

## **Chemicals used in plastic materials and articles in contact with food: compliance with statutory limits on composition and migration**

### **Summary**

- This survey was carried out to test for compliance with legislation on the composition of and chemical migration from plastic materials and articles in contact with food. There are controls in British law to protect consumers from unsafe levels in food of the chemical building blocks (monomers) that are used to make plastics for contact with food.
- 200 samples of plastics were tested. Samples of the following were included in this survey: polystyrene, polyvinyl chloride/polyvinylidene chloride, polyethylene terephthalate and nylon-6. These were analysed for the following monomers: 1,3-butadiene, divinylbenzene, ethylvinylbenzene, vinyl chloride, vinylidene chloride, terephthalic acid, 2,6-naphthalene dicarboxylic acid, monoethylene glycol, diethylene glycol, and caprolactam.
- All samples in this survey complied with the law. Although one monomer migrated - caprolactam from nine samples of nylon-6 packaging - the resulting levels in food were less than the legal maximum of 15 milligrams/kilogram (mg/kg). Caprolactam and other monomers were quantified in 38 per cent of packaging samples (75/200), in each case at levels within the legal limits.
- There is, however, a need for further work in applying Comité Européen Nationalisation (CEN) methods, intended for use with food simulants, to test samples of food for monomers. This will be taken forward with CEN.
- There also needs to be research on variation of monomer levels in different samples of food-grade plastic. This will be considered in a forthcoming review of possible projects by the respective, independent working party that advises the Food Standards Agency.

## **Background**

### **The survey**

This survey was carried out to test for compliance with legislation on the composition of and chemical migration from plastic materials and articles in contact with food. British law includes provisions to protect consumers from unsafe levels in food of the chemical building blocks (monomers) used to make food contact plastics. Residues of these monomers can be left in finished plastics because the chemical processes involved are not 100 per cent efficient. The residual monomer may then migrate from plastic into food.

The law in Great Britain defines the maximum residual amounts of many monomers that can be present in plastics and sets maximum levels for chemical migration from plastics into food. Controls on plastics used in contact with food are based on Directives that apply across the European Union (EU). These are negotiated by the Food Standards Agency and government representatives of other Member States of the EU, under the chairmanship of the European Commission. The key specific Directive for plastics is 2002/72/EC.<sup>1</sup> This repealed and at the same time re-enacted Commission Directive 90/128/EEC and its seven amendments in one Directive. Directive 2002/72/EC lists permitted monomers and sets overall and specific migration limits. In addition, Directive 82/711/EEC, as amended, and Directive 85/572/EEC set out the rules for testing compliance with migration limits.<sup>2,3</sup>

In Great Britain, Directive 90/128/EEC and its first five amendments were implemented in the Plastic Materials and Articles in Contact with Food Regulations 1998, which came into force on 1 July 1998.<sup>4</sup> Two later amendments to Directive 90/128/EEC were enacted in amending regulations to the 1998 Regulations. Thus all the provisions of the new single replacement plastics Directive, 2002/72/EC, are enacted in these Regulations. Until the year 2000, regulations were made for Great Britain with separate ones for Northern Ireland. Since then England, Scotland and Wales have made their own regulations to implement EU measures. Similar regulations are made in Northern Ireland. Further information about legislation on plastics and other materials in contact with food is available on the Agency's website at:

<http://www.food.gov.uk/foodindustry/foodcontactmaterials2>

This survey tested whether the following legal standards are being observed in practice:

- The maximum permitted quantity of the substance in the finished material or article (QM) or, the maximum permitted quantity of the substance in the finished material or article, expressed as milligrams (mg) per 6 square decimetres (dm<sup>2</sup>) of the surface in contact with foodstuffs (QMA).
- Specific migration limits (SMLs). SML (T) is the SML in foods or food simulant expressed as total of moiety/substance(s) indicated. These standards are expressed as milligrams/kilogram (mg/kg) of food or of a specified food simulant.

Widely used plastics were tested for residues of the respective monomers, as follows:

- Polystyrene: 1,3-butadiene, divinylbenzene and ethylvinylbenzene.
- Polyvinyl chloride and polyvinylidene chloride: vinyl chloride and vinylidene chloride.
- Polyethylene terephthalate: terephthalic acid, 2,6-naphthalene dicarboxylic acid, monoethylene glycol and diethylene glycol.
- Nylon-6: caprolactam.

The respective legislative limits are:

1,3-Butadiene: QM = 1 mg/kg; SML = not detectable (limit of detection 0.02 mg/kg).

Divinylbenzene: SML = not detectable (limit of detection 0.02 mg/kg).

Ethylvinylbenzene: divinylbenzene may contain up to 40 per cent ethylvinylbenzene.

Vinyl chloride: QM = 1 mg/kg; SML = not detectable (limit of detection 0.01 mg/kg).

Vinylidene chloride: QM = 5 mg/kg; SML = not detectable (limit of detection 0.05 mg/kg).

Terephthalic acid: SML = 7.5 mg/kg.

2,6-Naphthalene dicarboxylic acid: SML = 5 mg/kg.

Monoethylene glycol: SML (T) = 30 mg/kg; with

Diethylene glycol: SML (T) = 30 mg/kg.

Caprolactam (and its sodium salt): SML (T) = 15 mg/kg.

## **Methodology**

### **Samples and timing**

Two hundred samples (Table 1) were purchased mainly in northern England between August 2002 and May 2003. Analytical work was completed in July 2003. The results were collated, checked and reviewed in August and September 2003.

A wide variety of samples was obtained: desserts: 15 samples; meat/fish (including dried, cured and vacuum packed): 33 samples; sandwiches: 5 samples; coffee: 3 samples; dairy products: 4 samples; pizza: 3 samples; cakes: 12 samples; cookies/biscuits: 9 samples; salads/coleslaw: 4 samples; cheeses: 19 samples; carbonated non-alcoholic beverages: 7 samples; carbonated alcoholic beverages: 3 samples; spirit miniatures: 2 samples; still beverages: 10 samples; microwave/oven ready meals (trays and films): 14 samples; cooking oil: 3 samples; salad dressing/mayonnaise: 3 samples; fruit and vegetables: 14 samples; boil-in-the-bag products: 15 samples; other products: 22 samples.

The ratio of purchases between supermarket chains and small outlets was 80:20. Similar numbers of supermarkets' own brand goods and other goods were obtained.

Samples were obtained in triplicate. Each triplicate sample set was assigned a unique sample code. Two of the triplicate samples were overwrapped in aluminium foil and placed immediately in a freezer. The other triplicate sample was prepared for analysis. Any foodstuffs intended to be cooked in the packaging were prepared according to the cooking instructions given on the packs. Then the foodstuff was removed from the packaging, overwrapped in aluminium foil and stored in a freezer (less than  $-20^{\circ}\text{C}$ ). The packaging material was similarly overwrapped in aluminium foil and also stored in a freezer. Foodstuffs not intended to be cooked in the packaging were removed from the packaging on receipt at the laboratory and both the food and the packaging were overwrapped in aluminium foil and stored in a freezer.

## **Analytical methods**

Fourier transform-infrared (FT-IR) spectroscopy was carried out on the packaging of all samples to confirm the plastic type.

### ***1,3-Butadiene***

#### *Overview*

1,3-Butadiene in packaging and foods was determined by headspace gas chromatography coupled with mass spectrometry (HS-GC-MS). The methods used were based on Comité Européen Nationalisation (CEN) method EN13130 Part 4 for plastics and Part 15 for foods.<sup>5</sup> Pentane was used as an internal standard. Calibration standards were prepared in *N,N*-dimethylacetamide (DMA) for packaging samples and water for foodstuffs.

### *Standard solutions*

For packaging analyses, standard solutions of 1,3-butadiene and pentane were prepared in DMA at 10 milligrams per litre (mg/L). Stock solutions were diluted with DMA to form calibration standards (0 to 0.4 mg/L 1,3-butadiene) each containing 0.2 mg/L pentane as internal standard. Fresh solutions were prepared for each analytical batch.

For food analyses standard solutions of 1,3-butadiene and pentane were prepared in DMA at 1 mg/L. Stock solutions were diluted with water to form calibration standards (0 to 0.04 mg/L 1,3-butadiene) each containing 0.02 mg/L pentane as internal standard. Fresh solutions were prepared for each analytical batch.

### *Packaging material: sample preparation*

A specimen of the packaging material (0.5 g) was cut into small pieces (approximately 0.5 cm<sup>2</sup>) and dissolved in DMA (5 ml) in a headspace vial. Internal standard was added at a level equivalent to 2 parts per million (ppm) in the packaging. Duplicate specimens from each sample were prepared. Samples were incubated for 30 minutes at 90°C prior to analysis to release any volatile compounds from the packaging into the headspace.

### *Foods: sample preparation*

Solid foodstuffs were homogenised using a food blender and the homogenised food was slurried with an equal mass of water. A specimen of the slurry (5 g) was transferred to a headspace vial. Liquid foods were stirred to ensure thorough mixing and a specimen (5 g) transferred to a headspace vial. Internal standard was added to the vials at a level equivalent to 0.02 ppm in the foodstuff. Duplicate specimens were prepared. Samples were incubated for 30 minutes at 90°C prior to analysis to release any volatile compounds from the foodstuff into the headspace.

### *Analysis*

All samples were analysed by HS-GC-MS using a ThermoQuest Voyager operated in selected ion monitoring (SIM) mode with electron impact ionisation, under the following conditions:

Column:	Chrompack CP-PoraPLOT Q (styrene-divinylbenzene), 30 m x 0.32 mm id x 10 micrometre film thickness
Carrier gas:	Helium at 1 ml/minute
Autosampler:	Fisons Instruments HS 800
Injection volume:	1 ml, splitless

Splitless time: 60 seconds  
Syringe temperature: 100°C  
Injection temperature: 250°C  
Interface temperature: 280°C  
Oven programme: 40°C hold for 2 minutes  
Raise to 220°C at 13°C per minute  
Hold for 8 minutes  
Ions monitored: m/z 39 and 54 for 1,3-butadiene  
m/z 43, 57 and 72 for the internal standard, pentane

#### *Monomer identification and quantification*

Calibration curves were constructed for the solvent standards by plotting the peak area ratio of m/z 39 for 1,3-butadiene versus m/z 43, 57, 72 for pentane against the level of 1,3-butadiene. For each survey sample duplicate 'spikes' were prepared by fortifying with 1,3-butadiene at a level of 1 ppm for the packaging samples and 0.02 ppm for the food samples. The levels in these 'spiked' samples were determined from the appropriate calibration graph. The results for these 'spiked' samples were used to determine the analytical recovery.

