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Various food contact materials are used throughout the food chain and can give rise to migration (leaching) where chemical substances and metals can transfer to foods. Regulations are complex but a diversity of analytical techniques provide effective tools for monitoring and control.

Materials and articles intended to come into contact with food are used from the start to the end of the food chain. Foods are in contact with a variety of materials starting at the farm, during factory processing and packaging eventually to being used in the kitchen for preparation and cooking of food. The applications are diverse, as are the materials themselves, which include plastics, paper & board, rubber, ceramics, metal, glass, wood, and cork.

All of these materials must be safe in terms of not releasing chemicals or metals into foods that are of safety concern or cause taint to food. Worldwide this general principle is embedded in food contact materials legislation. In the EU there is a lengthy positive list of monomers and additives supported by specific migration limits. Regulation in the USA requires prior approval by the authorities for use of a new substance, whilst in Japan work is in progress to develop a positive list.

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Analysis plays a critical role in this area with identification of plastics by FTIR being useful to determine the type of polymer and perhaps some indication of the additives which are present. DART-MS has also shown potential for rapid screening of plastics for the presence of additives such as primary aromatic amines in polyamide utensils. Accelerated solvent extraction (ASE) is a valuable tool for isolating additives and other potential migrants from plastics. For the analysis of food and food simulants, a wide range of analytical techniques is employed to determine if migration has occurred. Headspace GC and GC-MS are both employed for monomer analysis, whilst HPLC and LC-MS are routinely used for the analysis of intermediate and low volatility additives. Elemental analysis by AAS or ICP-MS is important when studying leaching of heavy metals from ceramics and from metal cookware. LC-Orbitrap™ MS and GC-Orbitrap™ MS are both beginning to play an important role in the non-targeted analysis as food manufacturers and retailers now have the added responsibility to demonstrate that non-intentionally added substances (NIAS) do not present a food safety risk to consumers. The non-targeted analysis will play an increasingly important role in the future and innovations such as nanomaterials being used as food contact materials will present future analytical challenges.

Bisphenol A

Food packaging materials have frequently been the subject of high profile media scares ranging from BPA in polycarbonate baby bottles, photoinitiators in dry foods to mineral hydrocarbons from recycled cardboard, all being widely reported in the media. The public focus on the negative side of packaging and plastics, in particular, has led to very complex and detailed regulation in this area.

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From Farm to Fork

Food contact materials comprise any type of material which comes into direct or indirect contact with food or beverages at any point in the food chain, from the farm gate to the kitchen (from farm to fork). The materials can be diverse ranging from wood, ceramics, and metals to plastics, rubber and paper and board. For example, fruit and vegetables being harvested on the farm might be stored and transported in plastic crates, food factories might use conveyor belts, pipes, valves, mixing vessels, mechanical equipment and different types of containers for storage and transport. All of these articles are manufactured from materials which must comply with regulations establishing their suitability for use in contact with food.

After processing, food packaging can be fabricated from any one of a diverse range of plastics, paper and board, composites, glass, metal, or cork. Combinations of materials are frequently used for packaging such as plastic laminates, plastics coatings on paper-board and coatings on metal cans as well as different materials being used for primary and secondary packaging of food.

Adhesives and printing inks may not be in direct contact with food, but can nevertheless also contribute to the burden of chemicals migrating into food. When foods are being prepared in the home, cookware can be metal, ceramic, or plastic and other items such as spatulas, cutlery, kitchen surfaces, and chopping boards are all manufactured from various different types of food contact materials including wood.

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Migration

The concern about food contact materials is that they can transfer chemicals or metals to food or beverages at levels that have the potential to cause harm to human health or might cause organoleptic changes (taint) to foods. For plastics, paper and board, rubber, and silicones the process of transfer of chemicals is known as 'migration' and it is a diffusion process controlled by factors such as the type of material, size of migrant molecules, type of food, temperature, and length of time in contact between the material and food.

Thus, small molecules such as plastics monomers (e.g., vinyl chloride and styrene) move freely in the polymer and will rapidly migrate into liquids that are in intimate contact with the surface of the plastic. Larger molecules, such as some plastics additives move less freely in the polymer and migration will, therefore, be much slower. With dry foods or other solid foods, there is less intimate contact with the packaging material migration will also be slower. In the case of glass, ceramics, and metals the process is more one of 'leaching' than migration, and it is normally aggressive liquids such as alcoholic beverages or acidic foods, which can have a dissolving action leading to contamination arising mostly from the surface of the material. Thus, lead contamination can arise from 'cut glass' when alcoholic spirits are left in contact for an extended period or other metals can be leached by acidic foods (e.g., from the colored glaze of pottery or ceramic articles).

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Diversity of Additives

Despite the diversity of food contact materials, it is fair to say that most attention for food safety has focused on plastics, polymeric coatings and, more recently, on paper and board. The focus on plastics is due to the diversity of additives that are essential to convert a polymer into a finished article such as a bottle, tray, tub, or film, which need to be in a form that is suitable for the end use. Some plastics might need heat and processing stabilizers to prevent polymer decomposition during fabrication, whilst UV absorbers, antioxidants, and flame retardants are necessary for long-term storage. Pigments and fillers are widely used as colorants, whilst lubricants and blowing agents are necessary to aid fabrication, whilst anti-static agents, plasticizers and impact modifiers ensure the right physical properties needed for the intended end use. These additives and their transformation products are not 'bound' to the polymer chains and are free to migrate from the finished article into food. Plasticizers, in particular, are used at percentage amounts (5-25%) in films to impart the desired flexibility, by acting as a lubricant between the polymer chains, and can rapidly migrate into foods where there is intimate contact (e.g., cheese wrapped in PVC 'cling-film'). As well as the deliberately employed plastics additives, unreacted monomers which are not consumed in the polymerization process and other starting substances such as catalyst residues are also available for migration.

There are many hundreds if not thousands of potential chemicals which are used to manufacture plastics, and whilst most can be controlled through 'positive list' authorization, others such as impurities, additive transformation products, and oligomers cannot be controlled in this way and these are known as Non-Intentionally Added Substances (NIAS). It is the responsibility of polymer and packaging manufacturers and, ultimately, food and beverage retailers to ensure the safety of NIAS.

There have been large financial costs to the packaging industry in changing practices and re-formulating, but the more obvious costs have been in the recent scares related to photo-initiators in paperboard cartons, where there have been large-scale recalls of foods from the market.

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Diversity of Additives

Table 1 lists a few of the food scares which have occurred over the past 30-40 years related to food contact materials. The first real concern dates back to the 1970s when workers in the PVC industry were found to have a high incidence of angiosarcoma, which is a rare form of cancer, which was subsequently associated with exposure to vinyl chloride monomer (VCM). When VCM was found in beverages packaged in PVC bottles, the industry was forced to change PVC manufacturing practices to reduce residual VCM levels in PVC and the first regulations controlling specific migration were introduced in Europe. Over subsequent years there have been a series of media scares related to various migration scenarios, which have received different degrees of media attention. Mostly, the result has been that industry has either changed its practices or has been forced to switch from using one chemical to using another. With plasticizers, for example, there was a partial switch from using DEHA to using polymeric plasticizers with significantly lower migration behavior. In other areas such as the use of BADGE in can coatings, there was a switch to other substances as replacements. However, it was not always clear that the change was beneficial, particularly if the toxicology of the new substances was not established.

There is not only a financial cost to such scares but also reputational damage to major food brands. The issue of migration of mineral oils has been long-standing but has recently come to the fore again with concerns about migration of mineral hydrocarbons from recycled paperboard used to package dry foods. Media attention has forced major retailers to move away from recycled paperboard to reassure consumers.

