3 Regulations Covering the Use of Rubber as a Food Contact Material

3.1 European Union Legislation

At the moment there is no specific EU legislation for rubber food contact materials or articles (other than nitrosamines in babies’ dummies [3]). Thus all such materials or articles need to comply with the general Framework Directive 89/109/EEC [4, 5, 6] so that in normal use they will not transfer their constituents to food in quantities that could endanger health or cause unacceptable changes in the composition of food or deterioration in its organoleptic properties (i.e., taste, texture, aroma, or appearance).

3.2 Council of Europe (CoE) Resolution on Rubber Products

The CoE (an international organisation, separate from the European Union) has a committee of Experts on Materials and Articles Coming into Contact with Food, that meets under the auspices of the Partial Agreement in the Social and Public Health Field. Once adopted, the resolutions and supporting documents drawn up by these bodies are not legally binding, but members of the Partial Agreement, such as the UK, are expected to take note of them.

The CoE Rubber Resolution on food contact elastomers (APRes 2004) was adopted in 2004. This document has an inventory list of additives within it and a small section that deals with breakdown products – nitrosamines and amines. The inventory list is described as Technical document No. 1 – List of substances to be used in the manufacture of rubber products intended to come into contact with foodstuffs. An FSA project (Contract number A03038 – see Section 4.5.3) was commissioned to study the breakdown and reaction products from the curatives and antidegradants present in this inventory list.

3.2.1 Technical Documents

In addition to Technical Document No. 1, there are four other documents associated with the Resolution; they are:

- Technical document No. 2: Guidelines concerning the manufacture of rubber products intended to come into contact with foodstuffs

- Technical document No. 3: Good manufacturing practices of rubber products intended to come into contact with foodstuffs
3.2.2 Product Categories

The Resolution places rubber products into one of three categories:

**Category I** comprises the following rubber products for which migration testing is required:
- feeding teats
- rubber products to come in to contact with baby food, for which the R-total is equal or greater than 0.001 (a definition of R-total is given below).

**Category II** comprises rubber products for which the R-total is equal or greater than 0.001 and for which migration testing is required.

**Category III** comprises rubber products for which R-total is smaller than 0.001 and for which migration testing is not required, except for rubber products containing nitrosamines, nitrosatable substances or aromatic amines and Category III substances with substances with a specific migration limit (SML) in Technical document No. 1.

These three categories take into account the wide variety of applications for which rubber products are used and the fact that migration may vary with the application. The level of migration for rubber products may be estimated by taking into account four factors, R_1, R_2, R_3 and R_4 referring respectively to the relative contact area, contact temperature, contact time and number of times that the article is used. Categories are based on the intended use or on the result of the multiplication of the four factors (R_1 \times R_2 \times R_3 \times R_4 = R \text{ total}).

3.2.3 R Factors

The factors R_1, R_2, R_3 and R_4 can be defined as follows:

R_1 refers to the relative contact area (A_R) between rubber products and food or beverage, expressed in cm\(^2\) of rubber surface per kg of food or beverage. For a relative area smaller or equal to 100 cm\(^2\)/kg foodstuffs, R_1 has a value calculated according to the formula: R_1 = A_R/100. For a relative surface larger than 100 cm\(^2\)/kg, R_1 always has the value of 1.00.
R₂ refers to the temperature during the contact period of the rubber product with the food or beverage. At a temperature lower than or equal to 130 °C, R₂ has a value calculated according to the formula: \( R₂ = 0.05e^{0.023T} \). Where ‘e’ is the base of the natural or Napierian logarithms and T is the contact temperature, expressed in °C. For temperatures higher than 130 °C, R₂ always has the value 1.00.

R₃ refers to the time, t, expressed in hours, during which a rubber product is in contact with the food or beverage. For a contact time shorter than or equal to 10 hours, R₃ has a value calculated according to the formula: \( R₃ = t/10 \). For a contact time of more than 10 hours, R₃ has the value 1.00.

R₄ refers to the number of times, N, that one and the same rubber product, or part of that rubber product comes into recurrent contact with a quantity of food or beverage. If the number of contact times is greater than 1000, then R₄ is calculated according to the formula: \( 10\log R₄ = 6 - 2 \times 10\log N \). If the number of contact times is smaller than or equal to 1000, then R₄ always has the value 1.00.

The Resolution also states that, amongst other things:

1. Rubber products in Categories I and II should not transfer their constituents to foodstuffs or food simulants in total quantities >60 mg/kg food or food simulant (overall migration limit).

2. Rubber products of Categories I and II should comply with the restrictions laid down in Technical document No. 1. In addition these rubber products should comply with the requirements set out in Table 1 of the Resolution, excepting rubber teats which should comply with Directive 93/11/EEC.