#### *Confirmation criteria*

- The ion ratio for m/z 39/54 should be within  $\pm 25$  per cent of that obtained from the calibration standards.
- The retention time for the 1,3-butadiene peak relative to the internal standard (RRT) should agree to within  $\pm 5$  per cent of that obtained from the calibration standards.
- The full scan mass spectrum of the sample when compared to the calibration standards should give no additional ions (in excess of 20 per cent) in the sample spectrum which are not present in the standard spectrum.

## ***Divinylbenzene and ethylvinylbenzene***

### *Overview*

Divinylbenzene and ethylvinylbenzene in foods were determined by HS-GC-MS.  $d_{14}$ -Diethylbenzene was used as an internal standard. The packaging material was not analysed for divinylbenzene and ethylvinylbenzene due to changes in the legislation made during this survey (the QM was replaced with a QMA in 2002/72/EC).

### *Standard solutions*

For food analyses stock standard solutions of divinylbenzene:ethylvinylbenzene (a commercial mixture, 55:45 m/m) and  $d_{14}$ -diethylbenzene were prepared in acetonitrile at 1 mg/L. Stock solutions were diluted with water to form calibration standards (0 to 0.08 mg/L divinylbenzene:ethylvinylbenzene) each containing 0.02 mg/L  $d_{14}$ -diethylbenzene as internal standard. Fresh solutions were prepared for each analytical batch.

### *Foods: sample preparation*

The foodstuff was homogenised using a food blender. A specimen of the homogenised foodstuff (5 g) was transferred to a headspace vial. Liquid foods were stirred to ensure thorough mixing and a specimen (5 g) transferred to a headspace vial. Internal standard was added at a level equivalent to 0.02 ppm in the food. Duplicate specimens of each sample were prepared. Samples were incubated for 60 minutes at 70°C prior to analysis to release any volatile compounds from the foodstuff into the headspace.

### *Analysis*

All samples were analysed by HS-GC-MS using a ThermoQuest Voyager operated in SIM mode with electron impact ionisation, under the following conditions:

Column:	Chrompack CPSil 5CB (100 per cent dimethylpolysiloxane) 50 m x 0.32 mm id x 1.2 micrometre film thickness
Carrier gas:	Helium at 1 ml/minute
Autosampler:	Fisons Instruments HS800
Injection volume:	1 ml, splitless
Splitless time:	60 seconds
Syringe temperature:	80°C
Injection temperature:	280°C
Interface temperature:	280°C

Oven programme: 100°C hold for 2 minutes  
Raise to 280°C at 10°C per minute  
Hold for 5 minutes

Ions monitored: m/z 128 and 130 for divinylbenzene  
m/z 117 and 132 for ethylvinylbenzene  
m/z 130 for the internal standard, d<sub>14</sub>-diethylbenzene

### *Monomer identification and quantification*

Calibration curves were constructed for the solvent standards by plotting:

- the peak area ratio m/z 130 for divinylbenzene versus m/z 130 for d<sub>14</sub>-diethylbenzene against the level of divinylbenzene in the calibration solutions; and
- m/z 132 for ethylvinylbenzene versus m/z 130 for d<sub>14</sub>-diethylbenzene against the level of ethylvinylbenzene in the calibration solutions.

For each survey sample duplicate 'spikes' were prepared by fortifying with divinylbenzene:ethylvinylbenzene (55:45) at a level of 0.02 ppm. The 'spiked' samples were quantified from the calibration graphs and the results used to determine the analytical recovery.

### *Confirmation criteria*

- The ion ratios for m/z 128/130 for divinylbenzene and 117/132 for ethylvinylbenzene should be within  $\pm 25$  per cent of those obtained from the respective calibration standards.
- The retention time for the monomer peak relative to the internal standard (RRT) should agree to within  $\pm 5$  per cent of that obtained from the calibration standards.
- The full scan mass spectrum of the sample when compared to the calibration standards should give no additional ions (in excess of 20 per cent) in the sample spectrum which are not present in the standard spectrum.

## ***Vinyl chloride and vinylidene chloride***

### *Overview*

Vinyl chloride and vinylidene chloride in packaging and foods were determined by HS-GC-MS. The method of analysis for vinyl chloride in packaging and foods were based on those in Directives 80/766/EEC and 81/432/EEC respectively.<sup>6,7</sup> Analysis for vinylidene chloride was based on EN13130 Part 6 for plastics and Part 5 for foods.<sup>5</sup> 1,3-Butadiene was used as an internal standard for the determination of vinyl chloride and 1-chloropropane for vinylidene chloride. Calibration standards were prepared in DMA for packaging samples and in water for the foodstuffs.

### *Standard solutions*

For packaging analyses stock solutions of vinyl chloride, 1,3-butadiene, vinylidene chloride and 1-chloropropane were prepared in DMA at 10, 10, 50 and 50 mg/L respectively. Stock solutions were diluted with DMA to form calibration standards (0 to 0.4 mg/L vinyl chloride and 0 to 2 mg/L vinylidene chloride) each containing 0.2 mg/L 1,3-butadiene and 1 mg/L 1-chloropropane as internal standards. Fresh solutions were prepared for each analytical batch.

For food analyses stock solutions of vinyl chloride, 1,3-butadiene, vinylidene chloride and 1-chloropropane were prepared in DMA at 1, 1, 5 and 5 mg/L respectively. Stock solutions were diluted with water to form calibration standards (0 to 0.02 mg/L vinyl chloride and 0 to 0.1 mg/L vinylidene chloride) each containing 0.02 mg/L 1,3-butadiene and 0.1 mg/L 1-chloropropane as internal standards. Fresh solutions were prepared for each analytical batch.

### *Packaging materials: sample preparation*

A specimen of the packaging material (0.5 g) was cut into small pieces (approximately 0.5 cm<sup>2</sup>) and dissolved in DMA (5 ml) in a headspace vial. 1,3-Butadiene was added at a level equivalent to 2 ppm in the packaging and 1-chloropropane was added at a level equivalent to 10 ppm in the packaging. Duplicate specimens were prepared. Samples were incubated for 30 minutes at 90°C to release any volatile compounds from the packaging to the headspace.

### *Foods: sample preparation*

Solid foodstuffs were homogenised using a food blender and the homogenised food was slurried with an equal mass of water. A specimen of the slurry (5 g) was then weighed into a headspace vial. Liquid foods were stirred to ensure thorough mixing and an aliquot (5 g)

weighed into a headspace vial. Internal standards were added at levels equivalent to 0.04 ppm for the 1,3-butadiene and 0.2 ppm for the 1-chloropropane in the foodstuff. Duplicate specimens were prepared. Samples were incubated for 30 minutes at 90°C to release any volatile compounds from the foodstuff into the headspace.

### *Analysis*

All samples were analysed by HS-GC-MS using a ThermoQuest Voyager operated in SIM mode with electron impact ionisation, under the following conditions:

Column:	Chrompack CP-PoraPLOT Q (styrene-divinylbenzene), 30 m x 0.32 mm id x 10 micrometre film thickness
Carrier gas:	Helium at 1 ml/minute
Autosampler:	Fisons Instruments HS 800
Injection volume:	1 ml, splitless
Splitless time:	60 seconds
Syringe temperature:	100°C
Injection temperature:	250°C
Interface temperature:	280°C
Oven programme:	40°C hold for 2 minutes Raise to 220°C at 13°C per minute Hold for 8 minutes
Ions monitored:	m/z 62 and 64 for vinyl chloride m/z 61 and 96 for vinylidene chloride m/z 39 and 54 for 1,3-butadiene m/z 43, 52 and 78 for 1-chloropropane

### *Monomer identification and quantification*

Calibration curves were constructed for the solvent standards by plotting:

- the peak area ratio of m/z 62 for vinyl chloride versus m/z 39 and 54 for 1,3-butadiene against the level of vinyl chloride;
- the peak area ratio of m/z 61 for vinylidene chloride versus m/z 43, 52 and 78 for 1-chloropropane against the level of vinylidene chloride in packaging; and

- the peak area ratio of m/z 96 for vinylidene chloride versus m/z 43, 52 and 78 for 1-chloropropane against the level of vinylidene chloride in foods.

For each survey sample duplicate 'spikes' were prepared by fortifying with vinyl chloride and vinylidene chloride at 1 ppm and 5 ppm respectively for the packaging samples and 0.01 ppm and 0.05 ppm respectively for the food samples. The levels in these 'spiked' samples were determined from the appropriate calibration graph. The results were used to determine analytical recovery.

#### *Confirmation criteria*

- The ion ratios for m/z 62/64 for vinyl chloride and 61/96 for vinylidene chloride should be within  $\pm 25$  per cent of those obtained from the calibration standards.
- The retention time for each monomer peak relative to the internal standard (RRT) should agree to within  $\pm 5$  per cent of that obtained from the calibration standards.
- The full scan mass spectrum of the sample when compared to the calibration standards should give no additional ions (in excess of 20 per cent) in the sample spectrum which are not present in the standard spectrum.

### ***Terephthalic acid and 2,6-naphthalene dicarboxylic acid***

#### *Overview*

Terephthalic acid and 2,6-naphthalene dicarboxylic acid in packaging were determined by solvent extraction followed by analysis by high performance liquid chromatography with UV detection (HPLC-UV). This method was based on one used by Pira International in a study on the migration of monomers.<sup>8</sup> Orthophthalic acid was used as an internal standard. Terephthalic acid and 2,6-naphthalene dicarboxylic acid in foods were determined by solvent extraction followed by derivatisation to form the dimethyl esters using boron trifluoride diethyl etherate as a catalyst and further extraction with heptane prior to analysis by GC-MS. d<sub>4</sub>-Terephthalic acid was used as an internal standard. This method was based on that of Castle *et al.*<sup>9</sup>

#### *Standard solutions*

For packaging analyses standard solutions of terephthalic acid, 2,6-naphthalene dicarboxylic acid and orthophthalic acid were prepared in pH 7 buffer at 1, 1 and 0.5 g/L respectively. Stock solutions were diluted with pH 7 buffer to form calibration standards (0 to 80 mg/L terephthalic acid and 0 to 100 mg/L 2,6-naphthalene dicarboxylic acid) each

containing 20 mg/L orthophthalic acid as internal standard. Solutions were stored at +4°C and fresh solutions were prepared every week.

Buffer (pH 7) was prepared by diluting 1M aqueous sodium hydroxide solution (100 ml) to 350 ml with water, adjusting to pH 7 with glacial acetic acid and making up to 500 ml with water.