Food contact material	Year	Food type	Contaminant	Consequences
PVC bottles	1974	Beverages	Vinyl chloride monomer	EEC Directive – limit in plastics & food
Rubber teats, soothers	1980s	Liquids & saliva	N-nitrosamines	Regulator limits
Regenerated cellulose film (RCF)	1980s	Confectionary	Diethylene glycol (DEG)	Media attention – regulatory limits
PVC cling-film	1980s	Fatty foods	DEHA & other plasticizers	Industry switch to polymeric plasticizer
Plastics films & gaskets	1980s	Baby food	ESBO	Industry switch to other plasticizers
Metal cans – coated surfaces	1990s	Canned oily foods e.g. fish	BADGE & related compounds	Industry switch to other monomers
Polycarbonate baby bottles	2009-2010	Milk & infant formula	BPA	Polycarbonate banned for baby bottles
Tetra-Pak paperboard cartons	2005	Baby milk	ITX photo-initiator	Milk (2.0 million liters) withdrawn by Nestle = €1.6 million
Printed paper & board	2006	Fish fingers, biscuits, fries…	Benzophenone photo-initiator	Media attention & industry action
Polyamide kitchen utensils	2004-2009	All foods – kitchen use	Primary aromatic amines (PAAs)	RASFF market recalls
Recycled cardboard	2010-2016	Dry foods e.g. breakfast cereals	Mineral hydrocarbons MOSH & MOAH	Industry phase-out use of recycled paperboard

Table 1. Showing some historical incidents related to migration from food contact materials

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There are large differences in the choice of materials which are used for retail packaging of food and beverages between different countries and also differences in the extent of packaging. In countries where 'ready-meals' are popular, a microwaveable plastic PET tray, a heat-sealed film as a lid contained in sealed printed paperboard secondary packaging carton, would not be uncommon. In some countries, cheese would be sold at retail counters wrapped in wax-coated paper or aluminum foil, but in other places plasticized PVC or LDPE cling-film might be used. Fast food outlets generally use paperboard or expanded polystyrene (EPS) cups for dispensing hot drinks and use EPS containers for burgers, fries and fried chicken. A list of a selection of plastics used in food packaging and their food and beverage applications is shown in Table 2. There have also been significant changes over the last few years in plastics food applications as a consequence of prices as well as developments in packaging technologies. The biggest change has been the decline in the use of rigid PVC for bottles and the huge rise in the use of PET for trays and bottles. The rise in the use of PET has in part been driven by increased consumption of bottled water (still and sparkling) and a move from aluminum cans into PET for beverage bottles although cans are still significant in this market.

Plastic type	Acronym	Applications
Low- & high-density polyethylene	LDPE & HDPE	Plastic milk bottles, tubs, jars, bottle caps
Polypropylene	PP	Bottles, tubs, water dispensers
Polystyrene	PS	Cream & yogurt tubs, drinking cups
Expanded polystyrene	EPS	Drinking cups and trays for fast food
Polyethylene terephthalate	PET	Bottled water, beer, carbonated beverages, trays
Polyvinyl chloride	PVC	Cling-film for retail and domestic wrapping
Polyvinylidene chloride	PVDC	Films for biscuits, snack products
Polytetrafluroethylene	PTFE	Coatings for non-stick pans
Polycarbonate	PC	Drinking bottles for babies
Melamine	MEL	Plastic tableware – plates, dishes, bowls
Acrylonitrile-butadiene-styrene	ABS	Margarine tubs

Table 2. Summary of major plastics used in food contact applications

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PET is a particularly attractive polymer for food applications as it contains very few additives compared with some other polymers and, therefore, has low migration characteristics. PET is also suitable for plastics recycling thereby contributing to the circular economy. Different materials give rise to different migration species and many of these chemicals for convenience are known by acronyms rather than the full chemical names. A selection of these substances which have come to prominence is indicated in Table 3.

Chemicals that have been found in food as a result of migration range from simple small molecules, such as vinyl chloride (VCM) and acrylonitrile, DEG, and BPA, to highly complex mixtures of chemicals, such as the mineral hydrocarbons and some plasticizers. They also range from known approved substances, which are authorized for use through a positive list and have been toxicologically evaluated, to adventitious and unavoidable contaminants often not previously known or sought.

The latter can be impurities in additives and starting substances, plastics additive transformation and decomposition products arising during fabrication of the finished articles and oligomers (short chain polymers) resulting from incomplete polymerization. These NIAS chemicals, which are not specifically approved but have the potential to migrate, are probably one of the biggest analytical challenges in the area of food contact materials.

Chemical name	Acronym	Food contact material applications
Vinyl chloride monomer	VCM	Monomer used in PVC production
Di-(2-ethylhexyl)adipate	DEHA	Plasticizer used in PVC cling-film
Di-(2-ethylhexyl)phthalate	DEHP	Plasticizer in printing inks and in some coatings
Epoxidized soyabean oil	ESBO	Softener used in PVC gaskets to seal jar caps
Bis-phenol A diglycylether	BADGE	Epoxy derivatives used in can coatings
Bisphenol-F diglycidyl ether	BFDGE	Epoxy derivatives used in can coatings
Novolac glycidyl ethers	NODGE	Epoxy derivatives used in can coatings
Bisphenol A	BPA	Monomer used in polycarbonate baby bottles
Isopropyl-9H-thioxanthen-9-one	ITX	Photo-initiator used in printing inks
Diisopropyl naphthalenes	DIPN	Recycled paperboard – carbonless copy paper
Mineral oil saturated hydrocarbons	MOSH	Originate from printing inks & recycled fibers
Mineral oil aromatic hydrocarbons	MOAH	Hot-melt adhesives & recycled fibers
Diethylene glycol	DEG	Softener used in regenerated cellulose film
Primary aromatic amines	PAAs	Polymer starting substances – plastics utensils

Table 3. List of major chemical contaminants in food resulting from migration

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Regulation in the USA

In the USA, migrants from food contact materials were previously known as indirect food additives, but the FDA now refers to these materials as food contact substances (FCS). There is a food contact notification process (FCN) that is operated within the Center for Food Safety and Applied Nutrition's (CFSAN) Office of Food Additive Safety. The FCN process requires industry to supply sufficient scientific information to demonstrate that the substance that is the subject of the notification, is safe for the intended use.

Food contact notifications are required only for new uses of food contact substances that are food additives. Although a notification is not required for a food contact substance that has 'Generally Regarded as Safe (GRAS)' status or is prior sanctioned for its intended use in contact with food, some companies notify FDA in order to clarify the regulatory status of such substances or notify new uses of FCSs even though they are GRAS or prior sanctioned.

An important feature of the approval process in the USA is the 'Threshold of Regulation' which recognizes that some substances may migrate into foods at levels that are so low as to be insignificant, and therefore safety evaluation is not necessary.

The threshold-of-regulation process is an abbreviated review process for uses of indirect food additives that result in an exposure below 1.5 µg per person per day. The threshold-of-regulation process can result in a faster approval; however, that approval is not proprietary.

Table 4 lists the sections of the Federal Regulations that cover different food contact materials, from which it can be seen that coverage of areas other than plastics is much wider than is the case in the EU. The main difference between the approval system in the EU and USA is that in the EU approval is for unrestricted use of positive listed substances for plastics, whilst in the USA approval is for designated uses. In the EU, the specific migration limits are set on an unrealistic and very conservative assumption that all foods will come in contact with the specified plastic material and, therefore, in a worse-case situation, the toxicological exposure level (expressed as a TDI) can never be exceeded. In the USA, each specified food application is authorized on the assumption that part of the TDI will be used for that application based on estimated exposure from only that food.

For example, for an additive to be used only in a plastic container for use for ketchup, the exposure will be estimated only on exposure from ketchup consumption and approval is granted on the basis that exposure will be say 50% of the TDI for that application.

Coding	Area covered by Federal Regulation CFR:21
21 CFR 173	Secondary direct food additives permitted in food for human consumption
21 CFR 175	Indirect food additives: Adhesives and components of coatings
21 CFR 176	Indirect food additives: Paper and paperboard components
21 CFR 177	Indirect food additives: Polymers
21 CFR 178	Indirect food additives: Adjuvants, production aids, and sanitizers
21 CFR 180	Food additives permitted in food or in contact with food on an interim basis pending

Table 4. Code of Federal Regulations covering food contact materials

If another petitioner wants to use the same additive for a different plastics application, approval will be based on not exceeding the remaining 50% of the TDI and once the TDI is fully used, no more authorizations can be granted for that additive. This offers the same level of protection as the EU in terms of the TDI, but has the advantage that it does not impose very low SMLs because exposure is based on actual use rather than an overestimation.

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Regulation in Japan

The Japanese regulatory framework for food packaging materials combines government regulations based on the 1947 Food Sanitation Law and standards that have been established by industry trade associations. The Japanese Food Sanitation Law sets out a general safety standard that covers not only food, but also food additives, packaging materials, and equipment, detergents for vegetables and fruits, utensils, and toys. Regarding food contact materials, the legislation prohibits the sale of materials and articles containing toxic or harmful substances that could be deleterious to human health.

Japan does not currently have a "positive list" of substances that are permitted for use in food contact materials, nor does it require pre-market approval or review of food-contact substances prior to their use. The Food Sanitation Law authorizes the establishment of specifications for food containers and packaging, and the raw materials used to manufacture such articles. The Ministry of Health is responsible for developing these specifications which that apply to all containers and packaging, that are material-specific standards, and some that apply to the end-use application for the packaging. The general specifications address the use of certain metals, particularly lead, in various food contact applications and prohibit, for example, the use of di-(2-ethylhexyl) phthalate as a plasticizer for PVC used in contact with foods containing oils and fats. Colors intended for use in packaging materials must be approved unless it can be shown that they do not migrate into the food.

