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THE USE OF OVERALL MIGRATION METHODOLOGY TO TEST FOR FOOD CONTACT SUBSTANCES WITH SPECIFIC MIGRATION LIMITS

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ABSTRACT

This work investigated if overall migration (OM) test procedures could be used to test also for the migration of specific substances from plastics. The OM test procedure used was the evaporative gravimetric method used with volatile food simulants. Thirty (30) food contact substances (additives and monomers) were tested for their chemical stability and volatile loss during the heated evaporation stage of the OM procedure. 18 of the 30 were determined in acceptable yield. It is concluded that in the list of ca. 620 EU substances that have specific migration limit values of 5 mg/kg or higher, and based on considerations of stability and volatility, more than half could be amenable to control using OM methodology. This is particularly the case for inert plastics with low intrinsic overall migration values of oligomers. This means that, based on the OM test result found, testing laboratories could decide case-by-case if known additives and starting substances are covered by the OM result and no separate testing would be required for specific migration, with time and resource cost savings.

INTRODUCTION

EU legislation governing plastics materials and articles intended for food contact requires migration testing to demonstrate compliance [SCHAEFER 2007, VERAART and COULIER 2007]. The legislation specifies a limit on the total mass of substance permitted to migrate. This is called the overall migration limit (OML). The OML applies to all plastics and is 60 mg/kg of food or food simulant or 10 mg/dm^2 expressed on a contact area basis. The OML was established to ensure the inertness of plastics and prevent unacceptable adulteration of the food. In addition, chemicals used in the manufacture of plastics are in many cases assigned a specific migration limit (SML) if human exposure needs to be limited to ensure consumer protection. An SML is defined as the maximum concentration of a substance permitted to migrate to a food or to food simulating liquids. The food simulants used to test for OM and for SM are the same. For this work the simulants used were distilled water, 10% ethanol in water, 3% acetic acid in water, and the olive oil substitutes isooctane and 95% ethanol in water. The legal and technical situation on food simulants, alternative simulants, substitute food simulants etc is complex and is described in detail elsewhere [SCHAEFER 2007, VERAART and COULIER 2007].

Enforcement of the EU legislation is by migration testing. The test for migration using simulants as model foods has two steps [CASTLE 1996, 2007]. The first step is the exposure of the plastic to the food simulant(s) and during this step the migration of substances into the simulant occurs. The exposure conditions used are the same regardless of whether overall migration (OM), specific migration (SM), or both, is being tested for. These exposure conditions, the time, temperature and the nature of the simulant used, are related to the worst foreseeable conditions of use with actual foods [VERAART and COULIER 2007].

The second step of migration testing is the measurement of OM or SM. OM is a gravimetric determination of all chemical substances that migrate to the simulant. OM is determined simply by weighing either the residue after the simulant is evaporated (for volatile simulants only) or by weighing the plastic specimen before and after exposure to find its mass loss (for olive oil and related non-volatile fat simulants). In contrast, SM is measured using chemical tests which are, by definition, specific to the particular substance or group of substances under scrutiny. In principle - within the experimental error of each test and assuming that each and every specific substance is 'captured' by the OM procedure - the OM result is equal to the sum of all specific migrations.

Thus, for a plastic manufactured using several different monomers and additives, testing for compliance requires an OM test plus individual tests of the exposed simulant for all substances which have an SML. This can make testing for compliance costly and time consuming. In order to minimise the time and expense involved in testing; we have evaluated the advantages and limitations of using OM results to reduce the need for individual chemical analyses. This is potentially attractive because the OM test must be conducted in all cases regardless of what monomers or additives were used to make the plastic. Because the OM test is the cornerstone test and is always conducted, it would give efficiency savings if the OM test result could also be used to check for SM levels.

The approach is also attractive since it would automatically cover the situation where two or more related substances are given a so-called group SML [SCHAEFER 2007]. The present situation is that each substance in the group must be measured individually and the total group summed, or the group must be measured collectively using a moiety-specific method of test. Measurement via the OM result could offer considerable savings of time and money.

It can be anticipated from simple inspection of standard OM test protocols [CEN 1999] that not all substances will be measurable by this approach. The following factors need to be taken into account.