For food analyses standard solutions of terephthalic acid, 2,6-naphthalene dicarboxylic acid and d<sub>4</sub>-terephthalic acid were prepared in methanol at 0.75, 0.5 and 0.75 g/L respectively. Stock solutions were diluted with methanol to form calibration standards (0 to 15 mg/L terephthalic acid and 0 to 10 mg/L 2,6-naphthalene dicarboxylic acid) each containing 3.75 mg/L d<sub>4</sub>-terephthalic acid as internal standard. Solutions were stored at +4°C. Fresh solutions were prepared every week.

#### *Packaging materials: sample preparation*

A specimen of the packaging material (1 g) was cut into small pieces (approximately 0.2 cm<sup>2</sup>) and weighed into glass vials. The polymer was extracted for 4 hours at 60°C with occasional shaking using 1,1,1-trichloroethane:methanol (4:1 v/v). The solvent was removed under a stream of nitrogen and the residue was redissolved in pH 3.6 buffer (1 ml) containing internal standard (0.02 mg/ml). The resulting solution was filtered and analysed by HPLC-UV. The pH 3.6 buffer contained sodium acetate trihydrate (25 g in 350 ml water) and orthophosphoric acid (5 ml) adjusted to pH 3.6 with glacial acetic acid, and made up to volume (500 ml) with water.

#### *Foods: sample preparation*

Solid foodstuffs were homogenised. Concentrated hydrochloric acid (0.1 ml) and d<sub>4</sub>-terephthalic acid (equivalent to 7.5 ppm in the food) were added to 10 g specimens of the homogenised food. The foodstuff was extracted with methanol (20 ml) and the extract was dried over anhydrous sodium sulphate. The methanol extract was then extracted with heptane to remove any fat (which if present may interfere with the derivatisation procedure). The resultant extract was reduced to a volume of approximately 4 ml under a stream of nitrogen. Liquid foodstuffs were stirred to ensure thorough mixing and d<sub>4</sub>-terephthalic acid (equivalent to 7.5 ppm in the food) was added to 10 g specimens. The liquid food was evaporated to dryness under a stream of nitrogen. The residue was dissolved in methanol (20 ml) and concentrated hydrochloric acid (0.1 ml) was added. The methanol was dried over anhydrous sodium sulphate and was reduced in volume to approximately 4 ml under a stream of nitrogen. All extracts were derivatised using boron trifluoride diethyl etherate as a catalyst. The derivatised extracts were then shaken with 2M

aqueous sodium chloride to quench the reaction and further extracted with heptane. The heptane extracts were analysed by GC-MS. Duplicate specimens were prepared.

#### *Analysis*

All packaging extracts were analysed by HPLC-UV for terephthalic acid using an Agilent HP1100 system with a diode array UV detector under the following conditions:

Column: Phenomenex Luna C18 (ODS2) 3 micrometre particle size, 150 x 3 mm  
Injection volume: 20 microlitres  
Flow rate: 0.5 ml/minute  
Mobile phase: pH 3.6 buffer:methanol:water (15:5:80)  
Detection: UV, 242 nm

All packaging extracts were analysed by HPLC-UV for 2,6-naphthalene dicarboxylic acid using the following conditions:

Column: Phenomenex Prodigy C18 (ODS3) 5 micrometre particle size, 250 x 3.2 mm  
Injection volume: 20 microlitres  
Flow rate: 0.75 ml/minute  
Mobile phase: 0.01M tetrabutylammonium hydroxide (pH 6.9):methanol (60:40)  
Detection: UV, 242 nm

All derivatised food samples were analysed by GC-MS for both terephthalic acid, dimethyl ester, and 2,6-naphthalene dicarboxylic acid, dimethyl ester, using a ThermoQuest Voyager system operated in SIM mode with electron impact ionisation under the following conditions:

Column: J & W DB5 (5 per cent phenyl-methylpolysiloxane) 30 m x 0.25 mm id x 0.25 micrometre film thickness  
Carrier gas: Helium at 1 ml/minute  
Autosampler: Carlo Erba A200S  
Injection volume: 1 microlitre, split 10:1  
Injection temperature: 280°C  
Interface temperature: 280°C

Oven programme: 120°C hold for 1 minute  
Raise to 280°C at 20°C per minute  
Hold for 2 minutes

Ions monitored: m/z 126, 213 and 244 for 2,6-naphthalene dicarboxylic acid, dimethyl ester  
m/z 163 and 194 for terephthalic acid, dimethyl ester  
m/z 167 and 198 for d<sub>4</sub>-terephthalic acid, dimethyl ester

#### *Monomer identification and quantification*

##### *Packaging materials*

Calibration curves were constructed for the solvent standards by plotting the peak area ratio for the monomers versus the internal standard, against the mass of monomer added. The mass in the samples was determined from the calibration curves produced. For each survey sample duplicate 'spikes' were prepared by fortifying with terephthalic acid and 2,6-naphthalene dicarboxylic acid at levels equivalent to 20 ppm for both monomers. The masses of these substances in the 'spiked' samples were determined from the calibration graphs and the results obtained were used to determine the analytical recovery. The confirmation criteria were as follows:

- Any sample tentatively identified as possibly containing terephthalic acid and 2,6-naphthalene dicarboxylic acid was re-analysed using diode array (200 to 400 nm) to confirm the presence of the monomer.
- The UV spectra obtained were compared with those for the solvent standards.
- Samples were confirmed if the lambda max was consistent with that of the standards.

##### *Foods*

Calibration curves were constructed for the derivatised solvent standards by plotting:

- the peak area ratio m/z 194 for terephthalic acid, dimethyl ester, versus m/z 167 and 198 for the d<sub>4</sub>-terephthalic acid, dimethyl ester, against the mass of terephthalic acid in the calibration solutions; and
- the peak area ratio m/z 244 for 2,6-naphthalene dicarboxylic acid, dimethyl ester, versus m/z 167 and 198 for the d<sub>4</sub>-terephthalic acid, dimethyl ester, against the mass of 2,6-naphthalene dicarboxylic acid in the calibration solutions.

The mass of the monomers in the samples was determined from the calibration curves produced. For each survey sample duplicate 'spikes' were prepared by fortifying with a known mass of both terephthalic acid and 2,6-naphthalene dicarboxylic acid at levels equivalent to the SMLs (7.5 and 5 ppm respectively). The mass of the monomers in the 'spiked' samples was determined from the calibration graph and the analytical recovery was determined from this. The confirmation criteria were as follows:

- The ion ratios for m/z 163/194 for terephthalic acid, dimethyl ester, and 213/244 for 2,6-naphthalene dicarboxylic acid, dimethyl ester, should be within  $\pm 25$  per cent of those obtained from the calibration standards. In the presence of any interference the ion ratio of m/z 244/126 will also be used for 2,6-naphthalene dicarboxylic acid, dimethyl ester.
- The retention time for each monomer peak relative to the internal standard (RRT) should agree to within  $\pm 5$  per cent of that obtained from the calibration standards.
- The full scan mass spectrum of the sample when compared to the calibration standards should give no additional ions (in excess of 20 per cent) in the sample spectrum which are not present in the standard spectrum.

### ***Monoethylene glycol (MEG) and diethylene glycol (DEG)***

#### *Overview*

MEG and DEG in packaging and foods were determined by solvent extraction followed by analysis by gas chromatography coupled with flame ionisation detection (GC-FID). The determination of MEG and DEG in foods was based on CEN method EN13130 Part 7.<sup>5</sup> Butan-1,4-diol was used as an internal standard. Solvent-based calibration standards were prepared for packaging samples and solid foods. Standard addition was used to determine the level in liquid foods due to matrix enhancement/suppression effects identified during the sample check exercise.

#### *Standard solutions*

For packaging analyses standard solutions of MEG, DEG and butan-1,4-diol were prepared in dichloromethane at 5 g/L. Stock solutions were diluted with dichloromethane to form calibration standards (0 to 100 mg/L MEG and DEG) each containing 50 mg/L butan-1,4-diol as internal standard. Solutions were stored at +4°C and fresh solutions were prepared every week.

For food analyses standard solutions of MEG, DEG and butan-1,4-diol were prepared in methanol at 10, 10 and 3 g/L respectively. Stock solutions were diluted with methanol to form calibration standards (0 to 100 mg/L MEG and DEG) each containing 30 mg/L butan-1,4-diol as internal standard. Solutions were stored at +4°C and fresh solutions were prepared every week.

#### *Packaging materials: sample preparation*

A specimen of the packaging material (0.5 g) was cut into small pieces (approximately 0.2 cm<sup>2</sup>) and weighed into a glass vial. Internal standard solution (equivalent to 50 ppm in the packaging) and dichloromethane (5 ml) were added. The sealed vial was put on an orbital shaker for 24 hours at room temperature. The extract was removed and analysed by GC-FID. A second, repeat extraction was carried out on the plastic to ensure complete extraction of any monomer. Duplicate specimens were prepared from each sample.

#### *Foods: sample preparation*

Solid foodstuffs were homogenised using a food blender and a portion of homogenised food (10 g) weighed into a glass vial. Internal standard (butan-1,4-diol, equivalent to 30 ppm in the food) was added to the foodstuff which was extracted for 2 hours in methanol (5 ml) and hexane (10 ml) using an orbital shaker. The mixture was centrifuged and the methanol layer removed. Methanol (5 ml) was added to the residue, which was re-extracted for a further 2 hours. The methanol extracts were combined, filtered and analysed by GC-FID. Duplicate specimens were prepared. Liquid foods were stirred to ensure thorough mixing before a portion (10 g) was taken for extraction. Internal standard (butan-1,4-diol equivalent to 30 ppm in the food) was added to the sample (10 g) which was diluted with an equal mass of methanol prior to analysis by GC-FID. Duplicate specimens were prepared.

#### *Analysis*

All samples were analysed by GC-FID using a Carlo-Erba HRGC 5300 Mega series instrument under the following conditions:

Column:	J & W DBFFAP (nitroterephthalic acid modified polyethylene glycol) 30 m x 0.32 mm id x 1.2 micrometre film thickness
Carrier gas:	Helium 70 kPa
Autosampler:	Carlo Erba A200S
Injection volume:	1 microlitre, split 10:1

Injection temperature: 220°C  
FID temperature: 220°C  
Oven programme: 50°C hold for 1 minute  
Raise to 150°C at 10°C per minute  
Hold for 8 minutes

Where the levels of MEG and DEG identified by GC-FID were above the limit of quantification, the determination was confirmed by GC-MS using a ThermoQuest Voyager or a Hewlett Packard GC-MSD 5971 series operated in SIM mode. The method used was as for the GC-FID (above) but with the following additional parameters:

Interface temperature: 280°C  
Ions monitored: m/z 43 and 62 for monoethylene glycol  
m/z 45 and 75 for diethylene glycol  
m/z 71 for butan-1,4-diol

#### *Monomer identification and quantification*

Calibration curves were constructed for the solvent standards by plotting the peak area ratio for the monomers versus the internal standard, against the mass of monomer added. The mass of analyte in the samples was determined from calibration curves produced. For each survey sample duplicate 'spikes' were prepared by fortifying with MEG and DEG at a level of 500 ppm for the packaging and 30 ppm for the food samples. The masses of these substances in the 'spiked' samples were determined from the calibration graphs and the results obtained were used to determine the analytical recovery. The level of the analyte in liquid foods was determined from standard addition curves due to matrix enhancement/suppression effects identified during the sample check exercise.