The positive list system will take into account information on chemical identity, use level, migration level, as well as toxicity. Synthetic resins are the primary focus of the new positive list system, but monomers are also included due to the potential presence of harmful substances. Additionally, while additives are going to be among the main targets of the positive list system, it is also intended to eventually regulate colorants, adhesives, coatings, and printing inks.

In 2015, discussions started concerning future regulation of food contact materials, including the introduction of a new "Positive List" system to cover synthetic resins, paper, rubber, metal, and glass.

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EU regulations

In the EU, despite an apparently logical framework for the progressive development of regulations for food contact materials and articles, the immediacy of some issues, that have arisen over the years, have otherwise influenced priorities. Some seventeen groups of materials and articles were listed alphabetically in what is known as the Framework Directive 1935/2004, for which it is indicated these groups may at some future time be covered by specific measures. These materials are:

			(13) Silicones
(1) Active and intelligent packaging	(5) Rubbers	(9) Paper and board	(14) Textiles
(2) Adhesives	(6) Glass	(10) Plastics	(15) Varnishes and coatings
(3) Ceramics	(7) Ion-exchange resins	(11) Printing inks	(16) Waxes
(4) Cork	(8) Metals and alloys	(12) Regenerated cellulose	(17) Wood

(1) Active and intelligent packaging; (2) Adhesives; (3) Ceramics; (4) Cork; (5) Rubbers; (6) Glass; (7) Ion-exchange resins; (8) Metals and alloys; (9) Paper and board; (10) Plastics; (11) Printing inks; (12) Regenerated cellulose; (13) Silicones; (14) Textiles; (15) Varnishes and coatings; (16) Waxes and (17) Wood

It is also clearly laid down in a separate Regulation 2012/2006 that food contact materials and articles as with foods must be produced in compliance with Good Manufacturing Practice (GMP). Of the seventeen groups of food contact materials, plastics have always been the main priority, as they have been seen historically to present the most potential food safety risks.

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Table 5 lists some of the EU regulations related to materials and articles other than plastics. One of the longest standing regulations goes back to 1984 and controls the leaching of lead and cadmium from ceramics intended to be used in contact with food. The migration limits depend on the dimensions of the ceramic containers, but are set at 0.3 mg/L for cadmium and 4.0 mg/L for lead for containers, which can be filled with lower limits applied to vessels with a capacity greater than 3 L. Testing is with 4% acetic acid at 22 °C for 24 hours using a test method which must meet minimum performance criteria stipulated in the regulations. Concerns related to regenerated cellulose film arose in 1986 leading to a positive list of those additives that can only be used in these materials and a limit of 30 mg/kg for diethylene glycol migration into foods. Issues related to epoxy can coatings led to Regulation 1895/2005 restricting use of BADGE, BFDGE and NOGE, whilst as early as 1993 the potential for formation of *N*-nitrosamines in elastomers and rubber baby teats and soothers led to a limit of 0.01 mg/kg release of *N*-nitrosamines into artificial saliva.

Sometimes it has not been food scares that have influenced the regulators, but it has been rapid and uncontrolled developments and the introduction of novel products into the market place, which for example led to regulations in 2009 controlling active and intelligent packaging. Active packaging has the potential to extend the shelf-life and quality of foods, through packaging components interacting with the headspace gas above a packaged food by removing (scavenging) gases or by introducing additives (e.g., antioxidants that have not been directly added to the food).

EU Regulation or Directive	Scope of regulation
500/84/EEC	Pb & Cd migration limits from ceramics, test conditions & method
11/93/EEC	*N*-nitrosamines and *N*-nitrosatable substances in elastomer & rubber teats
1/2004/EC	Suspension of the use of azodicarbonamide as blowing agent
1935/2004/EC	Framework directive for food contact materials & articles
1895/2005/EC	Restriction of use of certain epoxy derivatives in can coatings
2023/2006/EC	Good manufacturing practice (GMP) for food contact materials
42/2007/EC	Regenerated cellulose film – positive list and restrictions
327/2007/EC	Migration limits for plasticizers in gaskets in lids
450/2009/EC	Active and intelligent materials & articles

Table 5. EU regulations for food contact materials other than plastics

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EU regulations

For plastics in the EU, there has been a series of regulations, which initially set out conditions for migration testing and a positive list of substances that could be used as monomers and starting substances.

Numerous amendments and updating took place over many years, as the science progressed, and this led to a confusing situation with a complexity of regulations, some superseding one another. A major reform of the legislation took place in 2011 with 10/2011/EC replacing and revoking all previous regulations on plastics as well as extending and further updating the regulations. This very extensive regulation contains a full list of authorized monomers and starting substances, additives, and processing aids for the manufacturing of plastics with certain restrictions in some cases.

This list which is now 983 substances (updated from 885 substances published in the 2011 regulation) is accessible through an EU database which can be searched by substance and by restrictions

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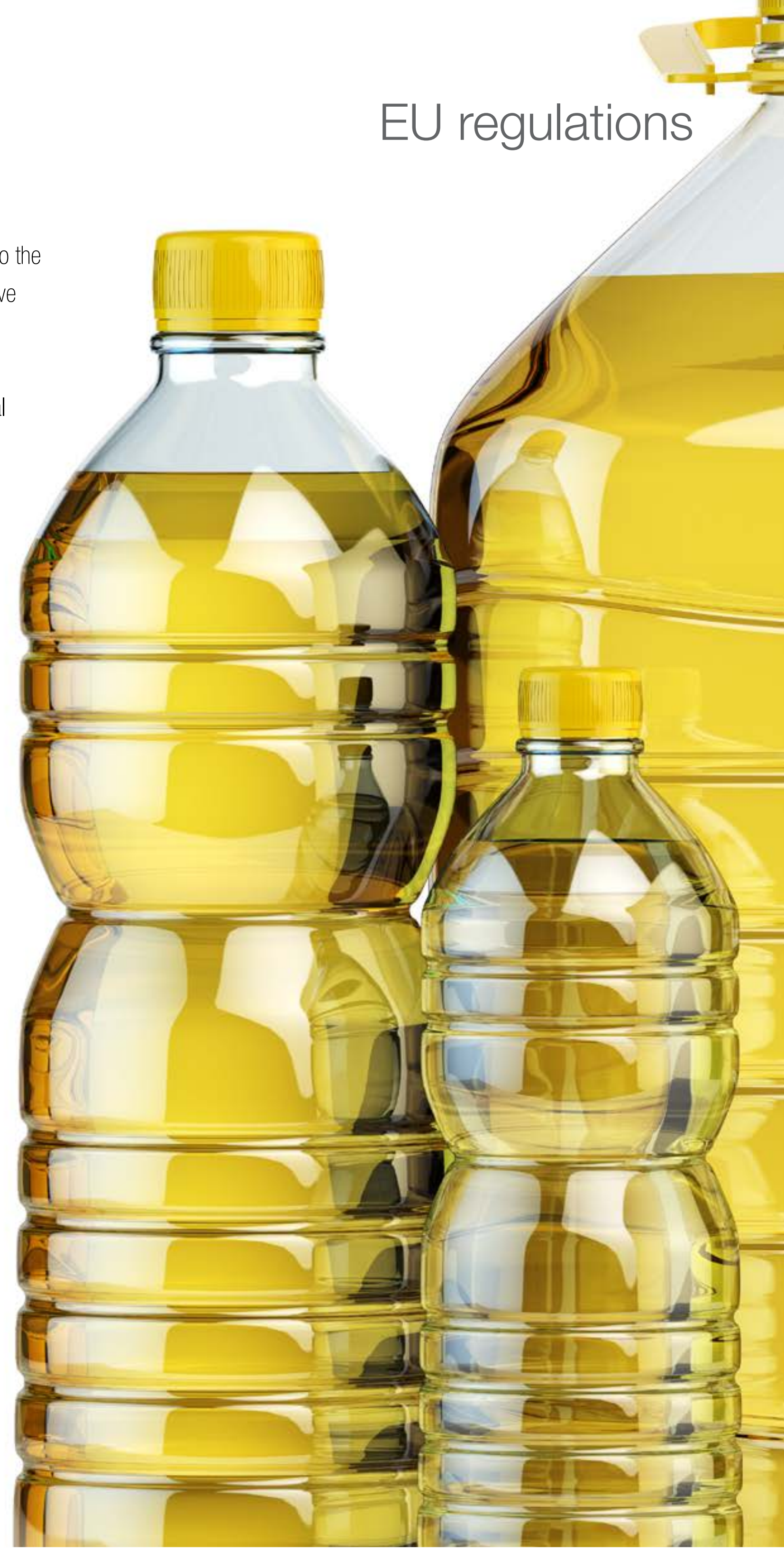
There is now a well-established process for packaging manufacturers for a submission of a dossier to the European Food Safety Authority (EFSA) for an evaluation of any new substances that they wish to have added to Table 1 of Regulation 10/2011. Detailed chemical and physicochemical data are required characterizing the substance, its manufacturing process, impurities, and required use. Its migration behavior under intended conditions of use needs to be submitted as well as a dossier of toxicological data which is prescribed, depending both on its potential to migrate as well as the presence of any structural alerts in its chemical structure. After an EFSA evaluation an Opinion is published which, even if favorable, may contain some restrictions for use. The EFSA Opinion, which is a risk assessment, is used by the European Commission to make a risk management judgement as to whether the substance will be authorized and added to the existing positive list.

