

**CHEMICALS USED IN PLASTIC MATERIALS AND ARTICLES IN CONTACT WITH FOOD: COMPLIANCE WITH STATUTORY LIMITS ON COMPOSITION AND MIGRATION – YEAR 2**

**Summary**

- This information sheet reports the results of the second year of this survey. This is the latest in a series of surveys that is testing for compliance with legislation on chemical migration, from plastic materials and articles in contact with food. Year 1 was reported in Food Survey Information Sheet no. 43/03. 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 were tested. Samples of the following were included in this survey: melamine-ware, and food packaged in either of two polyolefins (polyethylene and polypropylene) or in co-polymers containing vinyl acetate. Simulant exposed to 50 melamine-ware articles was analysed for melamine and formaldehyde (with hexamethylenetetramine expressed as formaldehyde). 100 samples of foods packaged in polyolefins were analysed for five alkenes: (1-pentene, 4-methyl-1-pentene, 1-hexene, norbornene, 1-octene). 50 samples of food packaged in co-polymers containing vinyl acetate were tested for this vinyl ester.
- The five alkenes and vinyl acetate were not found in food. Hence legal limits for these were not exceeded. Formaldehyde and melamine migrated, in most cases, at levels well below the limits in law. Melamine migrated from 43 out of 50 samples at 0.051-0.90 milligrams/square decimetre [ $\text{mg}/\text{dm}^2$ ] and 0.96-3.8 mg/kilogram [kg] – well below legal limits (5.0  $\text{mg}/\text{dm}^2$  and 30 mg/kg). Melamine did not migrate from the other seven samples. Formaldehyde migrated from all 50 samples tested. Its migration was within limits in law for 45 samples (0.055-2.3  $\text{mg}/\text{dm}^2$  and 3.6-12 mg/kg, compared to legal limits of 2.5  $\text{mg}/\text{dm}^2$  and 15 mg/kg). But in the other five samples formaldehyde levels

were clearly above the legal maximum. Indeed formaldehyde could be smelt during some of the laboratory testing of the illegal samples and the levels found could cause effects such as transient irritation of the mouth. The Food Standards Agency took immediate action, working with local enforcement authorities and the suppliers, so that the illegal products were withdrawn from the UK market and recalled from consumers who had purchased them. Related items in affected product ranges were also withdrawn. Local authorities visited the companies concerned to ensure that appropriate action was taken. The European Commission and other EU Member States were also informed so that they could take action.

## **Background**

### **The survey**

This is the second year of a continuing survey to test for compliance with legislation on chemical migration from plastic materials and articles in contact with food. Legislation governing the control of plastics in this country and the rest of the EU was described in detail in the report of the first year of this survey.<sup>1</sup> In summary, 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.

In Year 1 of the survey a need was identified for further work in applying Comité Européen de Normalisation (CEN) methods, intended for use with food simulants, to test samples of food for monomers. This has been taken forward with CEN. The first year of the survey also identified the need for research on variation of monomer levels in different samples of food-grade plastic. This was considered at the November 2003 meeting of the independent working party on food contact materials, which advises the Food Standards Agency.<sup>2</sup> A request for proposals on this research area will be made in a forthcoming issue of the Agency's Research Requirements Document.

The survey reported here tested whether the specific migration limit (SML) is being observed in practice for a range of widely used plastic materials. In some cases an SML (T) is quoted. This is the SML for food or food simulants expressed as total of

moiety/substance(s) indicated. SMLs are expressed as milligrams/kilogram (mg/kg) of food or of a specified food simulant.

Simulant or foodstuffs were tested for the respective monomers migrating from articles or packaging, as follows:

- Melamine-ware: melamine and formaldehyde (with hexamethylenetetramine expressed as formaldehyde).
- Polyethylene and polypropylene: 1-pentene, 4-methyl-1-pentene, 1-hexene, norbornene and 1-octene.
- Vinyl acetate-vinyl chloride and ethylene-vinyl acetate co-polymers: vinyl acetate.

The respective legislative limits are:

Melamine: SML 30 mg/kg

Formaldehyde: SML(T) 15 mg/kg (with hexamethylenetetramine expressed as formaldehyde)

1-Pentene: SML 5 mg/kg

4-Methyl-1-pentene: SML 0.02 mg/kg

1-Hexene: SML 3 mg/kg

Norbornene: SML 0.05 mg/kg

1-Octene: SML 15 mg/kg

Vinyl acetate: SML 12 mg/kg

## **Methodology**

### **Samples and timing**

Two hundred samples (50 melamine-ware food contact articles and 150 packaged foodstuffs, Table 1) were purchased mainly in northern England between February 2004 and April 2004. Analytical work was completed in April 2004. The results were collated, checked and reviewed in April and May 2004.

A wide variety of articles and food samples was obtained: bowls: 16 articles; cups/mugs: 19 articles; plates: 11 articles; spoons/forks: 2 sets; tray/dish: 2 articles; margarine: 8 samples; cheese: 10 samples; cottage cheese/coleslaw: 5 samples; yoghurt/dairy desserts: 8 samples; crisps/bagged snacks: 5 samples; chocolate bars: 5 samples;

biscuits: 17 samples; cereals: 5 samples; bread/bakery products: 7 samples; cakes: 5 samples; microwaveable foodstuffs: 12 samples; pot noodles: 2 samples; fruit and vegetables: 8 samples; meat: 5 samples; milk/flavoured milk/yoghurt drinks: 7 samples; fruit juice: 5 samples; savouries: 5 samples; cooked meat: 5 samples; sandwiches: 2 samples; salt portions: 2 samples; frozen food: 3 samples; other products: 19 samples.

Samples were purchased from a range of major and smaller national retail outlets as well as independent retail outlets and via the internet. The ratio of purchases between supermarket chains and small outlets was approximately 60:40. Similar numbers of supermarkets' own brand goods and other goods were obtained.

Nine examples of each melamine-ware sample were obtained to allow for possible further analysis, after initial testing. Each sample set was assigned a unique sample code. Three of the articles were labelled A, B and C and were used for the migration experiments. The remaining six articles were overwrapped in aluminium foil and stored at room temperature.

Food 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 sample was prepared for analysis. Any foodstuffs with on-pack instructions to be cooked in the packaging were prepared according to the instructions on the pack. Then the foodstuff was removed from the packaging, overwrapped in aluminium foil and stored in a freezer (-20°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 all melamine-ware articles and on the packaging of all food samples to confirm the plastic type. A few samples were not tested for migration (Table 1) because they were packaged in a different plastic or too many of a given type of food had been purchased.

### ***Melamine-ware***

*Determining compliance with the legislation*<sup>3</sup>

For articles with a capacity of less than 500 ml or more than 10 litres (L) the specific migration test results should be expressed in units of mg/dm<sup>2</sup>. In these cases the specific migration limits, expressed in mg/kg, are divided by the conventional conversion factor of 6 in order to express them in mg/dm<sup>2</sup>. Thus the SML of 30 mg/kg for melamine is equivalent to a limit of 5 mg/dm<sup>2</sup> and the SML(T) of 15 mg/kg for formaldehyde is equivalent to a limit of 2.5 mg/dm<sup>2</sup>. Similarly when the surface-to-volume ratio in actual use is not known (e.g. forks and spoons) the migration should also be expressed in units of mg/dm<sup>2</sup>. For articles with a capacity of greater than 500 ml but less than 10 L the migration should be reported as measured, i.e. in units of mgs of melamine per kg of simulant (mg/kg), assuming a density of 1.

Melamine-ware may come into contact with all types of foods. In such cases the CEN method states that it should be tested in 3 per cent (w/v) acetic acid in aqueous solution, 10 per cent ethanol and a fatty food simulant. However the greatest (i.e. worst case) migration is into acetic acid solution and this was therefore selected as the food simulant.<sup>4</sup> Three articles, labelled A, B and C, were exposed to 3 per cent (w/v) aqueous acetic acid for 2 hours at 70°C according to CEN methods EN1186 Part 9 for fillable articles and EN1186 Part 3 for non-fillable articles.<sup>5</sup> As all of the products purchased were intended for repeat use, three sequential exposures to the 3 per cent (w/v) aqueous acetic acid simulant were carried out, as described in CEN standard EN13130 Part 1.<sup>6</sup>

## **Melamine**

### *Overview*

Melamine, in 3 per cent (w/v) aqueous acetic acid, was determined by high performance liquid chromatography (HPLC) with ultra-violet (UV) detection. The method used was as described in CEN method EN13130 Part 27 but with an additional confirmation step with HPLC and mass spectrometric (MS) detection.<sup>6</sup> Quantification was achieved via external standard calibration using 3 per cent (w/v) aqueous acetic acid fortified with known amounts of melamine.