#### *Confirmation criteria*

Where suspect positives were identified by GC-FID, confirmation was undertaken using GC-MS.

- The ion ratios for m/z 62/43 for MEG and 75/45 for DEG should be within  $\pm 25$  per cent of those obtained from the calibration standards.

- The retention time for the monomer peak relative to the internal standard (RRT) should agree to within  $\pm 5$  per cent of that obtained from the calibration standards.
- The full scan mass spectrum of the samples when compared to the calibration standards should give no additional ions (in excess of 20 per cent) in the sample spectrum which are not present in the standard spectrum.

## **Caprolactam**

### *Overview*

Caprolactam in packaging was determined by solvent extraction followed by GC-FID. Caprolactam in foods was determined by solvent extraction followed by liquid chromatography coupled with mass spectrometry (LC-MS) loosely based on the CEN method EN13130 Part 16.<sup>5</sup> For both packaging and food analysis capryllactam was used as an internal standard and solvent-based calibration standards were used.

### *Standard solutions*

For packaging analyses standard solutions of caprolactam and capryllactam were prepared in 95 per cent aqueous ethanol at 2 and 4 g/L respectively. Stock solutions were diluted with 95 per cent ethanol to form calibration standards (0 to 160 mg/L caprolactam) each containing 160 mg/L capryllactam as internal standard. Solutions were stored at +4°C and fresh solutions were prepared every week.

For food analyses standard solutions of caprolactam and capryllactam were prepared in ethanol:water (1:2 v/v) at 10 and 3 g/L respectively. Stock solutions were diluted with ethanol:water (1:2 v/v) to form calibration standards (0 to 100 mg/L caprolactam) each containing 30 mg/L capryllactam as internal standard. Solutions were stored at +4°C and fresh solutions were prepared every week.

### *Packaging materials: sample preparation*

A specimen of the packaging material (0.5 g) was cut into small pieces (approximately 0.2 cm<sup>2</sup>) and weighed into a glass vial. Internal standard solution (capryllactam equivalent to 4,000 ppm) was added along with the extraction solvent (25 ml of 95 per cent aqueous ethanol). Extraction was carried out for 24 hours at 60°C with occasional shaking. The solvent extract was removed and analysed by GC-FID. A repeat extraction of the plastic was carried out to ensure complete extraction of the monomer. Duplicate specimens were prepared from each sample. All samples were analysed by GC-FID.

### *Foods: sample preparation*

Foodstuffs were homogenised using a food blender and a specimen of the homogenised food (10 g) was weighed into a glass vial. Internal standard (capryllactam, equivalent to 30 ppm in the food) was added to the foodstuff which was extracted on an orbital shaker for 16 hours in ethanol:water (10 ml, 1:2 v/v) and heptane (10 ml). The mixture was centrifuged and the ethanol:water layer removed, diluted (x 5) and filtered prior to analysis by LC-MS. Duplicate specimens were prepared.

### *Analysis*

All nylon-6 packaging samples were analysed by GC-FID using a Carlo Erba HRGC Mega series instrument under the following conditions:

Column: Chrompack CPSil 8CB (5 per cent phenyl 95 per cent dimethylpolysiloxane) 30 m x 0.32 mm x 1.2 micrometre film thickness

Carrier gas: Helium at 70 kPa

Autosampler: Carlo Erba A200S

Injection volume: 1 microlitre, split 10:1

Injection temperature: 200°C

FID temperature: 240°C

Oven programme: 130°C hold for 3 minutes  
Raise to 240°C at 10°C per minute  
Hold for 5 minutes

All food samples were analysed by LC-MS using a Micromass VG Platform instrument in SIM mode under the following conditions:

Column: Jones Chromatography, Genesis C18 120, 4 micrometer particle size, 250 x 3 mm

Injection volume: 20 microlitre

Flow rate: 0.5 ml/minute

Mobile phase A: water

Mobile phase B: acetonitrile

Gradient: at time = 0, 5, 20, 25, 26, 30, 31 and 35 minutes,

percentage mobile phase B = 10, 15, 45, 45, 90, 90, 10 and 10

Ionisation mode: +ve APcl

Source temperature: 150°C

Probe temperature: 450°C

Ions monitored m/z 69, 79 and 114 for caprolactam  
m/z 142 for capryllactam

### *Monomer Identification and quantification*

#### *Packaging materials*

Calibration curves were constructed for the solvent standards by plotting the peak area ratio for the caprolactam versus the internal standard, against the mass of caprolactam added. For each survey sample duplicate 'spikes' were prepared by fortifying with the monomer at a level of 4,000 ppm. The mass of caprolactam in the 'spiked' samples was determined from the calibration graphs and the results obtained were used to determine the analytical recovery.

#### *Foods*

Calibration curves were constructed for the solvent standards by plotting the peak area ratio m/z 114 for caprolactam versus m/z 142 for capryllactam against the mass of caprolactam in the calibration solutions. For each survey sample duplicate 'spikes' were prepared by fortifying with a known mass of caprolactam at a level equivalent to the SML (T) (15 ppm). The mass of caprolactam in the 'spiked' samples was determined from the calibration graph and the analytical recovery was determined from this.

#### *Confirmation criteria*

- The ion ratios for m/z 114/69, 114/79 and 79/69 for caprolactam should be within  $\pm 25$  per cent of those obtained from the calibration standards.
- The retention time for each monomer peak relative to the internal standard (RRT) should agree to within  $\pm 5$  per cent of that obtained from the calibration standards.

### **Quality assurance**

#### *'Spiked' samples*

The suitability of the analytical methods to determine monomers in packaging (where QMs are given) and in foods was tested by analysing representative materials and foods

'spiked' with appropriate levels of monomers. Known volumes of standard solutions were allowed to infuse into the materials or foods prior to extraction. Measurement accuracy on spiked samples ranged from 80-114 per cent as demonstrated in the check sample exercises. Samples were prepared as replicate specimens (n=6-8) to obtain repeatability data.<sup>10</sup> Repeatability ranged from 3.6-11 per cent depending on the monomer and the packaging/food type under investigation.

#### *Internal quality control*

A typical batch size was 5-10 analytical samples, each prepared in duplicate. Each analytical batch also included at least one method blank and duplicate control samples. Duplicate control samples were prepared for each survey sample since recovery varied depending on the monomer and matrix being analysed. Control samples were prepared by fortifying the packaging or homogenised foodstuff with a known mass of monomer to give a level equivalent to the QM or SML for that substance. Mean recovery for each sample was calculated from the analysis of the control samples compared to the analytical standard solutions. Sample extracts were interspersed in the sample sequence of each batch with bracketing standards.

#### *Check sample exercise – test for accuracy*

To demonstrate the accuracy of the analyses, six food samples were 'spiked' with each monomer so that they could be analysed 'blind' as a check of method accuracy. Where appropriate both solid and liquid foodstuffs were 'spiked'. Similarly six packaging samples were spiked with those monomers for which a QM restriction applies. Samples were prepared at the Department of Chemistry, University of Leeds. The samples were then supplied to the testing laboratory for extraction and analysis. In all cases the testing laboratory correctly identified the blanks and the 'spiked' samples. There was good agreement between added and determined levels of monomers (Tables 2-7). Polystyrene (PS) and polyvinyl chloride/polyvinylidene chloride (PVC/PVdC) packaging materials containing no detectable 1,3-butadiene, vinyl chloride and vinylidene chloride were selected as blank materials for the packaging analysis. Food types and spiking levels used for each monomer were:

- For 1,3-butadiene in PS packaging, portions of packaging (0.5 g) were 'spiked' with a solution of 1,3-butadiene (11.5 microgram/ml 1,3-butadiene in *N,N*-dimethylacetamide (DMA)).
- For 1,3-butadiene (from PS) in food, portions of yoghurt (2.5 g) were 'spiked' with a solution of 1,3-butadiene (1.15 microgram/ml 1,3-butadiene in DMA).

- For divinylbenzene/ethylvinylbenzene (from PS) in food, portions of yoghurt (5 g) were 'spiked' with a solution of divinylbenzene/ethylvinylbenzene (55:45, 1 microgram/ml in acetonitrile).
- For vinyl chloride and vinylidene chloride in packaging, portions of packaging (0.5 g) were 'spiked' with a solution of 10.1 microgram/ml vinyl chloride and 50.1 microgram/ml vinylidene chloride in DMA.
- For vinyl chloride and vinylidene chloride in food, portions of biscuit (2.5 g) were 'spiked' with a solution of 1.01 microgram/ml vinyl chloride and 5.01 microgram/ml vinylidene chloride in DMA.
- For monoethylene glycol (MEG) and diethylene glycol (DEG) (from polyethylene terephthalate (PET)) in solid food, portions of cottage pie (100 g) were 'spiked' with a solution containing 2.9 mg/ml MEG and 3.0 mg/ml DEG in dichloromethane.
- For monoethylene glycol and diethylene glycol (from PET) in liquid food, portions of cola (100 g) were 'spiked' with a solution containing 3.0 mg/ml MEG and 3.0 mg/ml DEG in dichloromethane.
- For terephthalic acid (from PET) in solid and liquid foods, portions of cottage pie (100 g) and cola (100 g) were 'spiked' with a solution containing 0.80 mg/ml terephthalic acid in methanol.
- For 2,6-naphthalene dicarboxylic acid (from PET) in solid and liquid foods, portions of cottage pie (100 g) and cola (100 g) were 'spiked' with a solution containing 0.49 mg/ml 2,6-naphthalene dicarboxylic acid in methanol.
- For caprolactam (from nylon-6) in food, portions of chicken (100 g) were 'spiked' with a solution containing 1.5 mg/ml of caprolactam in ethanol:water (1:2 v/v).

### *Reporting*

Brand names were reported as this survey was carried out in accordance with previous guidelines. The absence of a particular brand from Table 1 means only that the brand was not included in the survey. All sample results were corrected for recovery.

### **Results, interpretation and action**

Comité Européen Nationalisation (CEN) methods were used in this survey, with adaptations. It was found that these methods can result in false positives when applied to foods rather than simulants (for which they were developed). It is important to use robust confirmation methods to check such suspect positives and so ensure certainty in the

results. This was done in this survey. There is a need for further work in applying CEN methods, intended for use with food simulants, to test samples of food for monomers. This will be taken forward with CEN.

