland take into account in their national laws and regulations the principles set out in this resolution and the analytical methods and, in particular, specifications on the use of inorganic cadmium pigments set out in the appendix to this resolution.

Appendix to Resolution AP (89) 1

I. Field of application

This resolution applies to the use of colourants in plastic materials and articles coming into contact with food. Colourants are substances which are intentionally added to plastics to impart colour; they include dyes, organic and inorganic pigments.

II. General principles

1. *Migration from the final plastic material or article*

Under normal or foreseeable conditions of use, plastic materials and articles should not, by reason of their colouration, pose a risk to human health or bring about either a deterioration in the organoleptic characteristics or an unacceptable change in the nature, substance or quality of the food with which they come into contact.

Colourants should be sufficiently integrated within plastic materials and articles so as to preclude any visible migration into foodstuffs under normal conditions of use, as determined by an appropriate method.[1]

2. *Specification*

The colourants to be tested should be the active ingredients and not the active components predispersed in a medium to facilitate good distribution during processing of the plastic materials.

The colourants should meet the purity criteria of the provisions 2.1 — 2.4 and be based on good manufacturing practice.

2.1. *Metals and metalloids*

The content of metals and metalloids soluble in 0.1M hydrochloric acid, determined as a percentage in relation to the colourant, should not exceed:

antimony: 0.05%
arsenic: 0.01%
barium: 0.01%
cadmium: 0.01%
chromium[2]: 0.1%
lead: 0.01%
mercury: 0.005%
selenium: 0.01%

The analytical method is described in section III, paragraph 2.

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[1] It should be noted that, under normal or foreseeable conditions of use, the specific migration of aromatic amines and of metals from the finished plastic materials or articles should not exceed the limits specified in the Council of Europe booklet *Substances used in plastics materials coming into contact with food* (1).

[2] The use of chromium VI pigments may pose a risk to human health and should be discouraged.

2.2. *Aromatic amines*

The content of primary unsulphonated aromatic amines soluble in 1M hydrochloric acid and expressed as aniline should not exceed 500mg/kg. The content of benzidine, ß-naphthylamine and 4-aminobiphenyl, singly or in total, should not exceed 10mg/kg.[1] The analytical methods are referred to in section III, paragraph 3.

2.3. *Sulphonated aromatic amines*

Sulphonated aromatic amines are defined as those compounds sulphonated on all aromatic or conjugated ring structures.

The total content of sulphonated aromatic amines extracted by a suitable solvent and determined by an appropriate test, expressed as aniline sulphonic acid, should not exceed 500mg/kg. This limit can be exceptionally exceeded if adequate supporting data on the technological need are provided. An analytical method is discussed in section III, paragraph 4.

2.4. *Carbon black*

Carbon black should comply with any national requirement for food grade material. Furthermore, the toluene-extractable fraction of carbon black should not in any case exceed 0.15% in accordance with the method described in section III, paragraph 5.

2.5. *Polychlorinated biphenyls (PCBs)*

The content of extractable polychlorinated biphenyls should not exceed 25mg/kg, expressed as decachlorobiphenyl, as determined by an appropriate method indicated in section III, paragraph 6.

2.6. *Inorganic cadmium pigments*

A number of national and international authorities have recommended that the intake of cadmium by man should be reduced as far as possible. Furthermore, environmental contamination by cadmium is a recognised international problem. Consequently, a number of regulatory authorities have either taken steps to reduce the total industrial use of cadmium or are actively considering such measures.

The use of inorganic cadmium pigments in plastic materials and articles coming into contact with food is small but, in certain applications, such use is claimed to offer major technological advantages. The migration of cadmium from the use of such pigments is, in general, very low but extraction can vary from plastic type to plastic type. Thus, the use of inorganic cadmium pigments in plastics coming into contact with food will probably make only a minor contribution to man's total intake. However, in order to keep the contamination of food with cadmium in general as low as possible, the use of such pigments should be restricted to applications where their special technological advantages cannot be met by alternative products.

3. *Toxicological tests*

Toxicological data should be obtained from pages 71 to 77 of the Council of Europe booklet *Substances used in plastics materials coming into contact with food* (1).

III. Analytical methods

1. *Determination that there is no visible migration*

1.1. *Apparatus*

— filter paper for qualitative analysis, of medium porosity such as *Weissband Mittel* (Schleicher and Schull), chromatography paper (Archer, 302), etc.;

— glass desiccator or other suitable sealed container;

— oven regulated at 50° ± 2°C.

1.2. *Simulants*

— distilled water or water of equivalent quality;

— 3% acetic acid (w/w) in aqueous solution;

— 15% ethanol (v/v) in aqueous solution;

— decolourised edible oil or fat or a comparable synthetic triglyceride.

If foods do not correspond to the above simulants, for example because of high acid or alcohol content, the simulants should be modified accordingly.

1.3. *Contact surface*

At least 10cm^2.

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[1] In normal use, therefore, the quantity of these substances which may migrate into food will be toxicologically insignificant.

1.4. *Operating procedure*

Impregnate strips of filter paper with the simulant(s) for the food(s) intended to come into contact with the material. These strips should have a contact surface of 10 to 15cm². Eliminate excess simulant by wringing the strips between two glass rods.