### *Standard solutions*

Stock standard solutions of melamine were prepared in 3 per cent (w/v) aqueous acetic acid at 1000 mg/L. Stock solutions were diluted with 3 per cent (w/v) aqueous acetic acid to form calibration standards (0 to 40 mg/L melamine). Between use standards were stored in the dark at +5°C.

### *Sample preparation*

The 3 per cent (w/v) aqueous acetic acid food simulant obtained as described above was analysed directly.

### *Analysis*

All samples of exposed simulant, blanks and standards were analysed by HPLC-UV for melamine using an Agilent HP1100 system with a diode array UV detector under the following conditions:

Column:	ODS1 200 mm x 4.6 mm, particle size 5 µm
Injection volume:	20 microlitres
Flow rate:	1.0 ml/minute
Mobile phase:	pH 6.5, 5 mM phosphate buffer:acetonitrile (90:10)
Detection:	UV, 230 nanometres (nm)

### *Monomer identification and quantification*

Calibration lines were constructed for the standard solutions by plotting the peak area of the melamine against the concentration of this monomer in the 3 per cent (w/v) aqueous acetic acid. The migration of melamine from the melamine-ware food contact articles was converted into units of mg/dm<sup>2</sup> where applicable (EN13130 Part 1) taking into account the volume of simulant used in the migration test and simulant contact surface area.<sup>6</sup>

### *Confirmation criteria*

All survey samples in which a UV response is observed should be analysed using diode array detection over the range 190 nm to 300 nm. The presence of melamine in these samples is confirmed by applying the following criteria:

- Melamine should have an absorbance peak maximum at 205 nm.
- The absorbance ratios 205:230:260 nm for the samples should agree to within ± 10 per cent of ratios for the standards (at concentrations as close as possible to each other).
- If the peak is pure, overlaid spectral profiles of the front, apex and tail of the peak should be identical. Therefore, if the three profiles are normalised, they should superimpose on top of each other.

If the level of melamine is found to be in excess of 50% of the SML the sample should be re-analysed by HPLC-MS in the full scan mode. The presence of melamine should be confirmed by applying the following criteria:

- The ion ratios for m/z 127/85 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.

## ***Formaldehyde***

### *Overview*

Formaldehyde in 3 per cent (w/v) aqueous acetic acid was determined spectrophotometrically. Formaldehyde was reacted with chromotropic acid in the presence of sulphuric acid. The resulting complex was measured using a spectrophotometer at 574 nm. Quantification was achieved by means of external standard calibration using 3 per cent (w/v) aqueous acetic acid fortified with known amounts of formaldehyde. The method used was that described in CEN method EN13130 Part 23.<sup>6</sup>

### *Standard solutions*

Stock standard solutions of formaldehyde were prepared in 3 per cent (w/v) aqueous acetic acid at 1500 mg/L. The strength of the formaldehyde solution was determined by titration. Stock solutions were diluted with 3 per cent (w/v) aqueous acetic acid to form calibration standards (0 to 30 mg/L formaldehyde). Standards were prepared monthly and stored between use at +5°C in the dark.

### *Sample preparation*

The 3 per cent (w/v) aqueous acetic acid food simulant obtained as described earlier was analysed directly.

### *Analysis*

All samples of exposed simulant, blank simulant and standards were subjected to a reaction procedure with chromotropic acid. The absorption of the resulting complex was measured at 574 nm. Any samples which gave a response at 574 nm outside the calibration range were diluted with 3 per cent (w/v) aqueous acetic acid and re-analysed.

### *Monomer identification and quantification*

Calibration curves were constructed for the standard solutions by plotting the absorbance of the formaldehyde-chromotropic acid complex at 574 nm against concentration of this monomer in the 3 per cent (w/v) aqueous acetic acid.

#### *Confirmation criteria*

The absorption of the reaction product should be measured over the range of 650 nm to 450 nm. The presence of formaldehyde in these samples should be confirmed by applying the criteria:

- Formaldehyde should have two absorbance peak maxima at 480 and 574 nm.

Additional confirmation of the presence of formaldehyde was carried out spectrophotometrically:

- Formaldehyde reacts with pentane-2,4-dione in the presence of ammonium acetate to form 3,5-diacetyl-1,4-dihydrolutidine. A response should be observed at 410 nm when the absorbance of the complex is measured using a spectrophotometer.

#### ***1-Pentene, 4-methyl-1-pentene, 1-hexene, norbornene and 1-octene***

##### *Overview*

1-Pentene, 4-methyl-1-pentene, 1-hexene, norbornene and 1-octene were determined by headspace gas chromatography coupled with mass spectrometry (HS-GC-MS). The method of analysis was based on CEN methods EN13130 Parts 25 and 26.<sup>6</sup> Quantification was achieved using cyclohexane as an internal standard. Calibration curves were obtained by analysis of water containing known amounts of the alkenes and internal standard.

##### *Standard solutions*

Stock solutions of 1-pentene, 4-methyl-1-pentene, 1-hexene, norbornene and 1-octene, and cyclohexane (internal standard) were prepared in *N,N*-dimethylacetamide at 2000, 1000, 2000, 1000, 3000 and 1000 mg/L respectively. Stock solutions were diluted with water to form calibration standards (0 to 5 mg/L 1-pentene, 0 to 0.02 mg/L 4-methyl-1-pentene, 0 to 3 mg/L 1-hexene, 0 to 0.05 mg/L norbornene and 0 to 15 mg/L 1-octene) each containing 0.05 mg/L cyclohexane as an internal standard. Fresh solutions were prepared monthly and stored in the dark at +5°C.

##### *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, then mixed with an

equal volume of water and an aliquot (5 g) weighed into a headspace vial. Internal standard was added at a level equivalent to 0.1 mg/kg in the foodstuff. Duplicate samples were prepared. Samples were incubated for 30 minutes at 80°C to release any volatile compounds from the foodstuff into the headspace.

### *Analysis*

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

Column: Restek Rt-QPLOT (divinylbenzene porous layer open tubular column), 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, split

Split ratio: 100:1

Syringe temperature: 90°C

Injection temperature: 280°C

Interface temperature: 280°C

Oven programme: 70°C hold for 2 minutes  
Raise to 220°C at 12.5°C per minute  
Hold for 10 minutes

Ions monitored: m/z 55 and 70 for 1-pentene  
m/z 56 and 84 for 4-methyl-1-pentene  
m/z 56 and 84 for 1-hexene  
m/z 66 and 94 for norbornene  
m/z 70 and 112 for 1-octene  
m/z 57 and 84 for cyclohexane

### *Monomer identification and quantification*

Calibration lines were constructed for the solvent standards by plotting:

- the peak area ratio of m/z 70 for 1-pentene versus m/z 84 for cyclohexane against the level of 1-pentene;
- the peak area ratio of m/z 56 for 4-methyl-1-pentene versus m/z 84 for cyclohexane against the level of 4-methyl-1-pentene;
- the peak area ratio of m/z 84 for 1-hexene versus m/z 84 for cyclohexane against the level of 1-hexene;
- the peak area ratio of m/z 66 for norbornene versus m/z 84 for cyclohexane against the level of norbornene; and
- the peak area ratio of m/z 112 for 1-octene versus m/z 84 for cyclohexane against the level of 1-octene.

For each survey sample 'spikes' were prepared by fortifying the foodstuffs with the alkenes of interest at concentrations equivalent to their respective specific migration limits and at one tenth of these levels. The levels in these 'spiked' samples were determined from the appropriate calibration graph. The results were used to determine analytical recovery.