3. Rubber products intended for repeated use should be subjected to tests according to Directive 2002/72/EC Annex 1 [7].

4. Rubber products belonging to Category III do not require migration testing, unless otherwise specified.

5. Verification of compliance with the quantitative restrictions should be carried out according to the requirements laid down in Technical document No. 2 – Practical guide for users of Resolution AP (2004) on rubber products intended to come into contact with foodstuffs. This is an accompanying document to the Resolution and provides more detail regarding the following:

   - further definitions and data surrounding the R values
   - examples of rubber products that fall into Categories II and III and the calculations applied to them
   - migration tests
3.2.4 Silicone Rubbers

There is a separate CoE Resolution (APRes 2004) covering silicone materials for food contact. The resolution defines the silicone product group being comprised of silicone rubbers, silicone liquids, silicone pastes and silicone resins. Blends of silicone rubber with organic polymers are covered by the resolution where the silicone monomer units are the predominate species by weight. Silicones that are used as food additives (e.g., as defoamers in the manufacture of substances such as wine) are not covered by this resolution, but polysiloxanes used as emulsifiers are. The resolution gives an overall migration limit of 10 mg/dm² of the surface area of the product or material, or 60 mg/kg of food. There are restrictions on the types of monomers that can be used to produce the silicone polymers and there is an inventory list - Technical document No. 1 - List of substances used in the manufacture of silicone used for food contact applications.

3.3 Food and Drug Administration (FDA) in the USA

In America the FDA produces a Guidance for Industry document entitled ‘Preparation of Food Contact Notifications and Food Additive Petitions for Food Contact Substances: Chemistry Recommendations’. This is in addition to the Code of Federal Regulations Volume 21, Parts 170 to 199 Food and Drugs, which contains the FDA food contact regulations. This is published annually and rubber products for use with food are covered in Part 170, specifically Rubber articles intended for repeat use: 177.2600; and Closures with sealing gaskets for food containers: 177.1210. A review and comparison of the FDA and German regulations (Section 3.4) with particular emphasis on the inventory lists of approved ingredients has been carried out by Pysklo [8]. A schedule of commercial ingredients which meet the requirements of the two regulations is provided.

The FDA regulations are relatively straightforward. Providing that the ingredients in the rubber are listed as being approved, and the water (for aqueous food use) or hexane (for fatty food use) extractables under reflux conditions are within the prescribed limits, then the compound is considered suitable for food use (see Table 3).

In addition to listed compounding ingredients, the regulations also allow the use of prior sanctioned ingredients and also additives that are generally recognised as safe (GRAS). Prior sanctioned materials are listed in the appropriate sections of the FDA regulations, for example:

| 181.27 | Antioxidants | 181.28 | Release agents |
| 181.27 | Plasticisers | 181.29 | Stabilisers |
Other indirect food substances described as GRAS, which can be used as rubber additives, are listed in various parts of CFR 21. Substances listed in section 182 include zinc oxide (182.8991), zinc stearate (182.8994) and calcium silicate (182.2227). GRAS food substances listed in section 184 include calcium carbonate, calcium stearate and calcium oxide. Other multi-purpose GRAS food substances include kaolin clay (186.1256) and iron oxide (186.1374).

The FDA regulations specifically prohibit the use of the following ingredients in food contact rubbers:

- Section 189.220 Polymerised 1,2-dihydro-2,2,4-trimethylquinoline
- Section 189.250 Mercaptoimidazoline and 2-mercaptoimidazoline

The FDA also places severe restrictions (i.e., 0.003 /in²) on the migration of acrylonitrile monomer from nitrile rubbers (Section 181.32).

### 3.4 Bundesinstitut für Risikobewertung (BfR) German Regulations

Within Germany, the food contact legislation for rubbers is described in BfR (formerly BgVV) Recommendation XXI ‘Commodity Articles Based on Natural and Synthetic Rubber’. There are separate requirements for silicone rubbers and these are contained within Recommendation XV.

#### 3.4.1 Categories of Use

In the case of Recommendation XXI, four use categories and a special category are defined as follows:

<table>
<thead>
<tr>
<th>Table 3. FDA migration limits for repeat use articles</th>
<th>Fatty foods – Hexane extractables under reflux</th>
</tr>
</thead>
<tbody>
<tr>
<td>First seven hours</td>
<td>175 mg/in²</td>
</tr>
<tr>
<td>Succeeding two hours</td>
<td>4 mg/in²</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Aqueous foods – Distilled water extractables under reflux</th>
</tr>
</thead>
<tbody>
<tr>
<td>First seven hours</td>
</tr>
<tr>
<td>Succeeding two hours</td>
</tr>
</tbody>
</table>
• **Category 1** (Test conditions: 10 days at 40 °C). Rubber articles which come into contact with food for periods of more than 24 hours to several months, e.g., storage containers, container linings, seals for cans and bottles etc.