- Because the precision of OM measurements is relatively poor [CASTLE et al. 2004] the SML would have to be relatively high to be encompassed within the OM measurement.
- The OM from a plastic may be so high that it already exceeds many or all SM values, e.g. migration of oligomers or plasticisers could swamp migration of other additives.
- The substance in question must be relatively non-volatile or it would be lost in the evaporation procedure during the OM test. In this procedure [CEN 1999], samples are heated to evaporate the simulant and then dried to constant weight at 105°C.
- The chemical stability of the substance in the heated simulants is also important. Ideally it should be stable. However, if it reacts without significant change in mass, or reacts with a weight gain then there would not be a problem. However if a substance reacted with a weight loss (e.g. release of a volatile fragment) the test would then underestimate the true specific migration level.

This paper describes investigations into the suitability of 30 substances selected for study, for OM testing. Substances were spiked into simulants and subjected to the OM drying-down procedure for aqueous and volatile simulants. The substances which survived this procedure without loss through volatilisation or degradation were then spiked into a simulant solution of "migrate" from the relevant polymers and their recovery determined again.

MATERIALS

Test substances. Monomers and additives were obtained from the following sources. 11-Aminoundecanoic acid (99%), bisphenol A (99%), caprolactam (99%), didodecylthiodipropionate (99%), diethylene glycol (99%), diethylene glycol butyl ether (99%), 4,4'-dihydroxybenzophenone, dimethylethanolamine (99%), hexadecyltrimethyl ammonium bromide, diethyl phthalate, ethylene glycol (99%), hexamethylenetetramine (99%), maleic acid (99%), methyl salicylate (99%), oxalic acid (99%) and 2-phenylindole (95%) were all from Aldrich (Gillingham, Dorset). Melamine (99%), catechol (99%) dioctadecyldisulphide, and propyl gallate (98%) were from Lancaster synthesis. Butylated hydroxy anisole (BHA, 98%) and dioctylsulphosuccinate (99%) were from Sigma. Irganox 1076 and Irgafos P-EPQ were from Ciba-Geigy. Irganox 1520, Tinuvin 326 and Tinuvin 312 were obtained from the European additives reference collection [VAN LIEROP *et al*. 1998a, 1998b]. The remainder of the substances were available in the laboratory either from the monomer reference collection [BUSH *et al*. 1993] or from previous work.

Plastics. Polymers were available in pellet form in the laboratory from previous work [FORDHAM *et al*. 1995] except for the following: Nylon was from Dupont (UK) Ltd, polypropylene was from BASF; and PVdC was from Aldrich. The polymers were for laboratory use and were not necessarily commercial packaging-grade plastics.

Solvents. All solvents used including water were HPLC grade and were from Rathburn (Walkerburn, UK) with the exception of ethanol which was from Hayman Ltd (Essex).

METHODS

Measurement of recovery from simulants

A solution of each substance dissolved in simulant was subjected to a normal OM evaporation protocol [CEN 1999]. Thus, the substance (i.e. monomer or additive, 10 mg) was dissolved in a suitable simulant (10 ml). The small volume of 10 mL was chosen to represent the end stages of the evaporation of a larger volume (typically 100 to 200 mL) obtained from a migration test [CEN 1999]. The solution was transferred to a pre-weighed Pyrex glass dish (250 ml), placed on a hotplate and allowed to evaporate with close monitoring. When the simulant had almost completely evaporated, the dish was transferred to an oven at 105°C for 30 min to complete the drying process. In the case of isooctane, for safety reasons (fire) the solvent was evaporated under a stream of nitrogen at 50-60°C before oven drying. The dish was removed from the oven, placed in a desiccator, allowed to cool to ambient temperature and then weighed. The percentage recovery of the substance was determined from the exact weight used (ca. 10 mg) and the weight of the residue recovered after the evaporation protocol. All tests were performed in triplicate together with two procedural blanks (simulant alone, no added substance) to check for contamination or weighing errors.

Preparation of plastic overall migrate

The procedure for preparing plastic overall migrate into isooctane varied depending on the polymer type. The aim was to obtain a sufficient quantity of overall migrate for the spiking experiments and so the time, temperature, and mass:volume ratio exposure conditions used were simply for convenience. Isooctane was chosen rather than 95% ethanol (being another fat simulant) since it almost invariably gives higher extraction levels. As an example of the procedure used, for PE, PP and PVC, the polymer pellets (200-250 g) were weighed into a

Duran bottle and isooctane (400-500 ml) was added. The bottle was sealed and placed in a water bath at 40°C. The mixture was left for 10 days after which time portions of the extract were decanted into 36 ml vials. The solvent was removed by evaporation under nitrogen at 50-60°C. Details of extraction conditions for all the polymers are given in Table 1 along with the quantity of plastic migrate obtained.