#### *Confirmation criteria*

No migration was detected, but the following criteria were established before analytical work started. They are included here for completeness:

- The ion ratios for m/z 55/70 for 1-pentene, m/z 56/84 for 1-hexene and 4-methyl-1-pentene, m/z 66/94 for norbornene and m/z 70/112 for 1-octene 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 should agree to within  $\pm 2$  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 acetate***

##### *Overview*

Vinyl acetate was determined by HS-GC-MS. The method of analysis was based on CEN method EN13130 Part 9.<sup>6</sup> Quantification was achieved using methyl propionate as an internal standard. Calibration curves were obtained by analysis of water containing known amounts of vinyl acetate and internal standard.

### *Standard solutions*

Stock solutions of vinyl acetate and methyl propionate were prepared in *N,N*-dimethylacetamide at 3000 mg/L. Stock solutions were diluted with water to form calibration standards (0 to 6 mg/L vinyl acetate) each containing 6 mg/L methyl propionate as an internal standard. Fresh solutions were prepared monthly and stored in the dark at +5°C.

### *Sample preparation*

Solid foodstuffs were homogenised using a food blender and the homogenised food was slurried 1:3 with water. A specimen of the slurry (5 g) was transferred to a headspace vial. Liquid foods were stirred to ensure thorough mixing, mixed 1:3 with water and an aliquot (5 g) weighed into a headspace vial. Internal standard was added at a level equivalent to 12 mg/kg in the foodstuff. Duplicate specimens were prepared. Samples were incubated for 15 minutes at 80°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:	Restek Rt-QPLOT (divinylbenzene porous layer open tubular column), 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, split
Split ratio:	100:1
Syringe temperature:	90°C
Injection temperature:	280°C
Interface temperature:	280°C
Oven programme:	60°C hold for 2 minutes Raise to 280°C at 20°C per minute Hold for 2 minutes
Ions monitored:	m/z 43 and 86 for vinyl acetate

m/z 57 and 88 for methyl propionate

#### *Monomer identification and quantification*

Calibration curves were constructed for the solvent standards by plotting the peak area ratio of m/z 43 for vinyl acetate versus m/z 57 and 88 for methyl propionate against the level of vinyl acetate.

For each survey sample 'spikes' were prepared by fortifying the foodstuffs with vinyl acetate at a concentration of 12 mg/kg (the SML) and at one tenth of this level. The levels in these 'spiked' samples were determined from the appropriate calibration graph. The results were used to determine analytical recovery.

#### *Confirmation criteria*

No migration was detected, but the following criteria were established before analytical work started. They are included here for completeness:

- The ion ratios for m/z 43/86 for vinyl acetate 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  $\pm 2$  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.

#### **Quality assurance**

##### *'Spiked' samples*

Analysing representative foods and food simulants 'spiked' with appropriate levels of monomers tested the suitability of the analytical methods to determine monomers in foods and food simulants. Known volumes of standard solutions were allowed to infuse into the foods or food simulants prior to analysis. Measurement accuracy on spiked samples ranged from 88 to 115 per cent as demonstrated in the check sample exercises. Samples were prepared as replicate specimens (n=8) to obtain repeatability data.<sup>7</sup> Repeatability ranged from 0.16-8.5 per cent depending on the monomer and the food type/food simulant under investigation.

##### *Internal quality control*

For the foodstuffs a typical batch size was 5 survey samples, each prepared in duplicate. Each analytical batch also included at least one method blank and control samples. Two control samples were prepared for each food survey sample since recovery varied depending on the monomer and matrix being analysed. Control samples were prepared by fortifying the homogenised foodstuff with known masses of monomer at levels equivalent to the SML and one-tenth of the SML. 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.

For the simulants a typical batch size ranged from 2 to 5 survey samples, each prepared in duplicate. Each analytical batch also included at least one method blank. The simulant was analysed directly and matrix matched standards were prepared therefore recovery correction was not applicable.

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

Six food or food simulant samples were 'spiked' with each monomer so that they could be analysed 'blind' as a check of method accuracy. Samples were prepared at either the Procter Department of Food Science and Technology, University of Leeds, or the Department of Chemistry, University of Leeds. The samples were then supplied to the testing laboratory for analysis. In all cases the testing laboratory correctly identified the blanks and the 'spiked' samples. Food types and spiking levels used for each monomer were:

- For melamine (from melamine-ware articles) in food simulant, aliquots (30 ml) of 3 per cent (w/v) aqueous acetic acid were 'spiked' with a solution of formaldehyde (5.1 mg/ml in 3 per cent (w/v) aqueous acetic acid).
- For formaldehyde (with hexamethylenetetramine expressed as formaldehyde) from melamine-ware articles) in food simulant, aliquots (30 ml) of 3 per cent (w/v) aqueous acetic acid were 'spiked' with a solution of formaldehyde (1.4 mg/ml in 3 per cent (w/v) aqueous acetic acid).
- For 1-pentene, 4-methyl-1-pentene, 1-hexene, norbornene and 1-octene (from polyethylene and polypropylene) in food, portions of cereal (2.5 g) were spiked with a solution of 1-pentene, 4-methyl-1-pentene, 1-hexene, norbornene and 1-octene (0.50 mg/ml, 0.0020 mg/ml, 0.30 mg/ml, 0.005 mg/ml, and 1.5 mg/ml respectively in *N,N*-dimethylacetamide (DMA)).

- For vinyl acetate (from vinyl acetate-vinyl chloride and ethylene-vinyl acetate copolymers) in food, portions of biscuit (1.25 g) were spiked with a solution of vinyl acetate (0.30 mg/ml in DMA).

### *Reporting*

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

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

The analytical methodology used in this survey was shown to be working effectively before survey samples were obtained. There was good agreement between added and determined levels of monomers in the check sample exercises (Tables 2-9).

Testing of survey samples found no evidence of migration from polyethylene or polypropylene of the five alkenes tested (1-pentene, 4-methyl-1-pentene, 1-hexene, norbornene, 1-octene). The limits of detection depended on the foodstuff and analyte under investigation. The worst case limits of detection were: 1-pentene: 0.061 mg/kg (SML = 5 mg/kg); 4-methyl-1-pentene: 0.0020 mg/kg (SML = 0.02 mg/kg); 1-hexene: 0.095 mg/kg (SML = 3 mg/kg); norbornene: 0.0050 mg/kg (SML = 0.05 mg/kg); and 1-octene: 1.5 mg/kg (SML = 15 mg/kg).

Similarly vinyl acetate was not detected in any food sample. The limit of detection was dependent on the foodstuff. The worst case limit of detection was 0.14 mg/kg (SML = 12 mg/kg).

Formaldehyde and melamine migrated, in most cases, at levels well below the limits in law. Melamine migrated from 43 out of 50 samples of melamine-ware at 0.051-0.90 mg/dm<sup>2</sup> and 0.96-3.8 mg/kg – well below legal limits (5.0 mg/dm<sup>2</sup> and 30 mg/kg). Melamine did not migrate from the other seven samples. Formaldehyde migrated from all 50 samples tested. Its migration was within limits in law for 45 samples (0.055-2.3 mg/dm<sup>2</sup> and 3.6-12 mg/kg compared to legal limits of 2.5 mg/dm<sup>2</sup> and 15 mg/kg). But in the other five samples formaldehyde levels were clearly above the legal maximum. These samples were MEL19,

MEL27, MEL29, MEL37 and MEL50 (Table 10). Migration from these five samples was 8 to 76 times the legal limit. Following exposure to simulant, they became discoloured on the food contact surface, felt rough to the touch (pitted), and in some cases cracked or fell apart. At least one company undertook their own testing, the results of which supported these survey findings. Migration of formaldehyde in these five samples was clearly above the legal maximum. Indeed formaldehyde could be smelt during some of the laboratory testing of the illegal samples. Consumers' health might have been at risk. Although sustained exposure to formaldehyde would probably have been necessary for health to be at risk, formaldehyde is a carcinogen by inhalation if not necessarily from its presence in food. It is also a sensitiser, which may produce allergic dermatitis and transient irritation of the mouth.

The Food Standards Agency took immediate action to stop the sale of illegal melamine ware and encourage its return by the public to retailers. The Agency worked with local enforcement authorities and the suppliers. The illegal melamine-ware products were withdrawn from the UK market, recalls were put in place so that products purchased by consumers could be returned to the shops, recall notices were put in shops and related items in product ranges were also withdrawn. Local authorities visited the companies concerned to press for appropriate action to be taken. These processes appear to have been generally successful in removing illegal melamine-ware from the UK market. If any member of the public still has any of these products they are advised to return them to the retailer immediately.