There are some important concepts that exist in EU plastics regulations which are unique to the EU. There is the concept of 'overall migration' or 'global migration' which is seen as a quality, rather than a safety measure, and limits the total quantity of substances migrating into food simulants to a maximum of 60 mg/kg or 10 mg/dm^2. As it is impossible analytically to measure overall migration into real foods, the notion of 'food simulants' was therefore created. These food simulants are liquids, except for one solid material used to simulate dry foods, which are used to mimic the behavior of foods in terms of their interactions with plastics. Water, 3% acetic acid, ethanol solutions (10%, 20%, and 50%), vegetable oil, and poly-2,6-diphenyl-*p*-phenylene oxide known as Tenax™ are the recognized food simulants.

The aqueous simulants are used to test for overall migration using a gravimetric procedure, whereby after a period of contact between the plastic and simulant, the simulant is evaporated to dryness and the residue is weighed. For vegetable oil, which is a more aggressive food simulant for some plastics, and evaporation is clearly not possible, a rather lengthy and somewhat problematic procedure is used to measure overall migration. This method combines some gravimetric steps in terms of weighing the plastic before and after contact with the oil, with a gas chromatographic procedure to determine the quantity of oil which has diffused into the plastic.



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The list of 885 authorized monomers, other starting substances, macromolecules obtained from microbial fermentation, additives, and polymer production aids given in tabular format in Regulation 10/20011 indicates restrictions on use. In some cases, the restrictions are on the maximum residue amount (QM) allowed in the finished plastic article, whilst in other cases a specific migration limit (SML) is specified.

The SML applies to testing undertaken with the appropriate food simulant relevant to the intended end use of the plastic article under the intended conditions of use. SMLs range from ND meaning the substance shall not migrate in detectable quantities. This is not a very helpful concept as it clearly depends on the sensitivity of analytical procedures, which are always progressively improving, but in general a limit of 0.01 mg/kg has been adopted as being equal to ND. Where SMLs are given numerically they range from 0.02 mg/kg for substances such as methacrylic acid, 2,3-epoxypropyl ester to levels as high as 42 mg/kg for ricinoleic acid. Some limits are expressed as the total migration of several substances, where they have similar structural characteristics and this is indicated as SML(T). For example, six individual benzophenones have an SML(T) of 6 mg/kg which means that the sum of migration of the individual benzophenones must not exceed 6 mg/kg if more than one of these is used in the same formulation.

Specific migration testing is generally conducted using food simulants and 10/2011 provides the rules for deciding which simulant to use for which specific intended food application. In some instances for complex foods, more than one simulant should be used although compliance should be demonstrated with the more aggressive simulant. In some instances, it is recognized that migration testing (e.g., with vegetable oil) is far more aggressive than would be the case with a real food and 10/2011 provides reduction factors which are applied to the migration test result to compensate for this overestimation.

Although it is clearly analytically easier to conduct migration testing with food simulants, the regulations do allow testing to be conducted with real foods, which does give more meaningful results, and with modern analytical instruments is more readily achievable than was historically the case when legislation was originally put in place. The conditions for conducting migration testing are also specified in detail in Regulation 10/2011 depending on the intended end use, which in practice can vary from single use short-time contact at ambient temperature to repeat-use at high temperatures (cooking utensils) to long-term storage. The Regulation gives a range of worse foreseeable use times from <5 min to >30 days and a range of worse foreseeable use temperatures from <5 °C to >175 °C and indicates for each range the test conditions of time and temperature that should be applied. If the material or article is intended to come into repeated contact with foods, the migration test is carried out three times on a single sample using another portion of food simulant on each occasion. Compliance is checked on the basis of the level of the migration found in the third test.

Interestingly the EU regulations allow for the use of mathematical modeling to estimate the maximum level of a substance that would be found in food if testing had been conducted. To screen for specific migration the migration potential is calculated based on the residual content of the substance in the material or article applying generally recognized diffusion models based on scientific evidence that the models are constructed such as to overestimate real migration. There are a number of mathematical diffusion models which have been validated and are commercially available as a software package. There are also very helpful practical guidelines on the application of migration modeling for the estimation of specific migration which has been published by the European Commission joint Research Centre.

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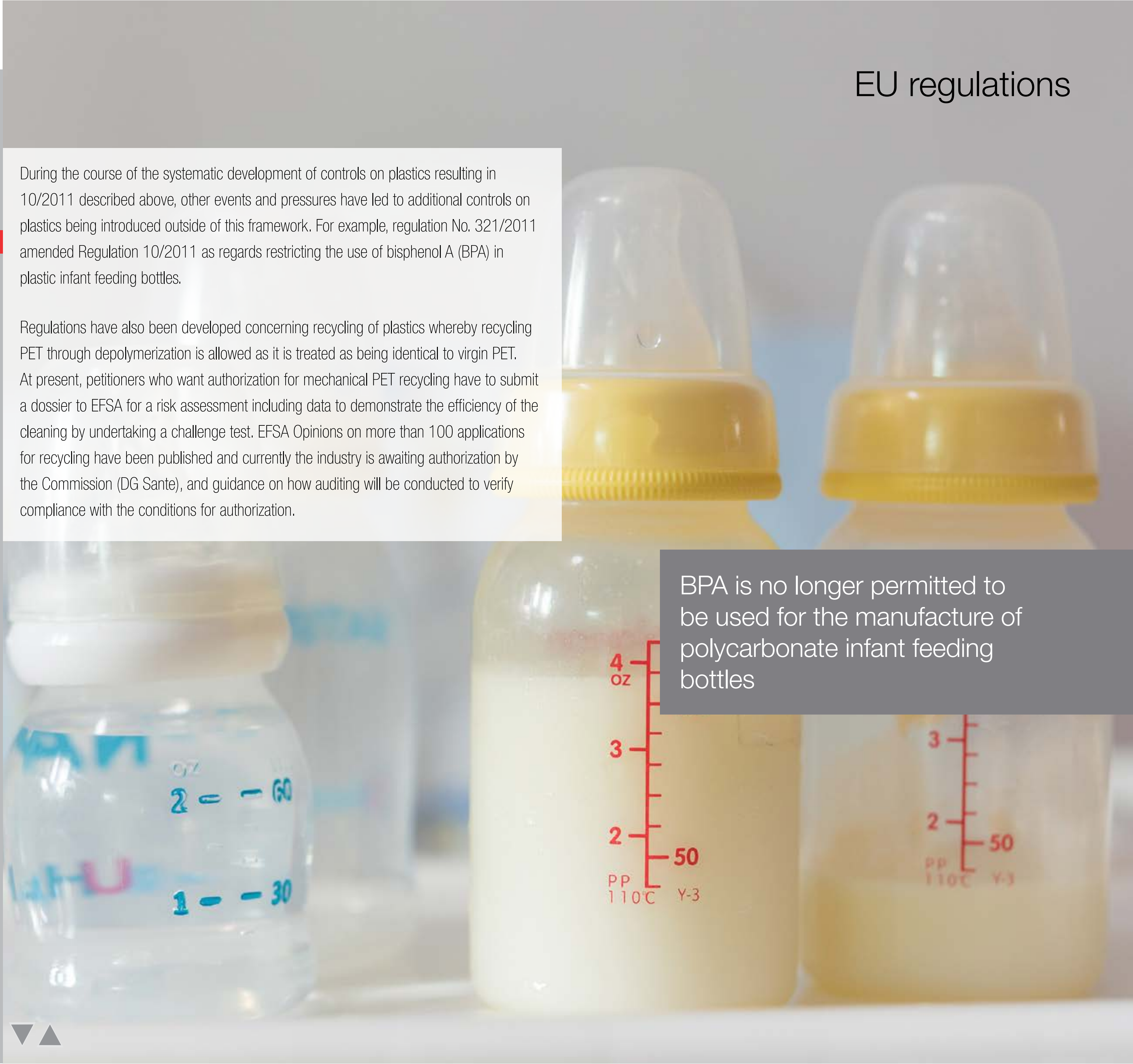

During the course of the systematic development of controls on plastics resulting in 10/2011 described above, other events and pressures have led to additional controls on plastics being introduced outside of this framework. For example, regulation No. 321/2011 amended Regulation 10/2011 as regards restricting the use of bisphenol A (BPA) in plastic infant feeding bottles.