Place the impregnated strips onto the food contact surface of the material or article to be tested, so that there is intimate contact, and fix with non-coloured adhesive tape. Ensure that there are no air bubbles between the surface of the sample and the impregnated paper and verify that the latter exerts an even pressure on the sample after the adhesive tape is affixed.

A simpler method of attachment may be applied in the case of flat samples. On a glass plate, place consecutively 100cm² of sample and a few impregnated strips, cover with a further flat glass plate and apply a weight of approximately 1kg.

Impregnate additional strips of filter paper with simulant to act as blanks. It is important that these do not come into contact with any coloured materials or articles.

Place samples and blanks in a desiccator and maintain for five hours in an oven regulated at 50° ± 2°C. Where an aqueous simulant is used, it is necessary to transfer a portion to the desiccator and maintain at 50° ± 2°C for thirty minutes prior to use. This will ensure that the atmosphere in the desiccator is saturated with the vapour of the simulant.

After the set contact time, separate the strips of impregnated filter paper from the samples and isolate the blank strips. In the case of aqueous simulants, place the strips in the oven at 50° ± 2°C until the impregnated liquid has evaporated (approximately fifteen minutes). In the case of the oil simulant, allow the strips to cool.

1.5. *Assessment of visible migration*

Compare the strips that have been in contact with the sample with the blank strips; these should have remained colourless. The visual comparison of strips is undertaken in daylight (2). The plastic materials or articles meet the requirements if comparison of test strips and blank strips reveals no difference.

2. *Determination of metals and metalloids*

2.1. *Apparatus*

— agitation device having a rotary mechanical agitator, non-magnetic, with a speed sufficient to maintain the colourant in suspension but without causing air bubbles to be formed;

— membrane filter with 0.15 micrometre porosity.

2.2. *Reagent*

— hydrochloric acid 0.1M of analytical purity.

2.3. *Operating procedure* (3)

Place in suspension for fifteen minutes, at 23° ± 3°C, 10g of colourant in 150ml of 0.1M hydrochloric acid, using a mechanical agitator. Filter the acid solution, after sedimentation of the colourant for ten minutes, through the membrane filter. The filtrate is then analysed for elements mentioned under section III.1 in accordance with known methods, for example (4 to 6). Blank determination on reagents is essential.

3. *Determination of primary unsulphonated aromatic amines*

An extract containing the aromatic amines is prepared by treatment of the organic colourant with 1M hydrochloric acid as described in ETAD Method No. 212 (7). The quantitative determination of primary unsulphonated aromatic amines in this extract may be undertaken either according to this method or to specific methods (8-11).

4. *Analytical methods for the determination of aromatic aminosulphonic acids*

The pigments are extracted with a suitable solvent (for example acetic acid, diluted sodium hydroxide solution, methanolic ammonia or 1 N HCl, as described in ETAD Method No. 212) and the aminosulfonic acids in the extracts determined by thin layer chromatography. On the TC (toluene column), the compounds are identified by colour reaction (for example diazotation and coupling with N-(1-naphthyl)-ethylene-diamine or with R salt) and the quantity determined by comparison with standards of the compounds.

5. *Determination of the toluene-extractable fraction of carbon black*

5.1. *Principle* (12)

Carbon black is extracted in a Soxhlet apparatus with toluene. Thereafter, the solvent is evaporated and the residue weighed.

5.2. *Apparatus*

- Soxhlet extraction apparatus;
- extraction vessels of paper fibre, capable of containing 10g of carbon black;
- laboratory glassware;
- oven, adjustable up to 140° ± 5°C.

5.3. *Solvent*

Toluene.

5.4. *Operating procedure*

Dry a sufficiently large sample of carbon black for one hour at 105° ± 2°C. Place 10g of the dried carbon black (weighed with an accuracy of 0.1g) in an extraction vessel previously washed with toluene. Close the vessel with cotton previously washed with toluene and place the vessel in the Soxhlet apparatus.

Extract the carbon black for eight hours with 150ml toluene. Adjust the heating element of the apparatus in such a way that the extraction solvent in the extraction vessel is replenished approximately ten times per hour.

Allow to cool, remove the extraction vessel from the apparatus and concentrate the extract to a small volume by evaporation. Wash the residue with a little toluene into a weighed glass crucible (weight: *a* g) and remove the toluene by evaporation on a water bath. Place the crucible in an oven for two hours at 140°C; allow to cool in a desiccator and reweigh the crucible (weight: *b* g).

5.5. *Calculation*

Calculate the extractable fraction E of the carbon black using the following formula:

$E = 10\,(b - a)\%$
E shall not exceed 0.15%.

6. *Determination of extractable polychlorinated biphenyls (PCBs)*

Polychlorinated biphenyls (PCBs) are extracted from organic colourants and determined as specified in the various methods described in the literature (13). For the determination of very low levels of PCBs (<5 mg/kg), it is necessary to use methods (14, 15) based on gas chromatography/electron capture and capillary gas chromatography/electron impact mass spectrometry.

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