Information on products with illegal migration of formaldehyde was communicated via *Rapid Alert System for Food and Feed* notices to the European Commission and hence to other EU Member States so that they could take action.

## **References**

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4. Ishiwata H., Inoue T. and Tanimura A., 'Migration of melamine and formaldehyde from tableware made of melamine resin.' Food Additives and Contaminants, 1986, (3), 63-70.
5. Comité Européen de Normalisation (CEN). European Standard EN1186. Materials and articles in contact with foodstuffs – Plastics. (Parts 3 and 9).
6. Comité Européen de Normalisation (CEN). European Standard EN13130. Materials and articles in contact with foodstuffs – Plastics substances subject to limitation. (Parts 1, 9, 23, 25, 26 and 27). Part 1 is in press as an EN standard, all other parts are currently in press as Technical Specifications.
7. International Organisation for Standardisation (ISO) Standard 78-2: Chemistry – Layouts for Standards – Part 2: Methods of Chemical Analysis (Second Edition, 1999).

### **Further information**

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This is the final report of year 2 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**

A few samples were not tested for migration because they were packaged in a different plastic or too many of a given type of sample had been purchased.  
NA: not applicable.

**Melamine-formaldehyde ware**

Code	Product	Brand	Contact area (dm <sup>2</sup> )	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
MEL01	Fimbles D-shape bowl	Trudeau	1.68	China	572-0140	0 065255 080948	NA	02/02/04	Boots
MEL02	Winnie the Pooh plate	Trudeau	1.67	China	5551050	0 065255 075173	NA	02/02/04	Boots
MEL03	Peter Rabbit bowl	euromark plc	1.63	China	00789	5 015717 007896	NA	02/02/04	Asda
MEL04	Animal print bowl (John Lennon real love)	Zak Designs Ltd.	1.75	China	JLNA-0362E	5 038202 007577	NA	02/02/04	Sainsbury's
MEL05	Boys mug	Asda Home	1.32	China	R3NS / ZCS390	2729 3423	NA	02/02/04	Asda
MEL06	Baby divided plate	Asda Home	2.38	China	R3NS / ZCS393	2729 3454	NA	04/02/04	Asda
MEL07	Tweenies bowl	Spearmark International Ltd.	1.94	China	5213	5 021703 052139	NA	02/02/04	Asda
MEL08	Barbie plate	Spearmark International Ltd.	2.55	China	5749/ Rev1	5 021703 157490	NA	02/02/04	Asda
MEL09	3 Piece spoon set (John Lennon real love)	Zak Designs Ltd.	0.45	China	JLNA3181E	5 038202 007584	NA	02/02/04	Sainsbury's
MEL10	Fork and spoon set	Asda Home Trends	1.06	None given	R1GV / ZCC453	2734 5580	NA	02/02/04	Asda
MEL11	Disney Princess Enchanting Beauties bowl	Trudeau	1.34	China	5341110AT	0 065255 073391	NA	03/02/04	Tesco

Code	Product	Brand	Contact area (dm <sup>2</sup> )	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
MEL12	Springfield soccer club bowl	Zak Designs Ltd.	1.39	China	SMPB1585E	5 038202 009182	NA	03/02/04	Tesco
MEL13	Fairy Girl bowl	Bang on the door™ licensed by Santoro	1.59	China	002 80	5 039388 007221	NA	03/02/04	Tesco
MEL14	Thomas the Tank Engine plate	Zak Designs Ltd.	2.03	China	TTTB0351E	5 038202 006471	NA	03/02/04	Tesco
MEL15	Bob the Builder cup	Born to Play	1.53	None given	BB 104	5 016126 681042	NA	07/02/04	Morrisons
MEL16	Barney bowl	Born to Play	1.94	None given	BN302	5 016126 613029	NA	07/02/04	Morrisons
MEL17	Humphrey's corner™ mug	euromark plc	1.38	China	00871	5 015717 008718	NA	11/02/04	Mothercare World
MEL 18	Transport design plate	None given	3.28	None given	None given	0195 0618	NA	14/02/04	Wilkinson
MEL 19	Striped mug	None given	2.07	None given	10 3701 style code TD 200200	5 050320 540781	NA	14/02/04	Internacionale
MEL20	Lilac bowl	Hi Gear	1.70	China	KD701LI	5 020148 062697	NA	Ordered: 11/02/04 Received: 16/02/04	www.ukcamping.co.uk
MEL21	White cup	None given	1.42	None given	W236	None given	NA	Ordered: 15/02/04 Received: 17/02/04	www.nisbets.co.uk
MEL22	Elmer plate	Elmer	2.43	China	EL 905	3 585190 449053	NA	Ordered: 14/02/04 Received: 19/02/04	www.childrenssalon.co.uk
MEL23	Anchor mug	euro-mel	1.94	None given	123	None given	NA	Ordered: 17/02/04 Received: 23/02/04	www.seamarknunn.com

Code	Product	Brand	Contact area (dm <sup>2</sup> )	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
MEL24	Taido mug and tray set	Gio'Style, Melamina	2.17	P.R.C	8785	8 009115 021292	NA	Ordered: 14/02/04 Received: 20/02/04	www.lakelandlimited.com
MEL25	Lemon tree plate	Royal Doulton	4.16	China	4660A58	0 55705 09327 7	NA	21/02/04	John Lewis
MEL26	Blue/white dessert bowl	Al fresco	2.67	None given	AM8970	5 015302 489700	NA	28/02/04	Max Value
MEL27	Flower mug	In the Pink Ltd.	1.86	None given	V-84023	5 050577 840238	NA	28/02/04	Everything £1
MEL28	Blue/white side plate	Royle Horizon	1.68	None given	AM6577	5 015302 465773	NA	28/02/04	Max Value
MEL29	Leaf tumbler	Woolbro	1.77	None given	55133	5 019702 551333	NA	28/02/04	Discount Warehouse
MEL30	Small flower plate	Zabar Housewares	1.30	China	None given	1 234567 152769	NA	28/02/04	Discount Warehouse
MEL31	Appetiser tray	PMS International	6.52	None given	083/799	5 022896 837992	NA	28/02/04	Home Bargains
MEL32	White cup	CTC	1.42	China	6025	None given	NA	28/02/04	A. Thompson
MEL33	White bowl	CTC	2.03	China	5006	None given	NA	28/02/04	A. Thompson
MEL34	Mermaid bowl	None given	1.96	None given	PLU175391	5 029551 849949	NA	28/02/04	Poundstretcher
MEL35	Marble green mug	Gimex®	1.90	Germany	5004	4 024374 511944	NA	06/03/04	Yorkshire Leisure Shop
MEL36	Blue/white bowl	Gio'style Melamina	1.93	P.R.C.	None given	8 009115 020592	NA	06/03/04	Yorkshire Leisure Shop
MEL37	Tuscany tumbler	Flamefield melamine essentials euro-mel	1.65	Turkey	117	5023412701179	NA	Ordered: 24/02/04 Received: 11/03/04	www.chemi-clean.co.uk

Code	Product	Brand	Contact area (dm <sup>2</sup> )	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
MEL38	Tuscany mug	Flamefield melamine essentials euro-mel	1.97	Turkey	123	5 023412 701230	NA	Ordered: 24/02/04 Received: 11/03/04	www.chemi-clean.co.uk
MEL39	Azores mug	Marine Business	1.97	Spain	None given	1018004 (for set of 6)	NA	Ordered: 11/02/04 Received: 17/03/04	www.galleyslave.co.uk
MEL40	3oz fluted white ramekin dish	None given	0.64	U.S.A.	97:32:130	None given	NA	Ordered: 14/02/04 Received: 19/03/04	www.andrewscateringequipment.com
MEL41	Animal design mug	Spearmark International Ltd.	1.67	China	PE198GE Toys R Us Ref no.5021	5 021703 050210	NA	20/03/04	Toys 'R' Us
MEL42	Thomas the Tank Engine divided plate	Zak Designs Ltd.	3.06	China	TMSH0351E	5 038202 010263	NA	21/03/04	Woolworths
MEL43	Stripe mug	None given	1.97	Taiwan	P.O.S. 270303/012	5 050595 151033	NA	23/3/04	Homebase
MEL44	Peter Rabbit cup	euromark plc	1.41	China	00790	5 015717 007902	NA	23/03/04	Asda
MEL45	Teletubbies bowl	Zak Designs Ltd.	1.69	China	00139	6 820990 000002	NA	23/03/04	Matalan
MEL46	Green beaker	Habitat	2.13	None given	None given	971847	NA	23/03/04	Habitat
MEL47	Patterned plate	Habitat	4.72	None given	None given	971845	NA	23/03/04	Habitat
MEL48	Butterfly and plant mug	The Garden at Debenhams	1.90	Thailand	1267 6 100 6057 33 green	8 5130 663	NA	25/03/04	Debenhams
MEL49	Stripe bowl	Sainsbury's	2.58	China	256/006 T1221	0103 0112	NA	25/03/04	Sainsbury's Savacentre