Chemical migration from packaging into food was found in 4.5 per cent of samples (9/200): caprolactam was found in nine samples of different foods at 2.8 to 13 mg/kg. A greater proportion of packaging samples contained quantifiable amounts of monomers (38 per cent; 75/200).

All samples in this survey complied with the law. The results for different types of packaging were as follows:

- Polystyrene: 1,3-butadiene was found in three samples. One of the samples (PS36) was tested as a whole retail pack (10 filter cups) and as individual cups (3-6 cups). The mean level of 1,3-butadiene in the whole retail pack was 0.55 mg/kg, compared to a QM of 1 mg/kg. There was some variation in the levels of 1,3-butadiene between different cups (Table 8). There was no measurable migration of 1,3-butadiene into food with an analytical method that was more sensitive than that required in law, which also requires that this substance should not be detectable in food.
- Polyvinyl chloride: vinyl chloride was found in two samples, in both cases at less than the QM (Table 9). It was not detected in food using an analytical method that was more sensitive than is required in law.
- Polyvinylidene chloride: no monomer found.
- Polyethylene terephthalate: diethylene glycol was not found; one or more of the other monomers was found in 40 samples; terephthalic acid found at 0.84 to 29 mg/kg in 33 samples (no QM); 2,6-naphthalene dicarboxylic acid at 0.62 to 5.2 mg/kg in seven samples (no QM); monoethylene glycol at 26 to 41 mg/kg in 11 samples (no QM). There was no measurable migration of any of these substances. The limits of quantification were less than the respective SMLs (SMLs: terephthalic acid: 7.5 mg/kg; 2,6-naphthalene dicarboxylic acid: 5 mg/kg; monoethylene glycol: 30 mg/kg; with diethylene glycol: 30 mg/kg).
- Nylon-6: caprolactam was found in 30 samples at 380 to 2,600 mg/kg; there was migration into food in nine samples but the SML was not exceeded (Table 10).

The results for 1,3-butadiene indicate that research is needed on variation of monomer levels in different samples of food-grade plastic. This will be considered in a forthcoming

review of possible projects by the respective, independent working party that advises the Food Standards Agency.

## **References**

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This is the final report of this survey. Copies of it have been placed on the Agency's website and in the library in Aviation House, 125 Kingsway, London WC2B 6NH.

**Table 1: Details of samples obtained****Polystyrene packaging**

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PS01	Blackcurrant flavour cup drink with sweeteners	Calypso	185 ml	None given	None given	50818679	Jul 03	01.09.02	Spar
PS02	Munch Bunch Pot shots	Eden Vale	250 g	None given	None given	50138340	15 Sep	01.09.02	Spar
PS03	Strawberries and cream creamy yogurt dessert	Waitrose	150 g	UK	None given	5000169896327	14 Aug	04.08.02	Waitrose
PS04	Goats yogurt with a burst of berries	Antony Worrall Thompson	125 g	UK	None given	50513802	20 Aug	04.08.02	Somerfield
PS05	Smart pots light smooth chocolate dessert	Cadbury	135 g	EU	308125-1	5029728260010	11.09.02	04.08.02	Somerfield
PS06	Olives	Waitrose	250 g	None given	None given	5000169567029	Consume within 2 days	04.08.02	Waitrose
PS07	Strawberry luxury cheesecake	Cottage catering	100 g	None given	None given	5020590212978	15 Aug	11.08.02	OM. Superstore
PS08	Cottage cheese	Lidl Stiftung & Co.	200 g	Austria	F60	20002183	16.08.2002	11.08.02	Lidl
PS09	Cottage cheese natural	Cool Country	170 g	None given	None given	50406302	09 Sep	15.08.02	Hunger Hill Stores
PS10	5 Rice crisp cakes chocolate flavour	Cotswold cakes Ltd.	137.5 g	None given	2207	5011688009013	31 Jan 2003	10.08.02	Woodside Stores
PS11	4 Muffins fruit and vanilla	Master Baker	4 x 59.4 g	EU	None given	5410543623034	22/10/2002	14.08.02	Poundline
PS12	Low fat pasteurised strawberry yogurt	Cool Country	113 g	None given	None given	50797622	14/9/2002	29.08.02	Westend Convenience Store
PS13	Dates	Czar's of London Ltd.	250 g	None given	None given	5033641000103	August 2002	11.08.02	Whistlestop
PS14	Seedless white grapes	Thompson	140 g	Greece	1059DEFRAA11092*0209	None given	None given	14.09.02	Red Apple Super Stop
PS15	2 Cheese & onion slices	Heritage	165 g each	None given	AC3R	5010893790631	17 Sep	14.09.02	Midland News
PS16	Greek style orange layered yogurt	Ubley	140 g	UK	None given	50006434	17 May	16.05.03	CSL canteen
PS17	2 Fresh pork chops	Iceland	300 g	Belgium	1149	5010482006044	08 Oct	03.10.02	Iceland
PS18	Fromage frais vanilla flavour dessert	Brooklea	200 g	UK	None given	22116567	17 Oct	03.10.02	Aldi

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PS19	Raspberry yogurt	Marks and Spencer Count on us	200 g	UK	None given	M0305983S	6-Oct-02	03.10.02	Marks and Spencer
PS20	Thin & crispy cheese & tomato pizza	Marks and Spencer	290 g	UK	None given	M0219440S	08 Oct	03.10.02	Marks and Spencer
PS21	Sherbourne Cumberland sausages	Netto	454 g	UK	004032	5021490982244	9 Oct	03.10.02	Netto
PS22	Lemon and lime yogurt mousse	Boots Shapers	90 g	UK	None given	01756975	04 Oct	03.10.02	Boots
PS23	British pork belly slices	Spar	0.264 kg	UK	None given	0206054000847	08.10.02	05.10.02	Spar
PS24	Braising steak	Morrisons	0.305 kg	UK	None given	0200168000595	08 Oct	05.10.02	Morrisons
PS25	Freshly prepared mackerel	Morrisons	400 g	None given	None given	0267313001524	07 Oct	05.10.02	Morrisons
PS26	Low fat jaffa orange yogurt with real fruit	Morrisons	150 g	UK	C5K	5010251045991	18 Oct	05.10.02	Morrisons
PS27	2 Sparerib chops	Tesco	0.450 kg	UK	1544	0232849001805	13 Oct	10.10.02	Tesco
PS28	Baby desserts made with follow on milk	Danone	12 x 50 g	France	None given	5410146403330	21/10	11.10.02	Tesco
PS29	Best braising steak	Asda	0.150 kg	UK	1213851	0227540000882	13-10-02	11.10.02	Asda
PS30	Individual cream for coffee	Kerrygold	100 ml	Ireland	None given	5000165072862	9-1-2003	11.10.02	Asda
PS31	Organic double cream	Sainsbury's	284 ml	UK	None given	01301007	13 Oct	11.10.02	Sainsbury's
PS32	10 Individual filter coffees decaffeinated blend	Sainsbury's	75 g	Belgium	2239	00407816	Aug 03	11.10.02	Sainsbury's
PS33	Turkey breast split fillets	Safeway	300 g	UK	151018	06554439	22-Oct-02	18.10.02	Safeway
PS34	Pork loin chops	Safeway	400 g	UK	7352-21	229877001853	19-Oct-02	18.10.02	Safeway
PS35	Pizzaroma stone baked margherita	Safeway	365 g	UK	None given	06535728	22-Oct-02	18.10.02	Safeway
PS36a	10 Individual filter coffees original	Rombouts	57 g	Belgium	2161	5010206510017	Jun-03	18.10.02	Safeway
PS36b	10 Individual filter coffees original	Rombouts	57 g	Belgium	3066B	5010206510017	Mar-04	07.07.03	Safeway
PS37	Andy Pandy fromage frais	Yoplait Dairy Crest Ltd.	50 g	UK	None given	5024581100435	11-Nov-02	18.10.02	Co-op
PS38	Double fresh pasteurised cream	Co-op	142 ml	UK	10:08YK100	5000128790413	5-Nov-02	29.10.02	Co-op
PS39	Fresh British beef rump steak	Co-op	274 g	UK	9012	0294191002039	02 Nov	29.10.02	Co-op
PS40	French one cup coffee filters	Tesco	10 x 7.5 g	Produce of more than one country	2274	5018374158147	Oct 03	29.10.02	Tesco
PS41	Pizzaroma stone backed spinach and ricotta	Safeway	400 g	UK	None given	06447014	31 Oct	29.10.02	Safeway

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PS42	Strawberry yogurt	Yeo valley	150 g	UK	None given	5036589200079	31 Oct	30.10.02	Boots
PS43	Cottage cheese with salmon and dill	Tesco	200 g	UK	None given	03031544	06 Nov	30.10.02	Tesco
PS44	Fresh chicken breast fillet	Somerfield	140 g	UK	102 021024	000192191154	31-Oct-02	29.10.02	Somerfield
PS45	Individual margarine portions	St Ivel Vitalite	10 g	None given	None given	None given	None given	30.10.02	Tesco
PS46	Italian gorgonzola	Safeway	180 g	Italy	None given	06478063	20.11.02	29.10.02	Safeway
PS47	Fresh green pears	Sainsbury's	150 g	Italy	W0432A	00491464	03 Nov	31.10.02	Sainsbury's
PS48	Vanilla dessert	Campina	500 g	Germany	345	40406374	16.Dec.02	31.10.02	Sainsbury's
PS49	Hot and spicy bowl noodle soup	Nong Shim	86 g	Korea	None given	031146250103	End 08.03	31.10.02	Sainsbury's
PS50	Red Leicester cheese slices	Asda	160 g	None given	260	W27196175G	26.11.02	11.10.02	Asda