Regulations have also been developed concerning recycling of plastics whereby recycling PET through depolymerization is allowed as it is treated as being identical to virgin PET. At present, petitioners who want authorization for mechanical PET recycling have to submit a dossier to EFSA for a risk assessment including data to demonstrate the efficiency of the cleaning by undertaking a challenge test. EFSA Opinions on more than 100 applications for recycling have been published and currently the industry is awaiting authorization by the Commission (DG Sante), and guidance on how auditing will be conducted to verify compliance with the conditions for authorization.

BPA is no longer permitted to be used for the manufacture of polycarbonate infant feeding bottles

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Council of Europe resolutions

The Council of Europe (CoE) is an intra-governmental body of EU and non-EU countries comprising 47 member states. The CoE advises on the regulation of many different social and legal areas, including food contact materials. Paper and board as a natural food packaging material has a remarkably long history of safe use and consequently there has been no great pressure to regulate in this area. There has however been a CoE Resolution AP (2002)1 on paper and board, which has served as an important reference in the EU and in the absence of harmonized regulation of paper and board has emerged as an important consideration for many manufacturers of paper and board materials and components.

This resolution applies to all food contact paper, including coated board and multilayers, but does not apply to non-woven materials, kitchen towels, napkins, and filter materials that are of a high base weight. Generally, compliance with the resolution requires that materials satisfy the safety requirements of Article 2 of the Framework Directive, are manufactured in accordance with good manufacturing practice (GMP), are manufactured from materials with the lowest possible level of dioxins, and do not release antimicrobial substances. The resolution contains at various stages of development a list of permitted substances as well as outline restrictions on heavy metal and organic contaminants. Realistic migration and extraction test methods for paper and board are not as easy to develop as for plastics but have been under consideration for some time. Most issues related to paper and board that have received prominence have concerned recycling of paper and board where the concerns have been chemicals derived from printing inks such as photoinitiators or have concerned mineral oils transferred during the recycling process.

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Council of Europe resolutions

To date, no specific EU harmonized legislation on printing inks for food packaging has been issued. However, even if printing inks are applied on the non-food contact surface of packaging, as a component of the printed package, they must comply with Regulation (EC) No 1935/2004 in terms of not endangering human health or changing the composition of the food or altering the organoleptic properties of the food. Printing on the non-food contact surface of a film can transfer to the food contact surface through what is known as 'set-off' when the film is stored as a roll. For other containers, such as tubs, which are printed on the outside but stacked one inside the other, again 'set-off' can lead to transfer to the food contact surface. There are also well-established cases of printing on the outside of packaging, but then gradual diffusion through the package, which does not necessarily provide a functional barrier, leading ultimately to food contamination. In 2005, the Council of Europe adopted the Resolution ResAP (2005)2 on "Packaging Inks Applied to the Non-Food contact Surface of Food Packaging."

Printing inks are a very complex area with much confidential information on the composition of inks. The substance inventory lists that were drafted were viewed as not sufficiently comprehensive, and there was also a view that they did not provide sufficient protection for consumer health or reflect current practices. Regulating the use of printing inks for food contact materials is, however, a priority area for the European Commission.

thermo scientific

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European Reference Laboratory (EURL)

The European Reference Laboratory for Food contact Materials (EURL-FCM) is located at the JRC Ispra (Italy) and is part of the Directorate for Health, Consumers and Reference Materials. Supported by a network of National Reference Laboratories (NRLs) the EURL provides scientific and technical assistance to DG Sante and to EU Member States, organizes inter-laboratory comparison exercises and conducts training courses for the benefit of EU food control laboratories and also of experts from third countries.

The EURL has successfully worked with the NRLs to develop technical guidelines for the control bodies to provide a unified approach for the practical implementation of EU food contact material legislation. Although this legislation is frequently very clear as to the intended principle of what is required, it frequently leaves substantial scope for interpretation and lacks practical advice on implementation. Guidance documents have therefore been developed on how to apply the fat reduction factor, what is meant by the functional barrier concept, and a clarification of the QM limit and SML for phthalate control from single and repeat-use articles. Guidelines on test conditions for kitchenware have been prepared as well as more specific guidelines for testing for primary aromatic amines from polyamide utensils and for formaldehyde from melamine-based kitchenware. A calculator has been developed for the correction of experimental specific migration data for comparison with the legislative limit, and a task force on migration modeling has examined the applicability of generally recognized diffusion models for the estimation of specific migration in support of EU Directive 2002/72/EC. As methods of analysis for migration have distinct differences from other methods used in the food contaminant area, guidelines have been developed to show how to evaluate method performance and conduct validation studies of analytical methods for food contact materials. These guideline documents are all freely available from the EURL website.

The EURL-FCM maintains a reference collection of plastics monomers and additives. Each applicant for the evaluation of a substance by EFSA is requested in the guidance document on the submission of a dossier on a substance to be used in food contact materials must submit a physical sample of 250 g to the EURL. Upon request aliquots of this sample can be made available for research purposes at no charge.

This collection is accompanied by a database of substances which contains searchable identification, characterization and specification information on substances including substance name, alternative name, URN, CAS or PM number, migration method, and analytical technique. Started in 1996, the database now has more than 500 entries.

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Rapid alert system for food & feed (RASFF)

The RASFF provides an informative record of notifications between EU member states as to food and feed samples which have been identified by food control authorities as being non-compliant with EU regulations.

These notifications can result in imports being rejected or a product recall from the market. In a 5-year period from 2012-2017, there were 507 notifications to RASFF related to food contact materials. A closer examination of specific alerts gives some impression of practical compliance issues, as well as indicating the priorities of the control laboratories, as analysis is targeted so there is a presumption that this indicates their particular priorities. What is striking is the fact that the majority of alerts relate to kitchen utensils and cookware rather than packaging. There were 152 instances of melamine articles, such as bowls, cups, spoons, plates, and cooking utensils from China, found to contain unacceptably high levels of formaldehyde or melamine monomer, which is a particular concern as many of these articles are intended for use by babies and infants.

There were 135 instances of kitchen utensils, such as spoons, ladles, and spatulas intended for cooking, which were found to give high levels of migration of primary aromatic amines and these occurred throughout the 5-year period. It is surprising that with this high level of rejection of these products and the costs incurred by the producers, that the issue was not resolved sooner. There were 65 cases of articles exceeding the overall migration limit of 60 mg/kg of which a large number related to metal utensils and cookware, but also several cases of plastic gloves intended for food handling where the limit was exceeded. There were a further 42 instances of leaching of various metals (chromium, manganese, nickel) from articles such as forks, spoons, knives, barbeque grill, bakeware & oven tray, pasta maker and even a meat grinder and toaster.

In many of these samples of metal items, not only were levels of specific metals above limits, but also the overall migration limit of 60 mg/kg was also exceeded making these samples unsuitable on both quality and safety grounds. It is also notable that there were many instances where the focus was on gaskets used to seal metal lids of which there were 20 cases involving excessively high levels of migration of epoxidized soya bean oil (ESBO) and many others where the issue was the migration of various phthalates. Other rejections were related to high levels of organic volatiles (1-2%) being released from drinking bottles, silicone cake molds & bakeware, migration of coloring materials as well as various monomers migrating from plastic drinking bottles. As these utensils would not have not been previously used, migration testing is reasonably easy to conduct with food simulants, as is also the case with plasticized gaskets used to seal glass jars where the food may not have been in direct contact. For packaging containing a foodstuff, compliance with migration limits can only be conducted by analysis of the food itself, which requires a more sophisticated instrumental approach preferably with mass spectrometric detection for unequivocal identification.