• **Category 2** (Test conditions: 24 hours at 40 °C). Rubber articles which come into contact with food for not more than 24 hours, e.g., food conveying belts, tubes and hoses, sealing rings for cooking pots, lock seals for milk can lids etc.

• **Category 3** (Test conditions: 10 minutes at 40 °C). Rubber articles which come into contact with food for not more than 10 minutes, e.g., milk liners and milking machine tubes, roller coatings and conveyor belts (fatty foods only in both cases), gloves and aprons for food handling etc.

• **Category 4** (No migration testing required). Rubber articles which are only used under conditions where no migration into food is to be expected, i.e., if the article comes into contact with the food for a very short time or only over a very small area and if it is not covered by Categories 1, 2 or 3. Examples of rubber products in this category include: conveyor belts, suction and pressure hosing for moving and loading/unloading dried food; tap washers, bevel seat valves, pump parts and other articles associated with the supply of drinking water.

• **Special Category** (Test conditions: 24 hours at 40 °C). Rubber articles directly associated with the consumption of food and which are being, or are expected to be, taken into the mouth, e.g., toys according to Recommendation XLVII, teats, soothers, gum shields, balloons etc.

The following food simulants are used in connection with the German regulations: distilled water, 10% ethanol and 3% acetic acid. The permissible migration limits vary according to the category and simulant.

The BfR regulations also include a number of specific composition and migration limits. For example:

1. N-Nitrosamines and nitrosatable substances
2. Amines (all categories)
3. Milking liners and milking tubes
4. Formaldehyde
5. Acrylonitrile
6. Zinc dibenzylidithiocarbamate
Pysklo and co-workers have compared the BfR list of ingredients approved for food contact rubbers with the equivalent Polish list. The Polish list was originally based on the German list and this exercise, carried out in 2002, showed that it was in need of updating [9].

### 3.4.2 Silicone Rubbers

Recommendation XV of the BfR regulations covers silicone oils, silicone resins and silicone rubbers. The section on silicone rubbers stipulates acceptable starting materials and the additives that may be used in processing and manufacture – both types and maximum levels. Separate restrictions are stated where silicone rubber is to be used for teats, dummies, nipple caps, teething rings or dental guards. Dummies and bottle teats must also comply with the requirements laid down in the Commodities Regulation (Bedarfsgegenstandeverordnung). The amount of volatile organic material is restricted to a maximum of 0.5%, as is the total extractable material. Test methods are referenced for these determinations as well as a test for residual peroxides which should be negative.

### 3.5 Other European Legislation

#### 3.5.1 Requirements in France

French requirements for food contact elastomers (excluding silicones) are given in the Arrete of November 9th 1994 which is published in the Journal Officiel de la Republique Francaise, December 2nd 1994 pages 17029-17036 [10]. Four use categories (A to D) and a special category (designated T) are described together with a positive list detailing permitted ingredients in each category. There is an overall migration limit set at 10 mg/dm² (60 mg/kg), the same as for plastics. Other specific restrictions also apply, such as an SML for primary and secondary aromatic amines of <1 mg/kg.

#### 3.5.2 Requirements in the Netherlands

These regulations, which closely resemble the CoE rubber resolution, can be found in Verpakkingen en gebruikscaraktenblust (Warenwet), Chapter III. There are positive lists of approved additives and food contact rubber products are divided into three parts [11].
3.5.3 Requirements in Italy

Italian requirements are given in the decree of March 21st 1973 contained with the supplemento ordinario alla Gazzetta Ufficiale della Repubblica Italiana, April 20th 1973 pages 12 to 14. There have since been updates, including the decree of June 3rd 1994.

3.5.4 Requirements in the United Kingdom

The UK legislation on food contact materials is published as a number of Statutory Instruments which were published in 1978 and came into operation in November 1979.

The use of rubbers in contact with food is covered by the legislation included in Statutory Instrument 1987 No. 1523 ‘Materials and Articles in Contact with Foodstuffs’. This states that any food contact material should not be injurious to the health of the consumer and that any contamination should not have an adverse effect on the organoleptic properties of the food (i.e., taint and odour).

The absence of any positive lists for compounding ingredients means that UK rubber compounders normally refer to either the FDA or the BfR regulations depending on the market to be addressed.

There are separate rules for the use of rubber in contact with potable water. These are given in the United Kingdom water fitting bylaws scheme and include tests for the following:

a) Taste [12, 13, 14]
b) Appearance [15]
c) Growth of aquatic microorganisms [16]
d) Migration of substances that may be of concern to public health [17]
e) Migration of toxic metals [18]

The test methods for the above are given in BS 6920, Parts 1 to 4 (2000-2001) [12-18].