### Polyvinyl chloride/polyvinylidene chloride packaging

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PVC01	Bourbon biscuits	Tesco value	500 g	UK	None given	5031021075864	12 Apr	03.10.02	Tesco
PVC02	Organic fennel	Waitrose	190 g	Holland	258/TT 7603	None given	09 Aug	04.08.02	Waitrose
PVC03	Baby corn	Somerfield	115 g	Thailand	15628	29012602	Display until 05 Aug	04.08.02	Somerfield
PVC04	6 Scotch pancakes	Somerfield	180 g	UK	X72	29028641	05 Aug	04.08.02	Somerfield
PVC05	4 Savoury eggs	Somerfield	213 g	UK	052092	29002320	10 Aug	04.08.02	Somerfield
PVC06	Classic Rich Tea	M <sup>c</sup> Vitie's	200 g	UK	HBD2	5000168001203	28 Dec 02	04.08.02	Rottingdean Wines
PVC07	Caramel shortcake	Bestway	65 g	None given	None given	5027876 054642	End Sep 02	10.08.02	Hothi Food and Wine
PVC08	Caramel shortcake	Boots	70 g	None given	26-75-048	02675046	1-Nov-02	11.10.02	Boots
PVC09	Cheese & onion quiche portion	Spar	150 g	None given	None given	5010358 051291	5 Sep 02	29.08.02	Spar
PVC10	Golden crunch creams	Fox's	200 g	England	12 S	5010035002738	23 11 02	15.08.02	Hunger Hill Stores
PVC11	Carrot cake	Cookie Coach Company	80 g	England	E3	5027952003267	11 Oct 02	29.08.02	Costcutter
PVC12	Seedless white grapes	Thompson	140 g	Greece	1059DEFRAA11092*0 209	None given	None given	14.09.02	Red Apple Super Stop

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PVC13	Chargrilled chicken and peppers sandwich	Mattesons	150 g	None given	None given	5000178101245	End 16 Sep	14.09.02	One Stop Convenience Stores
PVC14	4 Traditional Eccles cakes	Bobby's foods	45 g each	None given	N327	5015282118942	24 Sep 02	14.09.02	Roundabout Service Station
PVC15	Rich sultana cake slices	Rosette Quality Products	360 g	None given	None given	5038322100073	End Nov 02	16.09.02	The Food Weighhouse
PVC16	Double Gloucester with soft cheese and chives	Marks and Spencer	150 g	UK	None given	M0134323S	26 Oct	03.10.02	Marks and Spencer
PVC17	Wafer thin cured chicken	Marks and Spencer	90 g	Denmark	None given	M0272353S	09 Oct	03.10.02	Marks and Spencer
PVC18	Organic tomatoes	Marks and Spencer	200 g	None given	None given	00371483	07 Oct	03.10.02	Marks and Spencer
PVC19	Instant hot chocolate drink	Galaxy (Mars)	400 g	None given	U2392	5023471107110	Mar 2004	05.10.02	Spar
PVC20	Thin sliced beef	Morrisons	125 g	Produced in UK from South American Beef	None given	2310688200752	12 Oct	05.10.02	Morrisons
PVC21	Egg mayonnaise sandwich	Morrisons	190 g	None given	None given	0248873000998	6 Oct	05.10.02	Morrisons
PVC22	Chicken and stuffing lunchbox	Morrisons	280 g	UK	None given	0204257001395	07 Oct	05.10.02	Morrisons
PVC23	Bakewell tarts	Cakes for the Connoisseur	200 g	England	3B	5013387910980	28 Oct 02	05.10.02	Spar
PVC24	Large salad bowl	Sainsbury's	40 g	None given	None given	0268642000202	Consume within 24 hours	11.10.02	Sainsbury's
PVC25	Dry cured oak smoked ham	Sainsbury's Taste the difference	160 g	UK	P2	01270242	21 Oct	11.10.02	Sainbury's
PVC26	Blue stilton cheese	Iceland	225 g	UK	None given	5010482219086	22 Oct 02	11.10.02	Iceland
PVC27	Cheese spring onion baguette	Asda	202 g	UK	None given	234500000998	12-10-02	11.10.02	Asda
PVC28	Chocolate eclairs	Asda	150 g	UK	None given	G20417222M	12 Oct	11.10.02	Asda

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PVC29	Quorn deli turkey flavour with stuffing	Marlow Foods Ltd.	100 g	None given	None given	5019503004465	19 Oct	11.10.02	Tesco
PVC30	Milk chocolate digestives	M <sup>c</sup> Vitie's	400 g	UK	HCE6	5000168010311	29 Mar 03	11.10.02	Asda
PVC31	Crumbed Wiltshire cured ham	Tesco finest	140 g	UK	None given	0227459002182	29 Oct 02	11.10.02	Tesco
PVC32	Appetisers	S <sup>1</sup> Moret	100 g	France	2.253.2	3175333021002	09-11-02	11.10.02	Asda
PVC33	French garlic & parsley roule	Co-op	96 g	None given	None given	0295489001154	21 Oct	18.10.02	Co-op
PVC34	Shortbread fingers	Deans	150 g	Scotland	2074	5015473000025	15 Mar 03	18.10.02	Asda
PVC35	American style fresh dips	Safeway	4 x 100 g	None given	None given	06185015	21 Oct	18.10.02	Safeway
PVC36	Forest fruits real creams	Safeway	200 g	None given	2252A	06478919	09 Mar 03	18.10.02	Safeway
PVC37	Fresh club salad	Safeway	215 g	Produce of various countries	None given	06514907	31 Oct	29.10.02	Safeway
PVC38	3 Chocolate fudge slices	Sainsbury's	150 g	UK	C1373	01367706	31/10/02	29.10.02	Sainsbury's
PVC39	Party rings	Fox's	125 g	England	C2	5010035002486	28-12-02	29.10.02	Iceland
PVC40	Dairy cream raspberry turnover	Sainsbury's	180 g	None given	131708	00002790	30 Oct	29.10.02	Sainsbury's
PVC41	Freshly prepared salad bar	Sainsbury's	180 g	None given	None given	0270795000756	Eat within one day of purchase	29.10.02	Sainsbury's
PVC42	Mini Cornish pasties	Safeway	2 x 30 g	UK	None given	06564179	30 Oct	29.10.02	Safeway
PVC43	Strawberries	Tesco	227 g	England	None given	10010983	28 Oct	28.10.02	Tesco
PVC44	8 Meringue nests	Tesco	15 g per nest	Scotland	053477/G2 HC 2285	5000119162472	11-Apr-03	29.10.02	Tesco
PVC45	After dinner biscuit assortment	Boots	200 g	Various	None given	02544793	1-Mar-03	29.10.02	Boots
PVC46	Medium flan case	Tesco	75 g	UK	2249A	5000119166388	6 Dec	29.10.02	Tesco
PVC47	Chocolate chunk cookies	Cadbury	200 g	None given	2281A 19:32	072417083136	05 07 2003	31.10.02	Safeway
PVC48	Deep filled chicken and bacon sandwich	Asda go large	230 g	UK	None given	W00837408G	12 Oct	11.10.02	Asda
PVC49	Mozzarella tomato & basil, Italian style chicken, smoked ham, mozzarella and rocket sandwiches	Boots	236 g	None given	None given	02971520	11 Oct	11.10.02	Boots
PVC50	Fruit medley	Boots Shapers	140 g	None given	None given	03566633	5 Oct	03.10.02	Boots

### Polyethylene terephthalate packaging

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PET01	Cider	Strongbow	3 l	None given	L30378	5000104090193	15 Oct	14.02.03	Asda
PET02	Powerade ice storm drink	Coca-cola	500 ml	EU	L2338H112 6	54492653	02 06 2003	14.02.03	Asda
PET03	Indian tonic water	Asda	330 ml	UK	23580818K 4A	W27280867G	Jun 2003	14.02.03	Asda
PET04	Cottage pie	Asda	300 g	UK	None given	W20376000G	18 Feb	14.02.03	Asda
PET05	Chicken curry and rice	Asda	300 g	UK	None given	W20437039G	19 Feb	14.02.03	Asda
PET06	Caesar dressing	Asda	150 ml	UK	None given	G23041493M	02 Mar	14.02.03	Asda
PET07	Strong dry cider	Sainsbury's	2 l	UK	L3021B	01067279	Sep 03	18.02.03	Sainsbury's
PET08	Lemon and lime flavoured spring water sparkling drink	Sainsbury's	330 ml	UK	L3624	01320572	Jul 03	18.02.03	Sainsbury's
PET09	Macaroni cheese	Sainsbury's	300 g	UK	None given	00393768	24 Feb	18.02.03	Sainsbury's
PET10	Italian penne nicoise	Sainsbury's	300 g	UK	S1361	01271515	01 Mar	18.02.03	Sainsbury's
PET11	Ebly original tender wheat	Ebly	220 g	None given	<0029>3	5010034950009	16/12/03	20.02.03	Sainsbury's
PET12	Rice pudding	Sainsbury's	230 g	UK	KDJ	1329575	26 Feb	20.02.03	Sainsbury's
PET13	Fruit shoot blackcurrant and apple drink	Robinson's	300 ml	None given	30021940	5010102105379	Oct 03	22.02.03	One Stop Convenience Stores
PET14	Coca-cola	Coca-cola	500 ml	None given	None given	54491472	6/7/03	22.02.02	Treats
PET15	Irn-Bru	A.G.Barr p.l.c	2 l	None given	None given	5000382012290	Jan 04	24.02.03	Safeway
PET16	Sprite lemon-lime sparkling drink	Coca-Cola	500 ml	None given	023EK714: 41	54491069	Jun 03	24.02.03	Spar
PET17	Bac-O's	Betty Crocker	116 g	USA	A31406	0016000740006	20 Mar 03	24.02.03	Tesco
PET18	Lime and coriander dressing	Safeway	175 ml	None given	1543/3	06501259	10 Mar	24.02.03	Safeway
PET19	Instant hot chocolate	Cadbury High Lights	220 g	None given	3013	5000183064047	07 2004	24.02.03	Morrisons
PET20	Chicken korma with pilau rice	Spar	400 g	None given	None given	5010358057408	02 Mar	24.02.03	Spar
PET21	Indian plain naan breads	Sharwood's	260 g	UK	033B	5000197573450	05/05/03	26.02.03	Safeway
PET22	Traditional coleslaw	Marks and Spencer	225 g	UK	None given	M0951531S	02 Mar	27.02.03	Marks and Spencer
PET23	Lasagne	Authentic Cuisine	1 kg	Netherlands	None given	5021490982084	16/03/03	27.02.03	Netto
PET24	Sunflower oil	Happy Shopper	1 l	None given	3056 HA	5011295061176	Feb 04	20.04.03	The Lea Village Store