Code	Product	Brand	Contact area (dm <sup>2</sup> )	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
MEL50	Diagonal striped bowl	None given	2.54	None given	2593022	5 010251 159025	NA	25/03/04	Morrisons

### Polyolefin packaging

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
ALK01	Olive reduced fat spread	Sainsbury's	250 g	Republic of Ireland	20:27 E	0044 9120	20 MAY 04	02/02/04	Sainsbury's
ALK02	Fresh custard	Sainsbury's be good to yourself	500 g	None given	09:37 V1S	0123 6514	09FEB	02/02/04	Sainsbury's
ALK03	Spinach and ricotta pasta sauce	Sainsbury's be good to yourself	300 g	None given	None given	0134 8606	11 FEB 04	02/02/04	Sainsbury's
ALK04	Weetabix	Weetabix	225 g	England	4022 19:03 E	5 010029 000504	JAN 05	02/02/04	Sainsbury's
ALK05	Mars drink	Mars	330 g	UK	U4 16:01 *	5070 6464	06/03/04	02/02/04	Sainsbury's
ALK06	Tuna & sweetcorn sandwich filler	Tesco	170 g	UK	None given	0303 8635	08 FEB	03/02/04	Tesco
ALK07	Vegetable stock	Tesco	300 ml	UK	None given	5 010204 263243	13 FEB	03/02/04	Tesco
ALK08	Pasteurised standardised homogenised whole milk	Tesco	568 ml	UK	E31603L	5 031021 057976	10 FEB	03/02/04	Tesco
ALK09	Simply fruit organic strawberry and apple	Heinz	120 g	France	3393 00:01 864A	5015 7402	04 DEC 04	03/02/04	Tesco
ALK10	Actimel original 0% fat	Danone	4 x 100 g	Belgium	0608 1501 14 B	5 099417 010622	16/02	03/02/04	Tesco
ALK11	Rice & tomato pot meal	Barkat	71 g	None given	32091 07:14	8 043450 000598	28/07/04	04/02/04	Asda
ALK12	Creamy tomato soup	Asda	500 g	UK	None given	W 2746 4779 G	09 FEB 04	04/02/04	Asda
ALK13	Yazoo milk drink banana flavour	Campina	500 ml	None given	L530 13:20	5 410438 008465	30/04/2004	04/02/04	Asda

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
ALK14	Philadelphia	Kraft	125 g	Germany	FA 12:14	5086 8124	14 MAY 2004	04/02/04	Asda
ALK15	Aero chocolate mousse	Nestle	100 g	None given	None given	5 011546 455143	20 FEB	04/02/04	Asda
ALK16	Chocolate cornflake mini bites	Marks & Spencer	190 g	UK	None given	M 0347 884 S	25 FEB 04	05/02/04	Marks & Spencer
ALK17	Soup broccoli & Melton Mowbray stilton	Marks & Spencer	600 g	UK	None given	M 0335 041 S	12 FEB	05/02/04	Marks & Spencer
ALK18	Strawberry trifle	Marks & Spencer count on us...	140 g	UK	None given	M 0268 936 S	09 FEB	05/02/04	Marks & Spencer
ALK19	Tomato & mascarpone sauce	Marks & Spencer	350 g	UK	None given	M 0300 780 S	11 FEB	05/02/04	Marks & Spencer
ALK20	Oakham white 4 chicken fillets	Marks & Spencer	520 g	UK	None given	M 0210 409 S	11 FEB	05/02/04	Marks & Spencer
ALK21	Tuna snack pots Italian	Weght Watchers from Heinz	240 g	Germany	STI-210-2	5017 1705	AUG -2004	07/02/04	Boots
ALK22	Refreshing cranberry juice drink	Boots Shapers	250 ml	Produce of more than one country Made in the UK	None given	5 045094 623624	19 FEB	07/02/04	Boots
ALK23	Saccharin tablets	Boots	1000 tablets	England	4WW	0263 7495	NOV 06	07/02/04	Boots
ALK24	Cream of tomato soup	Heinz	275 ml	EU	324A 3 276 13:38	5 000157 061232	12-2004	07/02/04	Boots
ALK25	Double Decker	Cadbury	65 g	None given	L3315 21:45 B	5 034660 500926	08 04	07/02/04	Boots
ALK26	Coco Pops	Kellogg's	45 g	None given	12 16:25 MC	5 000127 114753	02 12 04	07/02/04	Morrisons
ALK27	Strawberry Nesquik	Nestle	375 g	None given	11:58 FRPORIJ	3 033710 079391	10.04.2005	07/02/04	Morrisons

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
ALK28	<b>Deleted due to too many fruit samples having been purchased</b>								
ALK29	Scotch broth soup	Morrisons	500 g	None given	A20 BY 17:50 0655	2 310095 701293	FEB 23	07/02/04	Morrisons
ALK30	Cucumber & mint raita	Morrisons	70 g	None given	0904 D	2 312003 000399	23 FEB	07/02/04	Morrisons
ALK31	Original Florida style Sunny D	Sunny Delight	1.5 L	UK	4012109910 1309	4 038300 501442	10-05-04	07/02/04	Iceland
ALK32	Fresh double cream	Iceland	284 ml	None given	LX D2 0951	5 010482 280284	15 FEB	07/02/04	Iceland
ALK33	Tomatoes	Iceland	750 g	Canary	0603 955 E53038	5 010482 290108	10 FEB	07/02/04	Iceland
ALK34	<b>Incorrect packaging material</b>								
ALK35	Snack stop bolognese style pasta	Crosse & Blackwell	98 g	None given	3259 08:47	5 050665 000018	SEP 2004	07/02/04	Iceland
ALK36	Nice quality plain biscuits	Safeway	200 g	UK	4009 NAU	0501 7119	24 JUL 04	12/02/04	Safeway
ALK37	Brown original wheatgerm medium sliced bread	Hovis	400 g	None given	(X12) 2	5 010003 000407	13 FEB	12/02/04	Safeway
ALK38	Fresh single cream	Safeway	284 ml	UK	RX F2 1107	0500 8551	18 FEB	12/02/04	Safeway
ALK39	Dairylea spread	Kraft	200 g	Belgium	NA 15:28 B	5022 4050	06 JUN 04	12/02/04	Safeway
ALK40	Chicken goujons	Safeway	400 g	UK	192 04210 2084/7	0660 4585	19 FEB	12/02/04	Safeway
ALK41	Italian grated cheese	Buitoni	80 g	Italy	L3212F2	5 011546 414997	JUL 2004	12/02/04	Somerfield
ALK42	Olive reduced fat spread	Somerfield	500 g	Republic of Ireland	01:35 JJ	2902 5251	28 APR 04	12/02/04	Somerfield
ALK43	Frijj chocolate flavoured milkshake	Dairy Crest	500 ml	None given	08:53 1E	5 000295 061422	27/02/04	12/02/04	Somerfield
ALK44	Custard and sponge jam Müllerpuds	Müller	175 g	None given	11:30F1NP	4 025500 021948	19 FEB 04	12/02/04	Somerfield
ALK45	Cottage cheese	Somerfield	250 g	None given	None given	2904 5860	24 FEB	12/02/04	Somerfield