Measurement of recovery from migrate solution

For each polymer type, the overall migrate was dissolved in the appropriate simulant to give a 1 mg/ml solution. Monomer or additive was then added to give a 1 mg/ml solution and then 10 mL portions of this solution were subjected to the evaporation procedure described above for the recovery experiments.

RESULTS AND DISCUSSION

Selection of the substances for the study

The selection of substances for study was made on the following basis:-

Value of the SML. Substances with an SML greater than 3 mg/kg were identified from the Community list of regulated substances. This is updated regularly [CEU 2008]. As discussed above, it was considered unlikely that substances with low SML values would be amenable to the approach proposed because of the rather imprecise nature of the OM test procedures. Other selection criteria were then applied to give the final list of substances tested. The selection-rejection criteria were as follows.

Boiling point of the substance. It was considered that substances with a boiling point of <180°C would volatilise under evaporation conditions and so they were generally excluded from the study except for 2 substances (Table 2) selected to test this assumption.

Commercial usage. The study placed emphasis on substances that find wide commercial usage [VAN LIEROP *et al*. 1998a, 1998b, 9]. and so for which migration testing is required

Chemical structures. Substances thought to be chemically unstable and likely to degrade on heating with a change of mass, were not studied. Further, if the candidate substances [CEU 2008] contained two or more essentially equivalent chemicals, being structurally similar and likely to exhibit similar behaviour, then only one of them was chosen to represent that chemical class.

Table 2 lists the 30 substances that were tested. They are listed in Table 2 using the nomenclature used in [CEU 2008]. They comprised 11 monomers and 19 additives. 12 of the 30 substances have SML(T) limits meaning that 2 or more substances are covered by the group (total) migration limit. Since the list of EU-authorised substances is updated regularly, substances are added and sometimes removed and SML values may change also. Three of the substances selected at the time this work was conducted, no longer appear in the EU list and they are indicated in Table 2. This has no influence of the general findings and conclusions drawn from this work.

Design of the tests

The work has focused on the volatile simulants for which the OM procedure calls for a simple evaporation step. With a large number of substances of interest and with several volatile simulants and several plastics available, the number of permutations possible was almost

endless. For this reason, the work was planned to cover a reasonable spread of combinations with sufficient coverage to allow general conclusions to be drawn.

No tests were conducted using the fat simulant olive oil or alternative non-volatile triglycerides [SCHAEFER 2007, VERAART and COULIER 2007]. This was for two reasons. First, because the test using olive oil is long, complicated and expensive and so it does not lend itself to testing the number of substances required for this work to have a general character. Second, and more importantly, the test result using olive oil simulant is subject to a large uncertainty (+/- 30% is expected in the standard test) and this would make interpretation of any findings also subject to a large uncertainty [CASTLE *et al*. 2004].

The evaporation procedure followed was that described in the last stages of CEN standards [CEN 1999]. It was of interest to check the maximum temperatures attained during the evaporation. To do this a thermocouple was attached to the inner surface of the base of an evaporating dish. Water simulant was added (50 or 10 ml), and the dish was heated on a hotplate in the normal way [CEN 1999] to evaporate the simulant. The final temperature reading was taken beyond the point at which the sample would normally be removed from the hotplate and placed in a 105ºC oven for final drying. The results are illustrated in Figure 1. It is evident that the timing of removing the sample is crucial, as the temperature rises sharply at this stage and thereafter there would be the risk that volatiles could be lost from the gravimetric assay or that the sample migrate may degrade by charring.

The tests were conducted by a trained analyst using CEN OM procedures [CEN 1999] that were UKAS accredited. All the AQA (analytical quality assurance) procedures described in the UKAS accredited procedure were used for this work. Further, the analyst had participated in all FAPAS® OM trials to date (since inception in 1994) [FAPAS 2008] with satisfactory

performance (Z<2) in all. It is sure therefore that the tests were conducted correctly and that the findings reported here are reliable.

Substance recovery from spiked simulants

The results of recovery experiments using spiked simulants evaporated down, are given in Table 3. Of the 30 substances tested, a total of 12 suffered significant loss. The acceptable limits were set at 70% recovery, i.e. no more than 30% loss. Seven of these twelve substances lost were monomers with a low boiling point and the remaining five were additives. Three substances merit individual discussion because simple volatility was not the sole factor in the outcome of the recovery tests.