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PET25	Original American style cola	Marks and Spencer	500 ml	UK	DU03/04/03 0	M0172912S	17/04/03	27.02.03	Marks and Spencer
PET26	Cider	Tesco value	2 l	UK	L3042B01: 27	5000436794905	13/10/03	28.02.02	Tesco
PET27	Ginger beer	W.T.Foods	500 ml	None given	3038	5029578001085	Nov 2003	28.02.02	Tesco
PET28	Sunflower oil	Aldi	1 l	Ireland	L3029A4	25008685	31/01/2004	28.02.03	Aldi
PET29	Ice valley natural mineral water	Shepley Spring Ltd.	500 ml	UK	2316 12:39C	5033022002009	Nov 03	28.02.03	Iceland
PET30	Sunsqueeze shots	Aldi	250 ml	None given	L3019 20:59	25033588	1/8/03	28.02.03	Aldi
PET31	Beef cannelloni	Tesco value	340 g	UK	None given	5000436925385	3/3/03	28.02.03	Tesco
PET32	Chinese takeaway chicken fried rice	Iceland	340 g	Ireland	R30141 16:29	5010482137564	14 Jan 04	28.02.03	Iceland
PET33	Chicago Town Diner deep dish pepper steak	Schwan's Consumer Brands	365 g	EU	L02329	5019312682038	25/8/03	28.02.03	Iceland
PET34	Fresh mash	Tesco	400 g	UK	None given	5000436896777	03 Mar	28.02.03	Tesco
PET35	Indigo liquid energy refreshment drink	Vimto Soft Drinks	375 ml	None given	13 08:58 T4	5010438014109	Jan 2004	05.03.03	Co-op
PET36	Dessert pears	Co-op	8 pears	Portugal	M060A	5000128634175	09 Mar	05.03.03	Co-op
PET37	Lancashire hot pot	Co-op	450 g	Made using lamb from various countries and EU potatoes	None given	5000128640398	15 Mar	05.03.03	Co-op
PET38	Spaghetti bolognese	Co-op	340 g	Made in UK using British beef and Italian spaghetti	None given	5000128628945	16 Mar	05.03.03	Co-op
PET39	Raspberries	Tesco	125 g	Spain	None given	10020722	10 Mar	05.03.03	Tesco
PET40	Apple juice	Copella Fruit Juices	250 ml	UK	BPC04:00	50974078	21/03/03	05.03.03	Boots
PET41	Mayonnaise	Hellman's	340 ml	UK	3030X	5000184321903	Nov 03	10.03.03	Somerfield
PET42	Still water	Highland Spring Ltd	500 ml	UK	22641346	5010459005216	Apr 2004	06.12.02	J & S Newsagents
PET43	Cherry tomatoes	Somerfield	250 g	Spain	085951/1	5000192100255	14 Mar	10.03.03	Somerfield

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
PET44	Seedless white grapes	Somerfield	350 g	Chile	016253	0208930001818	13/3/03	10.03.03	Somerfield
PET45	Whisky	Bell's	5 cl	Scotland	L28P03099 836	50387229	None given	14.09.02	Stars News Shops
PET46	Vegetable cooking oil	Best buy	1 l	None given	3058 HB	5019996053254	Feb 04	20.04.03	Rose & Castle
PET47	Lime and kiwi flavoured still drink	Slayker	500 ml	None given	2290 10:4	5034398300041	17/07/03	12.05.03	CSL canteen, York
PET48	Original Irish cream	Baileys	50 ml	Ireland	L1456198	5011013100231	Best taste before 01/2003	29.03.03	Boozebusters
PET49	Jacket potato with baked beans	Pro-Cuisine	340 g	None given	30712 11:10	5032824000497	21 Apr	20.04.03	General Garage, Huntley
PET50	Iced Tea, peach	Lipton	500 ml	None given	LT219 11:18	5000118059421	08.2003	20.04.03	Coleridge Store

### Nylon-6 packaging

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
NO1	Spaghetti bolognese	Birds Eye Wall's	340 g	UK	L23342041 22	5000116041329	End 05 2004	14.02.03	Asda
NO2	Roast beef in Gravy	Birds Eye Wall's	227 g	UK	L30310541 22	5000116020843	End 07 2004	14.02.03	Asda
NO3	Liver chubb pate	Asda smartprice	113 g	Belgium	24556	W20367978G	23/3/03	20.02.03	Asda
NO4	Smoked back bacon rashers	Sainsbury's	300 g	UK, Denmark or Holland	L0738898	01374315	20 Mar	20.02.03	Sainsbury's
NO5	Garlic smoked pork sausage	Mattesson's	227 g	Holland	2003D	5000178002832	05 04	14.02.03	Asda
NO6	Mature cheddar	Asda Smartprice	390 g	UK	None given	0260571001572	01 May	14.02.03	Asda
NO7	Chicken breast in gravy	Sainsbury's	200 g	UK	2354 11:48	01216462	End Dec 2003	18.02.03	Sainsbury's
NO8	Prawn curry	Sainsbury's	400 g	UK	2346 22:06	01210170	End Dec 2003	18.02.03	Sainsbury's

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
NO9	Cheesestrings	The Golden Vale Cheese Co.	21 g	Ireland	None given	53999498	02.05.03	22.02.03	One Stop Convenience Stores
NO10	Polony slicing sausage	Tesco value	150 g	UK	None given	5000436 618959	22 May	01.05.03	Tesco extra
NO11	Medium English cheddar	Safeway	250 g	UK	BP068219	06201982	09 Apr	24.02.03	Safeway
NO12	Half fat red cheese	Morrisons	234 g	None given	None given	0257070001103	12 Apr	24.02.03	Morrisons
NO13	Traditional black pudding	Galtee	300 g	Ireland	None given	5011037922932	09 May 03	24.02.03	Morrisons
NO14	Ready made polenta	Italfresco	500 g	Italy	None given	5060005420031	11 06 03	26.02.03	Safeway
NO15	Beef stew & dumplings	Birds Eye Wall's	320 g	UK	L23382241 22	5000116051595	06 2004	24.02.03	Spar
NO16	Sliced beef in gravy	Safeway	227 g	None given	3009 17:52	05026289	Jan 04	24.02.03	Safeway
NO17	Tortelloni four cheeses	San Michele	250 g	Italy	None given	8001532101502	09/04/2003	26.02.03	Sainsbury's
NO18	Scottish oak smoked salmon ribbons	Marks and Spencer	100 g	Scotland	None given	M0209038S	09 Mar	26.02.03	Marks and Spencer
NO19	Mild cheddar	Marks and Spencer	35 g	None given	None given	00138659	Apr 19	26.02.03	Marks and Spencer
NO20	Cod & parsley sauce	Marks and Spencer	184 g	Fish from Iceland or Norway. Made in UK	A07A	M0269919S	Jan 04	26.02.03	Marks and Spencer
NO21	Fish Choice fish steaks in butter sauce	Ross	600 g	UK	WB 3014T 43	5000149002441	End Jul 04	26.02.03	Netto
NO22	Beef curry with rice	Birds Eye Walls	375 g	UK	L22922331 22	5000116050994	End 04 2004	28.02.03	Tesco
NO23	Chicken stew and dumplings	Birds Eye Walls	320 g	UK	L23481541 22	5000116051533	End 06 2004	28.02.03	Tesco
NO24	Chicken supreme with rice	Birds Eye Walls	375 g	UK	L30280331 22	5000116050963	End 07 2004	28.02.03	Tesco
NO25	Chicken chow mein	Iceland	400 g	Ireland	R23501 09:39	5010482070427	16 Dec 03	28.02.03	Iceland
NO26	Double Gloucester	Iceland	185 g	UK	268ID2799 5A	5010482279950	28 Apr	28.02.03	Iceland
NO27	Kabanos	Tesco	150 g	Germany	None given	03030165	19 Mar	28.02.03	Tesco

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
NO28	2 Smoked gammon rounds	Iceland	255 g	Origin of pork Denmark Cured and packed in UK	487ID2364 1C	5010482236410	10 Mar	28.02.03	Iceland
NO29	Unsmoked rindless back bacon	Tesco value	300 g	British, Danish or Dutch pork	None given	5050179376609	01 Apr	28.02.03	Tesco
NO30	Bavarian smoked processed cheese	Co-op	150 g	Germany	2PH621	5000128653404	02 Aug 03	05.03.03	Co-op
NO31	Bake in the bag seasoning	The British Pepper and Spice Co. Ltd	35 g	None given	41262	5000385010453	End May 2003	05.03.03	Tesco
NO32	Seafood sticks	Macfisheries	250 g	None given	K2/260	5014547002460	22 Mar	05.03.03	Co-op
NO33	Cooked beetroot	Sainsbury's Organic	200 g	UK	3042OE	01133196	11 Jun 03	10.03.03	Sainsbury's
NO34	Tagliatelle carbonara	Co-op	340 g	UK	2290	5000128459938	End Apr 04	05.03.03	Co-op
NO35	Dutch gouda cheese	Sainsbury's	168 g	Netherlands	None given	0211102000915	24 Mar 03	10.03.03	Sainsbury's
NO36	Roast chicken in gravy	Birds Eye Wall's	156 g	UK	L21902341 22	5000116037803	End 01 2004	10.03.03	Somerfield
NO37	2 Smoked gammon steaks	Tesco	300 g	UK	None given	5010204498171	31 May	01.05.03	Tesco extra
NO38	Full flavoured cheddar cheese	Somerfield	276 g	UK	None given	0283298001243	04 May	10.03.03	Somerfield
NO39	2 Haddock steaks	Morrisons	2 x 170 g	None given	11023	5010251118589	End Aug 2004	10.03.03	Morrisons
NO40	6 smoked bacon rashers	Wall's	165 g	None given	2A	5000187103179	8 Oct	14.09.02	Stars News Shops
NO41	Unsmoked rindless back bacon	Happy Shopper	200 g	Holland	None given	5011295 062449	Use by 21 May	21.04.03	Colridge Store
NO42	German smoked sausage	Sainsbury's	250 g	Germany	None given	00558327	21.05.03	01.05.03	Sainsbury's
NO43	Unsmoked bacon	Key Country Foods	200 g	Holland	None given	502236240200	16.05.03	19.04.03	Rite Stop
NO44	Cheddar	Happy Shopper	250 g	None given	None given	5011295068731	09 Jul	19.04.03	Rose & Castle
NO45	Frankfurters	Herta	350 g	France	None given	3154230045984	05 May	29.04.03	GT Smith
NO46	Kipper fillets	Co-op	200 g	Caught in North East Atlantic	None given	5 000128 599177	08 May	29.04.03	GT Smith

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
NO47	Half spring chicken	House of Westphalia	300 g	None given	509	4006895040202	15.10.2003	20.04.03	General Garage, Huntley
NO48	Turkey viennas	Yarden	250g	Israel	None given	7290000 367767	29.06.03	01.05.03	Sainsbury's
NO49	Traditional white pudding	Galtee	300 g	Ireland	None given	5 011037922925	9 Jul 03	01.05.03	Sainsbury's
NO50	Cod steak in parsley sauce	Sainsbury's	150 g	UK	3062 N	01346930	Mar 2004	01.05.03	Sainsbury's

**Table 2: Results from check sample exercise for the analysis of 1,3-butadiene in polystyrene and yoghurt**

Results are from single determinations of each sample and are corrected for analytical recovery