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
ALK46	Maltesers	Masterfoods	37 g	None given	#06 U6B	5 000159 020312	05-06-04	15/02/04	Spar
ALK47	Liegeois chocolate dessert	Schweyer	200 g	Germany	MN	4033 8453	08.03.04	15/02/04	Spar
ALK48	Pure orange juice	Spar	250 ml	None given	01:22	5 010358 060316	25 FEB	15/02/04	Spar
ALK49	Strawberry flavour shake	MilkyWay	500 g	UK	EEC MN003M U4 U.K. 14:10 B	5 023471 105376	19 JUNE 2004	15/02/04	Spar
ALK50	Semi-skimmed milk	Spar	500 ml	None given	LV00723 11:11A	5 010358 249582	21 FEB	15/02/04	Spar
ALK51	Fruit juice mousse	Danone Shape	100 g	Belgium	A2 21H02	5053 8645	03/03	18/02/04	Co-op
ALK52	Real tropical fruit juice drink	St Ivel	1 L	None given	11:07 <K1>	5 000295 113800	28 FEB	18/02/04	Co-op
ALK53	<b>Deleted due to too many fruit samples having been purchased</b>								
ALK54	Extra mature cheddar	Pilgrims Choice	200 g	None given	SS207M 61176413	5 018929 009092	12 APR	18/02/04	Sainsbury's
ALK55	Baby potatoes with mint and parsley	Morrisons	468 g	None given	FT	0 260243 000995	21 FEB	18/02/04	Morrisons
ALK56	Salt & vinegar flavour potato crisps	Seabrook	31.8 g	None given	P7 11:48	5 016451 761136	29 APR 04	18/02/04	Morrisons
ALK57	Medium cheddar	Tesco	0.266 kg	None given	19:38 XCII	0 228152 001281	31 MAR	18/02/04	Tesco
ALK58	Buttery light touch	Pura	420 ml	None given	3331 HA	5033 5626	NOV 04	18/02/04	Tesco
ALK59	Thin arrowroot biscuits	Crawford's	200 g	UK	T2 A 20:56	5 000168 001265	17 APR 04	18/02/04	Spar
ALK60	Sultana bran	Kwik Save	500 g	UK	3 257 W 09:42	5 000192 274789	SEP 2004	19/02/04	Kwik Save
ALK61	Easter jelly mix	Royalle	500 g	None given	L:3342	5 016002 015343	MAY 2005	19/02/04	Kwik Save
ALK62	Queen olives with herbs	Safeway The Best	100 g	Spanish olives, Herbs de Povenca	None given	0659 8914	05 MAR	19/02/04	Safeway

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
ALK63	Margarine	Stork	500 g	None given	13:19 V357	5024 1316	10-04-04	19/02/04	Healds Day & Nite Grocers
ALK64	Stir-in sun-dried tomato sauce	Dolmio	150 g	EU	14:39 ISD 835	5 010034 592605	21/04/05	19/02/04	Healds Day & Nite Grocers
ALK65	Corned beef and onion sandwich filling	Richardsons Foods	0.200 kg	None given	None given	5 034141 008996	26 FEB 04	19/02/04	Netto
ALK66	Red grapes	Netto	500 g	Argentina	D47346	5 021490 898293	21 FEB	19/02/04	Netto
ALK67	Lemon juice	Just lemon	200 ml	None given	L4036	2506 0416	03.02.05	20/02/04	Aldi
ALK68	Smoky barbecue marinade	Aldi	375 g	None given	L3174	No numbers given	23/03/04	20/02/04	Aldi
ALK69	<b>Deleted due to too many fruit samples having been purchased</b>								
ALK70	White choc dips	KP	32 g	England	4 16:27	5023 7111	07 AUG 04	20/02/04	Broadway Superstore
ALK71	Malt vinegar	Sarson's	300 ml	None given	L3245J 19:37	5035 4283	None given	21/02/04	P & L Convenience Stores
ALK72	Strawberry jelly	Rowntrees	125 g	UK	L3342H	5035 4801	AUG 2004	21/02/04	Kingswood Supermarket
ALK73	Penguin flipper dipper	M <sup>c</sup> Vities's	50 g	UK and Holland	X L4022	5016 8231	30 AUG 04	21/02/04	M <sup>c</sup> Coll's
ALK74	Plum & tamarind stir-fry sauce	Eastern Inspirations	145 g	UK	AM1	5 024530 004098	10 MAR	21/02/04	Waitrose
ALK75	Benecol light	Benecol	200 g	None given	F160I62EY 12:21	6 411200 034964	28.04-04	21/02/04	Waitrose
ALK76	Fresh real mayonnaise	Fresh Ideas	250 g	UK	69P	5 01045 000166	15 MAR 04	21/02/04	Waitrose
ALK77	Orange flapjack fingers	Waitrose	5 x 32 g	UK	None given	5 000169 028605	07 JUN 04	21/02/04	Waitrose
ALK78	Oriental spice Thai bites	Jacobs	100 g	None given	4006A1	5 000137 929958	02.10.04	21/02/04	Waitrose
ALK79	Fresh amatriciana sauce	Waitrose	350 g	Produced in th UK from EU pork	AM1	5 000169 960578	07 MAR	21/02/04	Waitrose

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
ALK80	Bounty	Masterfoods	57 g	None given	N1C2	4011 1001	17-07-04	21/02/04	Karson Newsagent
ALK81	Milk chocolate digestives	Day-Today	200 g	UK	AA N02 15:39	5 016089 041761	15 MAY 04	24/02/04	Kingsway News
ALK82	Cheese spread	LifeStyle	150 g	Ireland	08:00	5 013668 125614	END APR 04	24/02/04	Kingsway News
ALK83	Sunflower spread	Golden Sun	500 g	None given	None given	2001 8139	29 06 04	28/02/04	Lidl
ALK84	Ready salted chipsticks	Smiths		None given	GBS215033 0663 23:26	5032 8925	08 MAY 04	28/02/04	Alldays
ALK85	Margarine	Flora pro-active	250 g	None given	17:50 YO41	5 000118 042553	26-06-04	28/02/04	Alldays
ALK86	Thick sliced white bread	Londis	800 g	UK	SK6 F0042 4058	5 011406 901094	03 MAR	28/02/04	Hiccups
ALK87	Cheddar	Londis	200 g	UK	TL	5 011406 101197	27 APR	28/02/04	Hiccups
ALK88	Medium sliced white bread	Roberts bakery	800 g	None given	2 WXOR AJBQF	5 011579 450085	06 MAR 04	28/02/04	Hobsons Choice
ALK89	Thai green curry sauce	Uncle Ben's	170 g	Holland	18:35 4A	4 002359 641985	04/08/04	01/03/04	Asda
ALK90	Four cheese pasta sauce	Asda	350 g	UK	None given	W 2742 8801 G	09 MAR	01/03/04	Asda
ALK91	Johnny's onion rings	Golden Cross Snacks	14.5 g	None given	4035D	5 016491 123703	16 JUL 04	28/02/04	Key News Plus
ALK92	Golden lights sour cream & onion crisps	Golden Wonder	21 g	None given	4008 16:27 W1	5 010052 104651	01 MAY 04	28/02/04	Key News Plus
ALK93	Margarine	"I can't Believe it's not Butter"	500 g	None given	07:10 W004	5 000241 007009	24-04-04	02/03/04	Heolgerrig Stores
ALK94	<b>Incorrect packaging material</b>								
ALK95	Margarine	Clover	500 g	None given	07:54	5 000295 008069	28 APR 04	03/03/04	Keelings
ALK96	Corn flakes	Kellogg's	250 g	None given	05 10:12 MC	5 000127 010093	27 01 05	03/03/04	Keelings

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
ALK97	Sun ripened tomato & basil pasta sauce	Dolmio Express	170 g	Holland	00 : 29 4B	4 002359 640469	30/11/04	03/03/04	Jacksons
ALK98	<b>Incorrect packaging material</b>								
ALK99	Mini chicken breast fillets	Sainsbury's	238 g	British	S0031G 10:01 25 C	00520133	06 MAR	03/03/04	Sainsbury's
ALK100	Mature cheddar	Wyke Farms	250 g	Somerset	PM 043 0620	5 021427 000317	14.05.04	03/03/04	Broadway Service Station
ALK101	Chicken drumsticks	Iceland	720 g	UK	18 MAR 1927	5 010482 305574	26 MAR	20/03/04	Iceland
ALK102	Sliced tin premium white bread	The Village Bakery	800 g	Wales	4 16:57	5 016926 010103	23 MAR	20/03/04	Barry James, Caergwrle Filling Station
ALK103	Frosties to go	Kellog's	40 g	None given	029 13:38 MC	5 000127 151765	07 JUN 04	20/03/04	Forbuoys
ALK104	Fruity sauce	Country Lane	485 g	None given	3283H 17:24	5 026043 016407	OCT 2004	20/03/04	Food co.
ALK105	Mature white cheddar cheese	Littleton	0.202 kg	None given	None given	2 025810 001113	10.05.04	20/03/04	Food co.
ALK106	Farmhouse soft white	Marks & Spencer	400 g	None given	(SEB) 2	M 0263 290 S	26 MAR	23/03/04	Marks & Spencer