Analytical approaches to identify packaging and measure migration

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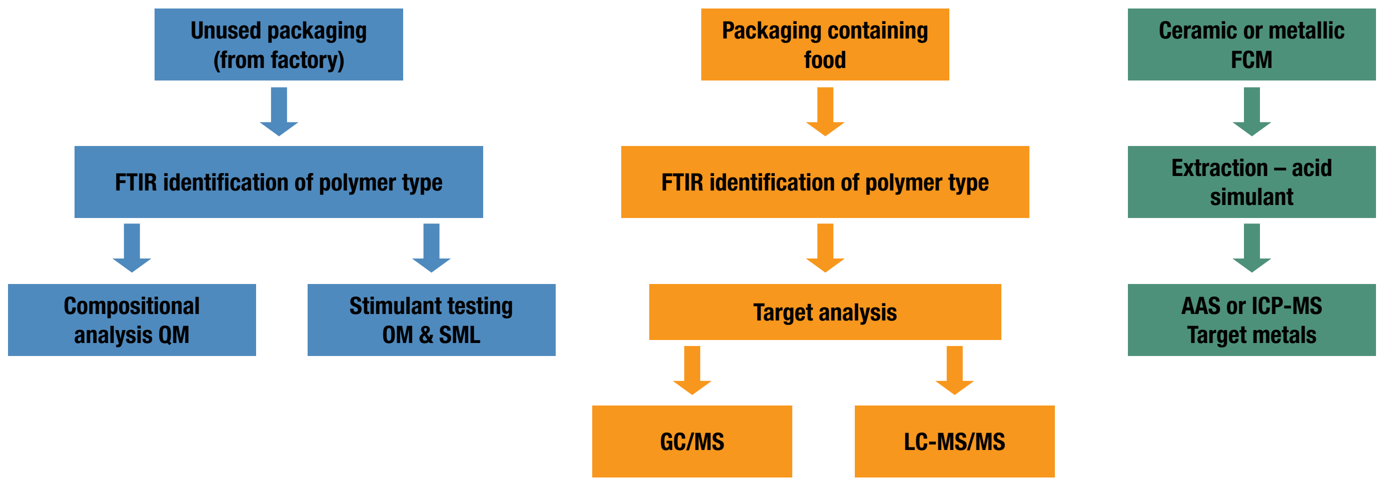
The ideal situation for accessing whether a food contact material is compliant with regulatory requirements is to obtain the unused material (tub, bottle, tray, or film) and then conduct migration tests with food simulants. It is only with virgin plastics packaging materials that it is possible to undertake overall migration testing. However, in many situations only the packaged food is available, so testing with simulants is not achievable and it is, therefore, necessary to carry out trace analysis of the foodstuff itself. In this situation, the starting point is to characterize the type of packaging material (particularly the surface in contact with the food) to provide an insight into the potential migrants which should be sought. There is no point in undertaking an analysis for a monomer or plastics additives unless one is sure it is present in the plastic and therefore available for migration. For example, there is no point in looking for formaldehyde migration unless one is sure that the plastic is actually made of melamine resin. For this target analysis, a good knowledge of polymer science and plastics technology is helpful in directing the analysis towards the most likely migrants.

The situation of testing unused packaging and when only packaged food is available is illustrated in the above schematic. Identification of both the polymer type and some of the additives present can be achieved by FTIR, or DART-MS might be fruitful in the detection of additives. An alternative approach can be conventional solvent extraction e.g., with diethyl ether or Accelerated Solvent Extraction (ASE) and then analysis by GC-MS or LC-MS for identification of additives and oligomers prior to target analysis of the food itself. Where there is interest in leaching of metals (e.g., from ceramics or cookware), the container is left in contact with acidic food simulant for a specified time/temperature and the simulant analyzed for target metals by atomic absorption spectroscopy (AAS) or ICP-MS.



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Identification by FTIR

FTIR in transmission or reflectance mode can provide IR spectra that can be matched against library spectra of different polymers to identify the type of plastic being used as food contact materials, bearing in mind the presence of additives, which will give additional bands in the spectra. In food packaging, most films are not single polymers, but invariably laminates for which multiple polymers are selected to produce in combination the desired physical and barrier properties of the material. FTIR with a microscope attachment such as the Thermo Scientific™ Nicolet™ iN™10 Infrared Microscope using Thermo Scientific™ OMNIC™ Picta™ software makes easy work of identifying each polymer in a laminate. Instead of manually searching layer by layer for the best match in search libraries, the wizard creates classes of materials in the cross section, searches all of them against the libraries, and assigns material identification and best match score to each layer. The information is then re-grouped considering the distribution across the section calculating the thickness of each layer. As an output, a table displays each material with a specific color, each layer as a spectral search match and the thickness calculated by spectral similarity.

FTIR analysis of plastic plaques in transmission mode is an excellent technique for multi-component quantitative analysis providing insights into the additives, which have been used to manufacture the finished article. It has been demonstrated that a number of ingredients of a polyethylene plaque can be identified, including multiple antioxidants, UV stabilizers etc. Where phthalates are being targeted a practical approach is to rapidly screen for the presence of these plasticizers in plastics initially using attenuated-total-reflectance IR (ATR-IR). If this does not give an unequivocal result, the sensitivity can be increased by a factor of 50 by hot pressing a 500-micron thick film and then acquiring a transmission spectrum by measuring the infrared energy getting through the sample. This technique enables the detection of phthalates at levels below 0.1% if other additives in the plastic formulation do not cause spectral interference.

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Identification of potential migrants in plastics by DART-MS

Although direct analysis in real time (DART) coupled to mass spectrometry (DART-MS) is, at the present time, a research tool in the area of food contact materials, it does show enormous potential. The drawback of currently used routine methods to identify additives in plastics even screening methods is that they all require a time-consuming (hours) extraction or a migration step prior to analysis. In contrast, DART-MS allows for the direct detection of compounds, with limited or no sample preparation, producing analytical results in minutes. To screen for primary aromatic amines in polyamide kitchen utensils, sample preparation for DART-MS analysis simply involves cutting small pieces (approx. 1 × 10 mm) from different locations of the food contact surface of each kitchen utensil. Each piece is manually placed using tweezers and held in the helium plasma of the DART ion source, close to the exit grid, for approximately 5 seconds and spectra can be recorded. In a wider study of different plastics/additive combinations it was demonstrated that 13 common food-packaging additives, including plasticizers, antioxidants, colorants, grease-proofing agents, and UV light stabilizers could be detected by DART-MS in samples of HDPE, LDPE, PVC/PVDC films, PET bottles, PVC gaskets and grease-proofed fast food sandwich-wrap paper.

DART-MS spectra for packaging additives appear to be quite unique, producing predominately molecular ions. The sensitivity, which can be an issue generally with DART, is less of a problem with food contact materials as the additives of interest are normally present at relatively high concentrations.

DART-MS using an Orbitrap MS with the additional specificity of producing high-resolution spectra has been used to screen for printing ink photoinitiators present on the surface of films as a result of 'set-off'.

Although the speed of being able to obtain results quickly by DART-MS is a tremendous advantage, it has been shown that there is a need to use multiple analytical techniques, when screening for these products in order to avoid the limitations of each technique and manage the incidence of false-positives and negatives. DART-HRMS has however provided unique insights into the detection of print-related compounds not included in the positive list of the EU FCM legislation, several of which were novel photoinitiator transformation products. This demonstrates the potential of DART-HRMS for future work on the untargeted analysis of food contact materials in order to detect compounds which are candidates for subsequent food analysis as packaging contaminants.

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Extraction of plastics and other food contact materials

Although non-destructive techniques like FTIR and DART-MS can be invaluable for characterizing plastics additives and other substances, both techniques are selective in what they see, and will not necessarily indicate all potential migrants. Extraction of food contact materials with appropriate organic solvents (e.g., using Soxhlet extraction) is exhaustive, can be time-consuming, and may also dissolve components which will probably never be significant in terms of migrating into foods. Accelerated solvent extraction (ASE) is a powerful technique that can be used to reliably extract additives from polymer materials. ASE uses organic solvents at temperatures above their atmospheric pressure boiling points to deliver extractions equivalent to traditional extraction techniques, but with faster extraction times, reduced solvent use, and automation of the extraction process.

Typically a plastic material would be ground into a powder perhaps under liquid nitrogen and then using a Thermo Scientific™ Dionex™ ASE™ Accelerated Solvent Extraction system, quantitative extraction could be undertaken in 20 minutes using a suitable solvent selected to dissolve additives whilst not dissolving the polymer.

The extract is then analyzed by HPLC with suitable detection or LC-MS/MS to identify the extracted additives and other components including NIAS.

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Techniques for analysis of food simulants & foods

The study of migration from food contact materials into foods is fairly unique insofar as migration species vary from the very volatile like vinyl chloride, which is a gas, to low volatility additives and oligomers and this wide range of compound type brings into play nearly all chromatographic and detection techniques. The very volatile compounds are best analyzed by headspace GC, preferably headspace GC-MS, employing either static headspace or solid phase microextraction (SPME) techniques. The Thermo Scientific™ Exactive™ GC Orbitrap™ GC-MS system now offers the possibility of focusing on previously unidentified volatile migrants harnessing the power of accurate mass measurement. Additives and other compounds of intermediate volatility can frequently be analyzed by either GC-MS or LC-MS, and whilst it is only species with a molecular weight below 1000 daltons which are of food safety interest, the higher molecular weight compounds are best determined by LC-MS (LC-MS/MS and/or LC-HRMS). In addition to organic migrants, there is also a need to determine migration or leaching of metals such as cadmium and lead from ceramics, chromium, manganese, and nickel from cutlery and kitchen utensils as well as metals such as antinomy in plastics such as PET, where it might be present as a catalyst residue. Traditional instrumental techniques such as atomic absorption spectroscopy (AAS) are used routinely, but ICP-MS is being used increasingly where multi-element capability is required. An indication of the techniques, which are used to measure both different classes of migrants as well as individual species, is shown in Table 6.