11-Aminoundecanoic acid gave a high recovery of around 120%. This was most likely due to salt formation of $[HOOC(CH_2)_{10}NH_3]^+$ $[OOCCH_3]^-$ between the amine moiety and the acetic acid simulant. The higher molecular weight of this salt (261 as opposed to 201) accounts for the high recovery.

Maleic acid showed a partial loss in simulants with lower recovery from ethanol than from water. It is known that maleic acid converts to fumaric acid at 138°C; and fumaric acid sublimes at 200°C. There is also the possibility that the volatile ethyl ester of maleic acid formed in the ethanol simulant and this could also be a reason for the higher loss from this simulant.

Oxalic acid showed almost total loss even though it has a melting point of 190°C. It sublimes however at around 157°C and can decompose into carbon dioxide, carbon monoxide, formic acid and water.

In addition to the volatile monomers that gave a low recovery, four additives were lost due to volatilisation. These were butylated hydroxyanisole; diethylphthalate, methyl salicylate and diethylene glycol monobutyl ether, which have boiling points of 264, 298, 222 and 230°C respectively.

Some additives showed a partial loss. Irgafos P-EPQ showed losses of around 60% and monooctyltin-tris-2-ethylhexyl mercaptoacetate showed around 50% loss. It is known from previous work [JICKELLS 1998, JICKELLS *et al.* 1994] that these additives are generally stable in simulants at room temperature (the organotin compounds are only unstable in aqueous simulants) so it must be presumed that they are degraded at the higher temperature used for the evaporation process.

In general, substances of boiling point less than ca. 250-300°C did not survive the evaporation procedure. Two exceptions were ricinoleic acid (bp. 245°C) and phenylindole (bp. 250°C) which were recovered at 94% and 74% respectively. Clearly the boiling point of a substance is not a full description and the vapour pressure - temperature relationship would indicate better the possibility of volatilisation below the boiling point temperature.

Recovery from evaporated simulants with plastics coextractives present

Thirteen of the substances which had showed an acceptable recovery from spiked simulants, were then tested in the presence plastics migrate (1 mg/ml) to determine if the presence of other plastics coextractives would have any effect. Table 1 lists the plastic migrate samples obtained. The choice of which plastic migrate to combine with each substance was based largely on the polymer type that the monomer or additive would normally be used in. Table 3 shows the testing programme which was carried out.

It was first necessary to ensure that no plastic migrate was lost during the evaporation procedure. This was thought unlikely, since the migrate had itself been obtained by evaporation of an extract of plastics, but the check was done for completeness. The recovery was quantitative with a mean recovery for all plastics of 98%.

It can be seen from the results in Table 3 that the presence of plastic co-extractives gave rise to uniformly higher recovery levels for the monomer or additive under study. It is clear therefore that the extractives do not have any undue influence on the stability of the substances. Rather, the higher recovery can be attributed to a 'keeper' effect during the evaporation step. The co-presence of the plastic migrate (largely oligomers, except for PVC and PVdC where the migrate would be largely plasticiser) and the test chemical (monomer or additive) reduced the chemical activity of the substance in the residue. This in turn reduces the vapour pressure of the substance (compared to the vapour pressure for the pure substance when tested in the absence of the plastic extractives) and the lowered vapour pressure reduces volatilisation losses.

Consideration of reduction factors

If the intended food application is known or for specified lipophillic substances, the simulant D reduction factor (DRF) or the fat consumption factor (FCF) may be applicable [SCHAEFER 2007, VERAART and COULIER 2007]. These two factors increase the level of interest (i.e. the SML) into food simulants (DRF) or into foods (FRF) by up to 5-fold. Obviously this can be a considerable advantage if using OM methods to test for SM since it effectively increases the sensitivity by up to 5-fold.

Consideration of alternative or additional (emerging) food simulants

This work has used the non-fatty food simulants water, 3% acetic acid and 10% ethanol along with the fatty food substitute simulants 95% ethanol and isooctane. It is possible that the list of available EU food simulants may be refined or may even undergo a complete root-and-branch revision [FOODMIGROSURE 2008]. Since this would most likely include additional volatile food simulants and perhaps even a phasing-out of the non-volatile triglyceride fat simulants (olive oil, sunflower oil, etc) the existing OM methodology would still be appropriate and the findings made here would continue to be applicable.