### Vinyl acetate copolymer packaging

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
VA01	Shortbread fingers	Cookie Coach	50 g	None given	E3310	5 034782 000656	31 MAY 04	21/02/04	Roadchef
VA02	Pink 'n' white jammies	Caxton	100 g	None given	3275B	5 010074 531787	03 APR 04	21/02/04	Broadway Superstore,
VA03	Mini classics 6 victoria sponge cakes	Mr Kipling	222 g	None given	03 6NH	5 000221 015628	03 MAR	21/02/04	Waitrose

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
VA04	<b>Incorrect packaging material</b>								
VA05	Chocolate brownie	McVitie's	93 g	UK	F1Y2	5 000168 091525	21 APR 04	20/03/04	Forbuoys
VA06	6 Snowballs	Bobby's	110 g	None given	4036A	5 015282 118966	31 JUL 04	20/03/04	Barry James, Caergwrle Filling Station
VA07	Thick sliced white bread	William Jackson	800 g	None given	RSTB	5 000227 021036	07 MAR	03/03/04	Jacksons
VA08	Medium sliced white bread	Warburtons	400 g	None given	4IZPW EPBP4	5 010044 001234	11 FEB	07/02/04	Iceland
VA09	6 Snack sausage rolls	Tesco	192 g	UK	02:53 4F	5 050179 845013	04 APR	30/03/04	Tesco
VA10	<b>Incorrect packaging material</b>								
VA11	Carrot and sultana salad	Sainsbury's be good to yourself	200 g	UK	14091	0105 1094	05APR	30/03/04	Sainsbury's
VA12	Roast chicken sandwich	Boots Shapers	175 g	None given	None given	0363 4967	01 APR	31/03/04	Boots
VA13	Bacon, lettuce and tomato sandwich	Marks & Spencer	194 g	UK	09:10	M 0680 158 S	01 APR	31/03/04	Marks & Spencer
VA14	Sausage dippers	Asda	105 g	UK	AZN (Code on dip 41603 13.00)	P 2041 1633 L	05 APR	31/03/04	Asda
VA15	6 Mini pork pies	Bowyers	300 g	None given	None given	5 000275 002544	04 APR	31/03/04	Asda
VA16	<b>Incorrect packaging material</b>								
VA17	<b>Incorrect packaging material</b>								
VA18	All butter melters	Haywood & Padgett's	316 g	None given	07700:47L1	5 018156 200163	09 MAY 04	31/03/04	Asda
VA19	<b>Incorrect packaging material</b>								
VA20	Mozzarella & cherry tomato pasta salad	Boots	200 g	None given	LINE 7 12:16 86	0475 2844	02 APR	31/03/04	Boots

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
VA21	Chocolate chunk cookies	Cadbury	200 g	UK	4061 A 20:12	0 72417 08313 6	29 11 2004	05/04/04	Safeway
VA22	10 Cooked pork cocktail sausages	Marks & Spencer	100 g	UK	None given	M 0159 098 S	10 APR	05/04/04	Marks & Spencer
VA23	Six Yorkshire puddings	Marks & Spencer	132 g	UK	None given	M 0798 716 S	09 APR	05/04/04	Marks & Spencer
VA24	Cooking salt	Safeway	1.5 kg	UK	4069N	0500 1880	None given	05/04/04	Safeway
VA25	4 Empire biscuits	Sainsbury's	270 g	None given	0752 06	0037 0455	10 Apr	07/04/04	Sainsbury's
VA26	6 Scotch pancakes	Co-op	180 g	UK	X42	5 000128 310444	08 APR	07/04/04	Co-op
VA27	<b>Incorrect packaging material</b>								
VA28	2 Smoked salmon parcels	Somerfield	110 g	UK	None given	5 000192 292981	12 APR	07/04/04	Somerfield
VA29	Cooked turkey	Bernard Matthews	150 g	None given	None given	5 010236 018767	17 APR	07/04/04	Somerfield
VA30	Honey roast ham	Safeway	125 g	UK	None given	0644 9087	25 APR	07/04/04	Safeway
VA31	Prime cooked ham	Nisa	200 g	UK	None given	5 010893 780687	19 APR	07/04/04	Costcutter
VA32	Reduced sugar shortbread	Tesco	150 g	UK	4021	5 000436 323808	07 AUG 04	13/04/04	Tesco
VA33	White seedless grapes	Tesco	400 g	Chile	GA55967	1005 4239	18 APR	13/04/04	Tesco
VA34	Figs 4 pack	Tesco	120 g	Brazil	None given	1003 0509	15 APR	13/04/04	Tesco
VA35	6 Real fruit jam tarts	Mr Kipling	210 g	None given	089 EB	5 000221 002253	22 APR	13/04/04	Spar
VA36	Fresh dips	The Fresh Dip Company	340 g	UK	18:45	5 024530 000151	19 APR	14/04/04	Kwik Save
VA37	8 Scotch pancakes	Kwik Save	240 g	UK	X42	5 000192 290260	19 APR	14/04/04	Kwik Save
VA38	Half fat mature 8 cheese slices	Morrisons	160 g	None given	DR	2 311443 400998	02 JUN	14/04/04	Morrisons
VA39	Dolce Fritas	Aviko	600 g	None given	L0203287 08:11	8 710449 003846	04 2005	14/04/04	Sainsbury's
VA40	Garlic & herb roule	Co-op	100 g	France	None given	0295482001304	04 MAY	14/04/04	Co-op

Code	Product	Brand	Size / Product weight	Country of origin	Batch code	Barcode	Best-before-date	Date of purchase	Retail outlet
VA41	Eccles cakes	Lowthers	150 g	None given	34085	5 026420 320332	17 JUN 04	14/04/04	One Stop Convenience Stores
VA42	Mature white sliced cheddar	The Cheese Company	160 g	None given	None given	5 000246722044	21 MAY	14/04/04	Iceland
VA43	Boasters	McVitie's	150 g	UK	CCW	5 000168 015477	24 JUL 04	14/04/04	Iceland
VA44	Table salt	Sainsbury's	500 g	None given	3329S	0057 1791	None given	14/04/04	Sainsbury's
VA45	Breaded chicken burgers	Iceland	456 g	None given	L4 026 19 A1	5 010482 240134	26Jan05	15/04/04	Iceland
VA46	20 Pork, sage & onion stuffing balls	Morrisons	500 g	None given	None given	5 010251 154280	FEB 2005	14/04/04	Morrisons
VA47	Dried soft prunes	Morrisons	250 g	U.S.A	4091B	0 267183 000993	11 MAY 2004	14/04/04	Morrisons
VA48	Caramel shortcake	Boots	70 g	None given	None given	0267 5046	17 JUN 04	15/04/04	Boots
VA49	Cadbury fingers	Cadbury	150 g	UK	None given	5 000183 948101	28 11 04	15/04/04	Spar
VA50	Roast beef	Spar	110 g	None given	None given	0 205797 001609	22 APR	15/04/04	Spar
VA51	Havarti slices	Waitrose	250 g	None given	CR	5 000169 025567	28 MAY	17/04/04	Waitrose
VA52	Four Easter biscuits	Cotswold cookies	150 g	None given	7621	5031368076210	JUL 04	17/04/04	Londis
VA53	Shortbreads rounds	Duncan's of Deeside	150 g	Scotland	A2	5 017605 000019	AUG 04	17/04/04	Costcutter
VA54	Pork luncheon meat	Mattesons	110 g	None given	None given	5 000178 101962	22 APR	17/04/04	One Stop Convenience Store
VA55	Duck & orange paté	Nisa	170 g	None given	504851	5 010893 710103	14 MAY	17/04/04	Booths
VA56	2 Chocolate digestive slices	McVitie's	58 g	UK	PW	5 000168 109602	22 MAY 04	19/04/04	Premier News, Food and Wine