Migrants	Instrumental technique								
	GC	HS GC-MS	GC-MS	LC-MS/MS	ICP-MS	LC-Orbitrap	GC-Orbitrap	DART- MS	LC-GC
Total migration	X								
Monomers	X	X	X				X		
Plasticizers			X					X	
Additives			X	X		X	X	X	
Mineral oils	X								X
Cr, Ni, Mn, Sb					X				
BADGE				X			X		
BPA			X	X					
PAAs				X				X	
Untargeted						X	X	X	

Table 6. Selection of appropriate analytical technique for different migration species

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Official methods of analysis for migration

For at least 25 years, the European Normalization Committee CEN/TC 194 now called 'Utensils in contact with food' has been working on developing official methods for migration testing.

Originally only focusing on migration from plastics, the scope of TC194 has subsequently been broadened to include cookware, cutlery, utensils, and ceramics. There are in total some ninety-five CEN standards which have been published, of which sixty-two standards relate to testing methods for overall migration into food simulants, together with HPLC and GC methods to determine residue levels of individual substances in plastics (QM levels) and to test for specific migration with respect to SMLs.

The earlier published test methods have been updated, so that the oldest now dates from 2002. All methods are only accepted by CEN for adoption as standards if there is full inter-laboratory validation data in support of the method, with the performance data for methods needing to meet specific acceptance criteria.

Of the twenty-eight specific migration methods, those for acrylonitrile, 1,3-butadiene, 4-methyl-pentene, and vinylidene chloride are for testing in food simulants by headspace GC. Methods for 1,3-butadiene, vinylidene chloride, carbonyl chloride, ethylene oxide, and propylene oxide are for determining residue levels in the plastics again by GC analyzing the headspace gas above a solution of a dissolved polymer. Methods using HPLC have been published for the determination of terephthalic acid, monoethylene and diethylene glycol, acetic acid, vinyl ester, acrylamide, and 11-aminoundecanoic acid. 1,3-benzenedimethanamine, BPA, 3,3-bis(3-methyl-4-hydroxyphenyl)-2-indoline, caprolactam and caprolactam salt, 1,2-dihydroxybenzene, 1,3-dihydroxybenzene, 1,4-dihydroxybenzene, 4,4'-dihydroxybenzophenome, 4,4'-dihydroxybiphenyl, dimethylaminoethanol, ethylenediamine, formaldehyde, hexamethylenetetramine, hexamethylenediamine, maleic acid, maleic anhydride, 1-octene, tetrahydrofuran, 2,4,6-triamino-1,3,5-triazine, and 1,1,1-trimethylolpropane. Additionally, standards are available for HPLC analysis of isocyanates and epichlorohydrin in plastics. As these standards relate to the analysis of food simulants with the exception of vegetable oil there is little or no background interference allowing UV or fluorescence detection to be reliably employed. Due to the problems in using vegetable oil frequently, ethanol or hexane is used with correction factors being employed to allow for the more aggressive nature of the solvents compared to oils. Only one of the standards that of acrylamide is applicable for food analysis by headspace GC.

There are detailed CEN standards providing guidance on how to conduct overall and specific migration tests. This guidance covers preparation of samples whether plaques, pouches or whether testing is by filling containers, selection of conditions of exposure to food simulants as well as guidance on validation and interpretation of analytical methods, migration testing and analytical data for materials and articles in contact with food (CEN/TR 15356-1:2006 & EN 13130-1:2004).

Standards have been developed for paper and board in the absence of EU legislation and are focused on the determination of specific substances (organic and elements) in pulp or extracts or overall migration. They also target the preparation of aqueous and organic extracts or the determination of certain aspects/contents such as color fastness, dry matter content, tests with dry simulant, transfer of antimicrobial constituent, sensory analysis, or cytotoxicity.

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GC and GC-MS in migration testing

There are a wide range of plastics additives that are amenable to GC and GC-MS analysis after suitable preparation of the food sample prior to analysis. Mineral hydrocarbons are used at 3-6% levels as flow promoters in expanded polystyrene drinking cups, and in polystyrene and ABS containers (e.g., in individual servings of milk used in catering establishments). Wax coatings on cheese, waxed paperboard cartons for milk and cream and casings for sausages are all further potential sources of migration of mineral oils. These oils are complex mixtures of hydrocarbons which are recognizable by an envelope of peaks, but which have no characteristic mass spectrometric fingerprint. For food samples which have been in contact with these mineral oil containing materials, hexane extraction, saponification of fats, permanganate oxidation and SPE clean-up have all been employed in various combinations to isolate the hydrocarbons and remove co-extractives from the food. Clean chromatograms are achievable by GC-FID analysis where the individual hydrocarbon peaks can be readily recognized. Analysis by GC-MS offers no advantage compared to using an FID, which is, in fact, better for quantification when hydrocarbon internal standards are employed.

The analysis of plasticizers such as di-(2-ethylhexyl adipate) (DEHA) which is used in cling-film together with various phthalates, which can occur as food contaminants originating from printing inks, is readily undertaken using GC-MS after suitable extraction and clean-up of the food sample. The use of stable isotope labeled internal standards has proved to be an effective way to achieve reliable quantification of these plasticizers in foods. Epoxidized soya bean oil (ESBO) is used as a plasticizer in PVC gaskets, at levels up to 30%, to seal metal lids to glass jars.

ESBO can be analyzed in a variety of foods by GC-MS after saponification and derivatization. The packaging for twist-wrapped confectionary is regenerated cellulose film which is softened with various glycols (propylene, mon-, di- and triethylene glycols) and these can be found to migrate into chocolate and sugar confectionary. Confectionary can be dissolved in hot water, sugars precipitated and the glycols analyzed by GC-MS as trimethyl silyl (TMS) derivatives. GC and GC-MS have proved to be the mainstay of studies of migration from plastics into foods.

Although GC-Orbitrap MS is relatively new, it is already showing promise for characterization of hitherto unknown substances in food contact materials which would not otherwise be identifiable by conventional GC-MS. For example, the coating of a metal can, intended for use for food packaging, was solvent extracted for 16 hours and then the extract concentrated prior to injection into a Thermo Scientific™ Q Exactive™ GC Orbitrap™ GC-MS/MS system.

The accurate mass system was operated in EI full-scan mode using 120,000 (FWHM at m/z 200) resolving power. Tentative identification of compounds was based on the derived elemental composition from the scanned spectra, whereby the excellent mass accuracy and isotopic pattern was used to limit the number of possible chemical formulae. An elemental composition calculator was used to propose a formula for the [M+H]+ ion. The software assigns formulae by using an isotopic pattern matching algorithm that accounts for isotope accurate mass and intensity ratios. The algorithm uses a single mass to calculate all possible elemental compositions that lie within a tolerance window and then calculates the theoretical isotopic pattern for each suggestion. It then gives a score between 0 and 100%, where 0 is completely different and 100 an exact isotopic match. Six new compounds were detected in the extract from the can coating, none of which were in the NIST library but for which elemental composition and empirical formula could be proposed.

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HPLC, LC-MS/MS and LC-Orbitrap MS in migration testing

For many monomers and plastics additives such as antioxidants, UV absorbers, and antistatic agents, HPLC with UV or fluorescence detection is the best approach for determining these single entities in food simulants, where high specificity in detection is not required. In many instances nowadays, LC-MS/MS is automatically selected as the preferred analytical tool offering good specificity and the option of simultaneous analysis of several different migrants in a single chromatographic run. LC-MS/MS is the technique most often used for the analysis of perfluorinated compounds, primary aromatic amines and photoinitiators in plastic packaging, and plastic utensils as well as complex extracts from paper and board.

In addition, LC-MS/MS has been used for the analysis of perfluorinated compounds in foods such as corn, popcorn, and chicken eggs. BPA and BADGE have been analyzed in canned food and phthalates in milk-based products, beverages, grain, meat, oil, biscuits, canned foods, and wine. Similarly to GC, recently published methods in LC, have a narrow scope, of usually between 1 and 25 of compounds.