Sample	Spike level (micrograms/kilogram)	Level reported (micrograms/kilogram)	Measurement Accuracy (per cent)
<b>Polystyrene</b>			
1	Blank	ND*	NA
2	460	490	107
3	Blank	ND*	NA
4	1800	1800	100
5	460	490	107
6	1800	1600	89
<b>Yoghurt</b>			
1	9.2	10	109
2	Blank	ND <sup>#</sup>	NA
3	32	33	103
4	Blank	ND <sup>#</sup>	NA
5	9.2	9.1	99
6	32	32	100

\* not detected; limit of detection = 35 micrograms/kilogram

<sup>#</sup> not detected; limit of detection = 0.69 micrograms/kilogram

NA: not applicable

**Table 3: Results from the check sample exercise for the analysis of divinylbenzene/ethylvinylbenzene (from PS) in yoghurt**

Results are from single determinations of each sample and are corrected for analytical recovery

<b>Sample</b>	<b>Spike level (micrograms/kilogram)</b>	<b>Level reported (micrograms/kilogram)</b>	<b>Measurement Accuracy (per cent)</b>
<b>Yoghurt</b>			
1	Blank	ND*	NA
2	16	16	100
3	Blank	ND*	NA
4	16	14	88
5	30	24	80
6	30	24	80

\* not detected; limits of detection = 1.2 and 1.0 micrograms/kilogram for divinylbenzene/ethylvinylbenzene respectively

NA: not applicable

**Table 4: Results from check sample exercise for the analysis of vinyl chloride (VC) and vinylidene chloride (VdC) in polyvinyl chloride (PVC) and polyvinylidene chloride (PVdC) packaging, and biscuit**

Results are from single determinations of each sample using matrix-matched standards and are corrected for analytical recovery

Sample	Spike level (micrograms/kilogram)	Level reported (micrograms/kilogram)	Measurement Accuracy (per cent)
<b>PVC (VC)</b>			
1	810	830	102
2	Blank	ND*	NA
3	1,600	1,800	113
4	Blank	ND*	NA
5	1,600	1,500	94
6	810	760	94
<b>PVdC (VdC)</b>			
1	4,000	4,200	105
2	Blank	ND <sup>#</sup>	NA
3	8,000	7,700	96
4	Blank	ND <sup>#</sup>	NA
5	8,000	7,800	98
6	4,000	4,000	100
<b>Biscuit (VC)</b>			
1	8.1	8.9	110
2	Blank	ND <sup>a</sup>	NA
3	8.1	7.8	96
4	28	25	89
5	28	24	86
6	Blank	ND <sup>a</sup>	NA
<b>Biscuit (PVdC)</b>			
1	40	46	114
2	Blank	ND <sup>b</sup>	NA
3	40	44	110
4	140	119	85
5	140	120	86
6	Blank	ND <sup>b</sup>	NA

\* not detected; limit of detection = 30 micrograms/kilogram

<sup>#</sup> not detected; limit of detection = 150 micrograms/kilogram

NA: not applicable

<sup>a</sup> not detected; limit of detection = 0.30 micrograms/kilogram

<sup>b</sup> not detected; limit of detection = 1.5 micrograms/kilogram

**Table 5: Results from the check sample exercise for the analysis of monoethylene glycol and diethylene glycol (from PET) in cottage pie and cola**

Results are from single determinations of each sample and are corrected for analytical recovery

Sample	Monoethylene glycol (MEG)			Diethylene glycol (DEG)		
	Spike level (milligrams /kilogram)	Level reported (milligrams /kilogram)	Measurement Accuracy (per cent)	Spike level (milligrams /kilogram)	Level reported (milligrams /kilogram)	Measurement Accuracy (per cent)
<b>Cottage Pie</b>						
1	Blank	ND*	NA	Blank	ND <sup>#</sup>	NA
2	20	19	95	21	23	110
3	20	18	90	21	21	100
4	Blank	ND*	NA	Blank	ND <sup>#</sup>	NA
5	58	52	90	60	60	100
6	58	57	98	60	62	103
<b>Cola</b>						
1	Blank	ND <sup>a</sup>	NA	Blank	ND <sup>b</sup>	NA
2	18	16	89	18	18	100
3	Blank	ND <sup>a</sup>	NA	Blank	ND <sup>b</sup>	NA
4	100	100	100	110	100	96
5	100	96	96	110	110	100
6	18	18	100	18	20	111

\* not detected; limit of detection = 4.4 milligrams/kilogram

<sup>#</sup> not detected; limit of detection = 3.2 milligrams/kilogram

NA: not applicable

<sup>a</sup> not detected; limit of detection = 1.7 milligrams/kilogram

<sup>b</sup> not detected; limit of detection = 4.0 milligrams/kilogram

**Table 6: Results from the check sample exercise for the analysis of terephthalic acid and 2,6-naphthalene dicarboxylic acid (from PET) in cottage pie and cola**

Results are from single determinations of each sample and are corrected for analytical recovery

Sample	Terephthalic acid			2,6-Naphthalene dicarboxylic acid		
	Spike level (milligrams /kilogram)	Level reported (milligrams /kilogram)	Measurement Accuracy (per cent)	Spike level (milligrams /kilogram)	Level reported (milligrams /kilogram)	Measurement Accuracy (per cent)
<b>Cottage Pie</b>						
1	6.4	6.2	97	4.0	3.8	95
2	Blank	ND*	NA	Blank	ND <sup>#</sup>	NA
3	Blank	ND*	NA	Blank	ND <sup>#</sup>	NA
4	6.4	6.4	100	4.0	4.1	103
5	16	16	100	9.9	10	101
6	16	17	106	9.9	9.6	97
<b>Cola</b>						
1	Blank	ND <sup>a</sup>	NA	Blank	ND <sup>b</sup>	NA
2	4.8	4.8	100	3.5	3.9	111
3	4.8	4.7	98	Blank	ND <sup>b</sup>	NA
4	12	11	92	3.5	3.6	103
5	Blank	ND <sup>a</sup>	NA	7.4	7.4	100
6	12	11	92	7.4	7.1	96

\* not detected; limit of detection = 0.43 milligrams/kilogram

<sup>#</sup> not detected; limit of detection = 0.60 milligrams/kilogram

NA: not applicable

<sup>a</sup> not detected; limit of detection = 0.061 milligrams/kilogram

<sup>b</sup> not detected; limit of detection = 0.047 milligrams/kilogram

**Table 7: Results for check sample exercise for the analysis of caprolactam (from nylon-6) in chicken**

Note: Results are from single determinations of each sample and are corrected for analytical recovery

<b>Sample</b>	<b>Spike level (milligrams/kilogram)</b>	<b>Level reported (milligrams/kilogram)</b>	<b>Measurement Accuracy (per cent)</b>
<b>Chicken</b>			
1	10	10	100
2	Blank	ND*	NA
3	37	40	108
4	10	11	110
5	37	37	100
6	Blank	ND*	NA

\* not detected; limit of detection = 0.67 milligrams/kilogram

NA: not applicable

**Table 8: Levels of 1,3-butadiene in polystyrene packaging samples and their associated foodstuffs**

Notes:

Each retail sample comprised a pack of 10 individual coffee filter cups. Where some but not all cups in a sample were tested, cups were taken at random from each pack (excluding the first and last). The maximum permitted quantity (QM) of 1,3-butadiene in a finished material or article = 1 mg/kg. The specific migration limit (SML) for 1,3-butadiene is: not detectable with a method having a detection limit of 0.02 mg/kg. Limits of quantification for this substance in associated with these cups were 0.0001 to 0.00081 mg/kg. The analytical tolerance associated with these measurements was 6.7 per cent.

Sample no.	Sample	1,3-butadiene level (mg/kg) in packaging	Mean 1,3-butadiene level (mg/kg) in packaging	1,3-butadiene level (mg/kg) in food
PS32	Individual coffee filter cups	Cup 2 0.26, 0.30 Cup 3 0.24, 0.28	0.27	Not found
PS36a(i)	Individual coffee filter cups	Cup 1 1.4, 1.4 Cup 2 1.1, 1.1 Cup 3 0.96, 1.1 Cup 4 1.6, 1.5 Cup 5 1.1, 1.0 Cup 6 0.97, 0.99	1.2	Not found
PS36a(ii)	Individual coffee filter cups	Cup 1 2.2, 2.0 Cup 2 1.8, 1.9 Cup 3 1.9, 1.8	1.9	Not found
PS36b(i)	Individual coffee filter cups	All 10 cups homogenised	0.55	Not analysed
PS40	Individual coffee filter cups	Cup 1 0.19, 0.21 Cup 2 0.41, 0.45 Cup 3 0.23, 0.30	0.30	Not found

**Table 9: Levels of vinyl chloride in polyvinyl chloride packaging samples**

The maximum permitted quantity (QM) of vinyl chloride in a finished material or article = 1 milligram/kilogram (ppm). The analytical tolerance associated with these measurements is 8.5 per cent. Worst case limit of quantification = 0.022 mg/kg. Full confirmation criteria (in terms of full scan spectra) could not be met at levels less than 0.10 mg/kg.

<b>Sample no.</b>	<b>Sample</b>	<b>Vinyl chloride level (mg/kg) in packaging</b>
PVC03	Baby corn	0.23*
PVC35	American style dips	0.13*

\* Mean of 2 analyses, one using standard addition.

**Table 10: Levels of caprolactam in nylon-6 packaging samples and their associated foodstuffs**

There is no maximum permitted quantity (QM) for caprolactam in a finished material or article. The specific migration limit (SML (T)) for caprolactam is 15 mg/kg. The analytical tolerance associated with the measurement of caprolactam in packaging was 9.0 per cent and in foods was 6.5 per cent

<b>Sample no.</b>	<b>Sample</b>	<b>Caprolactam level (mg/kg) in packaging*</b>	<b>Caprolactam level (mg/kg) in food</b>
NO1	Spaghetti bolognese <sup>a</sup>	1,100	3.6
NO3	Liver chubb paté	<LOD	11
NO10	Polony slicing sausage	<LOD	13
NO13	Traditional black pudding	<LOD	2.8
NO16	Sliced beef in gravy <sup>a</sup>	620	6.1
NO21	Fish steaks in butter sauce <sup>a</sup>	900	5.9
NO31	Bake-in-the-bag seasoning <sup>#</sup>	<LOD	12
NO31b	As above pre-cooking	2,600	
NO39	Haddock steaks <sup>a</sup>	490	6.5
NO49	Traditional white pudding	<LOD	3.1

\* The worst case limit of detection was 78 mg/kg

# A nylon bag used to cook chicken with added seasoning according to the instructions given on the packet.

<sup>a</sup> Boil-in-the-bag products