**Table 2: Results from check sample exercise for the analysis of melamine in 3 per cent (w/v) aqueous acetic acid**

Results are 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>
1	17	17	100
2	17	17	100
3	38	39	103
4	38	39	103
5	Blank	ND	NA
6	Blank	ND	NA

ND: not detected; limit of detection = 0.085 mg/kg

NA: not applicable

**Table 3: Results from check sample exercise for the analysis of formaldehyde in 3 per cent (w/v) aqueous acetic acid**

Results are 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>
1	Blank	ND	NA
2	Blank	ND	NA
3	19	19	100
4	19	19	100
5	6.0	6.3	105
6	6.0	6.4	107

ND: not detected; limit of detection = 0.12 mg/kg

NA: not applicable

**Table 4: Results from check sample exercise for the analysis of 1-pentene in cereal**

Results are 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>
1	2.0	2.2	110
2	Blank	ND	NA
3	Blank	ND	NA
4	8.0	8.6	108
5	8.0	8.7	109
6	2.0	2.3	115

ND: not detected; limit of detection = 0.0030 mg/kg

NA: not applicable

**Table 5: Results from check sample exercise for the analysis of 4-methyl-1-pentene in cereal**

Results are 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>
1	0.0080	0.0087	109
2	Blank	ND	NA
3	Blank	ND	NA
4	0.032	0.034	106
5	0.032	0.032	100
6	0.0080	0.0088	110

ND: not detected; limit of detection = 0.00080 mg/kg

NA: not applicable

**Table 6: Results from check sample exercise for the analysis of 1-hexene in cereal**

Results are 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>
1	1.2	1.2	100
2	Blank	ND	NA
3	Blank	ND	NA
4	4.8	4.5	94
5	4.8	4.7	98
6	1.2	1.3	108

ND: not detected; limit of detection = 0.0018 mg/kg

NA: not applicable

**Table 7: Results from check sample exercise for the analysis of norbornene in cereal**

Results are 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>
1	0.020	0.021	105
2	Blank	ND	NA
3	Blank	ND	NA
4	0.080	0.083	104
5	0.080	0.077	96
6	0.020	0.021	105

ND: not detected; limit of detection = 0.00070 mg/kg

NA: not applicable

**Table 8: Results from check sample exercise for the analysis of 1-octene in cereal**

Results are 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>
1	6.0	5.3	88
2	Blank	ND	NA
3	Blank	ND	NA
4	24	23	96
5	24	23	96
6	6.0	6.2	103

ND: not detected; limit of detection = 0.0090 mg/kg

NA: not applicable

**Table 9: Results from check sample exercise for the analysis of vinyl acetate in biscuit**

Results are 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>
1	7.2	7.6	106
2	7.2	8.1	113
3	17	18	106
4	17	16	94
5	Blank	ND	NA
6	Blank	ND	NA

ND: not detected; limit of detection = 0.020 mg/kg

NA: not applicable

**Table 10: Levels of melamine and formaldehyde (with hexamethylenetetramine expressed as formaldehyde) in simulant associated with melamine-ware.**

By convention, for articles with a capacity of less than 500 ml or more than 10 L the migration is expressed in mg/dm<sup>2</sup>; for articles with a capacity greater than 500 ml but less than 10 L migration is expressed in mg/kg. In compliance with the law, results are given for the third exposure (average of triplicate specimens).

Code	Product	Melamine (mg/dm <sup>2</sup> unless specified)	Formaldehyde (mg/dm <sup>2</sup> unless specified)
MEL01	Fimbles D-shape bowl	0.33	0.81
MEL02	Winnie the Pooh plate	<LOQ <sup>a</sup>	0.055
MEL03	Peter Rabbit bowl	0.41	0.83
MEL04	Animal print bowl (John Lennon real love)	0.37	0.86
MEL05	Boys mug	0.44	0.91
MEL06	Baby divided plate	<LOQ <sup>a</sup>	0.14
MEL07	Tweenies bowl	0.90	2.2
MEL08	Barbie plate	0.30	0.70
MEL09	3 Piece spoon set (John Lennon real love)	0.68	2.3
MEL10	Fork and spoon set	0.68	1.2
MEL11	Disney Princess Enchanting Beauties bowl	0.41	0.93
MEL12	Springfield soccer club bowl	0.18	0.73
MEL13	Fairy Girl bowl	0.41	1.0
MEL14	Thomas the Tank Engine plate	0.13	0.67
MEL15	Bob the Builder cup	0.22	0.77
MEL16	Barney bowl	0.33	0.95
MEL17	Humphrey's corner™ mug	0.17	0.55
MEL18	Transport design plate	0.21	0.50
MEL19	Striped mug	<LOQ <sup>a</sup>	190

<b>Code</b>	<b>Product</b>	<b>Melamine (mg/dm<sup>2</sup> unless specified)</b>	<b>Formaldehyde (mg/dm<sup>2</sup> unless specified)</b>
MEL20	Lilac bowl	0.79	2.0
MEL21	White cup	0.30	1.0
MEL22	Elmer plate	0.20	0.59
MEL23	Anchor mug	0.36	0.91
MEL24	Taido mug and tray set	0.13	0.35
MEL25	Lemon tree plate	0.16	0.35
MEL26	Blue/white dessert bowl	3.8 mg/kg	12 mg/kg
MEL27	Flower mug	[<LOQ] <sup>a,b</sup>	[19] <sup>b</sup>
MEL28	Blue/white side plate	0.16	0.34
MEL29	Leaf tumbler	<LOQ <sup>a</sup>	140
MEL30	Small flower plate	0.32	0.72
MEL31	Appetiser tray	0.42	1.7
MEL32	White cup	0.49	1.2
MEL33	White bowl	0.097	0.73
MEL34	Mermaid bowl	0.31	0.77
MEL35	Marble green mug	0.17	0.87
MEL36	Blue/white bowl	0.051	0.36
MEL37	Tuscany tumbler	[<LOQ] <sup>a,c</sup>	[65] <sup>c</sup>
MEL38	Tuscany mug	0.20	0.90
MEL39	Azores mug	0.16	0.50
MEL40	3oz fluted white ramekin dish	0.19	0.84
MEL41	Animal design mug	0.13	0.47
MEL42	Thomas the Tank Engine divided plate	0.15	0.68
MEL43	Stripe mug	0.30	0.91
MEL44	Peter Rabbit cup	0.27	0.60

Code	Product	Melamine (mg/dm <sup>2</sup> unless specified)	Formaldehyde (mg/dm <sup>2</sup> unless specified)
MEL45	Teletubbies bowl	0.16	0.64
MEL46	Green beaker	[0.12] <sup>d</sup>	[0.66] <sup>d</sup>
MEL47	Patterned plate	0.17	0.48
MEL48	Butterfly and plant mug	0.054	0.33
MEL49	Stripe bowl	0.96 mg/kg	3.6 mg/kg
MEL50	Diagonal striped bowl	<LOQ <sup>a</sup>	340 mg/kg

a: LOQ varied with article depending on the simulant volume to contact area ratio. The worst case LOQ was 0.073 mg/dm<sup>2</sup> for the 6 samples with the results reported on an area basis. For MEL50 the LOQ was 0.060 mg/kg

b: 2 articles cracked during the 2<sup>nd</sup> exposure and the third during the 3<sup>rd</sup> exposure, therefore the result quoted is the mean of 4 tests obtained on the 1<sup>st</sup> and 2<sup>nd</sup> exposures. Pitting observed. Cracking believed to be associated with high levels of formaldehyde found.

c: One article cracked during the 3<sup>rd</sup> exposure therefore the result is the mean of 2 tests obtained on the 3<sup>rd</sup> exposure. Pitting observed. Cracking believed to be associated with high levels of formaldehyde found.

d: One article cracked on the 1<sup>st</sup> exposure therefore result quoted is the mean of 2 tests obtained on the 3<sup>rd</sup> exposure. No signs of pitting observed. Low levels of formaldehyde, believed that the cracked specimen was flawed.