For studies of previously unidentified substances in plastics such as oligomers, trans-formation products or reaction products when developing new polymeric coatings for metal cans, it is necessary initially to confirm identification. LC-Orbitrap MS has proved to be a powerful technique for initial identification of new compounds based on predicted structures, which is subsequently used for target analysis for migration into food simulants or foods. Additionally, the power of using HRMS has been demonstrated in the analysis of phosphoric acid ester flame retardants in aqueous extracts from plastics using stir bar absorption prior to DART-Orbitrap MS and direct analysis of phthalates in soft drink and beverages samples at levels of 1 µg/mL.

LC-Orbitrap MS and DART-Orbitrap MS are yet to be fully exploited in unraveling the complexity of non-intentionally added substances in food contact materials.

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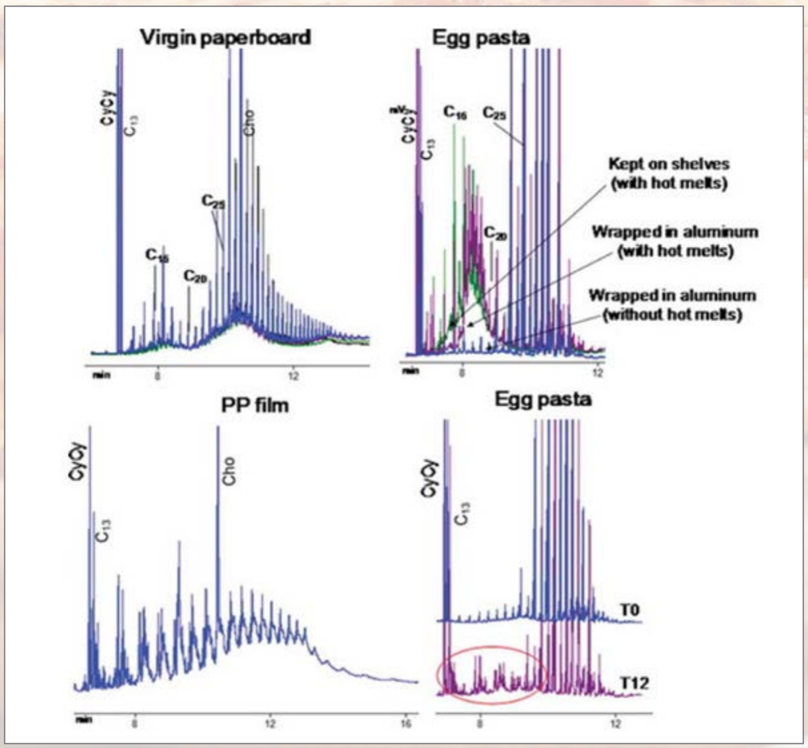
Mineral hydrocarbons by LC-GC-FID

Whilst the relatively simple saturated mineral hydrocarbons used as additives can be analyzed by GC-FID, the mineral oil contaminants found in recycled paper and board are altogether more complex and require alternative technologies. A rather specialized combination of LC-GC-FID has been successfully used separate saturated hydrocarbons (MOSH) from aromatic hydrocarbons (MOAH). The illustration shows how a characteristic pattern of [hydrocarbons is detectable in recycled paperboard](#) and also in pasta packaged in the same material.

Foods, such as the pasta, require extensive treatment and clean-up prior to LC-GC-FID analysis. Hot-melt adhesives also provide a source of mineral oil contamination of foods and in addition to LC-GC it has also been shown that GC-GC can be effective in providing the additional separating power needed to isolate these hydrocarbons for measurement in foods.



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Analysis of trace elements leaching from food contact materials

Many decorative ceramic articles are sold as intended to be used in contact with food and beverages such as ceramic cookware, cups, mugs, bowls, and plates. Barium is predominantly the heavy metal of interest in all modern ceramic wares, whilst cobalt, chromium, tin, and zinc are also found to be present. Cadmium is significantly present in the red colored modern ceramic wares. Leaching studies are generally carried out following the ASTM standard test methods for specific metals leaching into 4% acetic acid solutions after a 24-hour exposure time, or following CEN standards. The acetic acid solution is either analyzed directly by atomic absorption spectroscopy (AAS) for target metals or a multi-element analysis which can be more informative is conducted by ICP-MS.

Ceramic articles intended for high temperature applications are obviously more prone to metal contamination of foods. To gain a more realistic estimate of leaching, these articles can be exposed to real beverages such as hot tea or hot coffee. Other acidic beverages such as orange juice or Coca Cola may give more realistic estimates of metal leaching than 4% acetic acid, but require more sample preparation by acid or microwave digestion prior to i-CAPICP-MS multi-element analysis.

In general the pH of the leaching solutions, its temperature and duration of exposure to the leaching solution have influence in extracting metals from ceramic articles.

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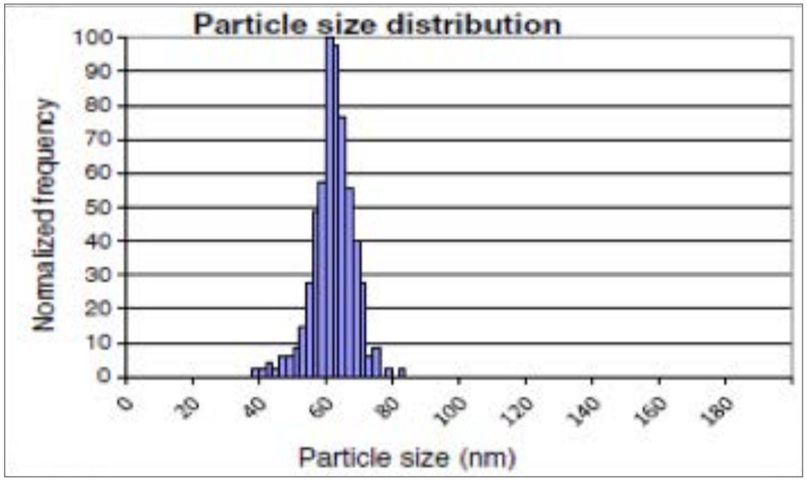
Analysis of nanoparticles migrating from food contact materials

A nanomaterial is defined as an insoluble or bio-persistent and intentionally manufactured material with one or more external dimensions, or an internal structure, on a scale from 1 to 100 nm. The use of nanomaterials in the food packaging market is expected to reach $20 billion by 2020. Currently, a variety of engineered nanomaterials has been introduced into food packaging as functional additives including silver nanoparticles, nanoclay, nano-zinc oxide, nano-titanium dioxide, and titanium nitride nanoparticle. EFSA published a scientific opinion in 2011 entitled "Guidance on the risk assessment of the application of nanoscience and nanotechnologies in the food and feed chain."

Silver nanoparticles have strong antimicrobial activity and are incorporated into active packaging and other food contact articles such as polypropylene plastic storage bags, polystyrene and ABS plastics films, polypropylene food storage containers, baby bottles, and cutting boards. A totally different approach is required to analyze nanoparticles, as it is not the concentration but the number of particles and their size distribution which is important in making a risk assessment. For a sample of chicken meat containing silver nanoparticles, enzymatic digestion of the sample was carried out in two steps and the digest diluted 100,000 times before measuring using single particle-ICP-MS.

The silver ion (m/z 107) was measured with gold as an internal standard. Acquiring data for 60 s at a dwell time of 3 ms resulted in 20,000 data points consisting of background signals and signals with a much higher intensity originating from nanoparticles. These particle signals were isolated from the background by plotting a signal distribution as shown in the figure (i.e. the frequency with which a signal height occurs as a function of that signal height).

This allows a determination of a cut-off point to separate background signals from particle signals. From the number of the particle signals in the time scan and the nebulization efficiency, the particle number concentration in the diluted sample suspension is calculated.



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This white paper reflects on the current state-of-the-art in terms of types of food contact materials in use and problems which have come to light in terms of migration and safety of food. However, food packaging technologies are evolving rapidly with innovations in novel materials as well as novelty in terms of active and intelligent packaging. Whilst the area of food contact materials is highly regulated particularly in the EU, there are often unintended consequences of some of these developments and innovations. It was long assumed that dry foods present a minimal risk of contamination from materials such as paper and board and it was also assumed that many materials provided a functional barrier to say transfer of components from inks on the outside of the packaging. With food analysis becoming ever more sophisticated, both of these assumptions were proven to be wrong and the packaging and food industries to their cost, had to make appropriate changes.

Target analysis of positive list substances is demanding at low levels in foods but is achievable using GC-MS and LC-MS/MS. Non-intentionally added substances (NIAS) present an entirely different challenge but, fortunately, powerful approaches such as LC-Orbitrap MS and GC-Orbitrap MS offer good possibilities for characterization and identification of novel contaminants. Nanoparticles are just beginning to be employed in food contact materials initially in repeat-use articles for storing perishable foods, and whilst single particle ICP-MS has been shown to be a viable approach it remains a specialized technique and work is required for method validation by other laboratories.

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