CONCLUSIONS

This study has concluded that testing for SM by using OM methods is most applicable for polymers with a low intrinsic migration. For polymers with higher intrinsic migration, the approach is only suitable for substances with high SMLs.

In terms of suitability of individual substances, it was found that in general, substances of boiling point less than 250°C were not covered as they are lost wholly or partly due to volatilisation. However there were exceptions to this rule and the chemical nature (e.g. stability to degradation, isomerisation, salt formation) of the substance should be taken into account when assessing volatility. The presence of co-extractives from the plastic helps to keep recovery losses low, but too much co-extractives would dominate any OM result and so make the approach of testing for SM using the OM value less useful.

The precision of the gravimetric procedure [CEN 1999] is +/- 0.5 mg which corresponds to +/- 0.5 mg/dm^2 or +/- 3 mg/kg migration [CEN 1999]. In the Commission list of regulated substances [CEU 2008] up to and including the 5th amendment to the Plastics Directive there

are approximately 434 substances without a SML specified (and therefore effectively subject to the limit value of 60 mg/kg as the OML) and a further 188 substances with a numerical SML specified of 5 mg/kg or higher. This list is updated regularly and the exact numbers used here are not important. The 5 mg/kg limit is an important cut-off value in the tiered toxicological evaluation of migrating substances in the EU [SCHAEFER 2007, CASTLE 2007]. Based on stability and volatility considerations (full data not shown) it can be anticipated that more than half of these substances could be evaluated using OM methods - particularly for plastics with a low intrinsic overall migration (e.g. low oligomer release) and for foods/substances for which DRF and FRF factors come into play. This means that, based on the OM test result found, testing laboratories could decide case-by-case if known additives and starting substances are covered by the OM result and no separate testing would be required for specific migration, with time and resource cost savings.

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Table 1. Amount of migrate obtained from isooctane extraction of polymers

Polymer	Exposure time (days)	Exposure temp (C)	Migrate obtained (g/kg polymer)
ABS (poly-acrylonitrile-butadiene-styrene)	36	40	0.080
PA (polyamide)	19	60	0.096
PE (polyethylene)	10	40	6.0
PP (polypropylene)	10	40	4.5
PS (polystyrene)	19	60	0.46
PVC (polyvinylchloride)	10	40	210
PVdC (polyvinylidene chloride)	10	40	14

Table 2. Substances selected for testing

Monomers	Other names	SML mg/kg	Bp ºC
11-Aminoundecanoic acid		5	Mp 188
Bisphenol A	BPA	3	Mp 150
Caprolactam		15	267
Diethylene glycol	DEG	30 (T)	244
1,2-Dihydroxybenzene	Catechol	6	245
4,4'-Dihydroxybenzophenone		6 (T)	350
Dimethylaminoethanol	Dimethylethanolamine	18	133
Ethylene glycol	MEG	30 (T)	197
Hexamethylene tetramine	HMTA	15 (T) (as HCHO)	280
Maleic acid		30 (T)	138 (dec)
2,4,6-Triamino-s-triazine	Melamine	30	Mp 345

Additives	Other names	SML mg/kg	Bp ºC
2,4-Bis-(octylthiomethyl)-6-methylphenol	Irganox 1520	5 (T)	532 (est)
tert-Butyl-4-hydroxyanisole	BHA	30	264
Diethylene glycol monobutyl ether *		3	230
Dioctadecyl disulphide		3	>600
2-Ethoxy-2'-ethyloxanilide	Tinuvin 312	30	>400
Gallic acid, propyl ester	Propyl gallate	30 (T)	Mp 150
Hexadecyltrimethyl ammonium bromide	CTAB	6	Mp 218
2-(2-Hydroxy-3-*tert*-butyl-5-methyl-phenyl)-5-chlorobenzotriazole	Tinuvin 326	30 (T)	Mp 147
Mono-n-octyltin tris (2-ethylhexylmercaptoacetate)		1.2 (T) (as Sn)	Not found
(Octadecyl 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate	Irganox 1076	6	Mp 50
Oxalic acid		6 (T)	Mp 190
2-Phenylindole		15	250
Phthalic acid, diethyl ester *	Diethyl phthalate	12	298
Ricinoleic acid		42	245
Salicylic acid, methyl ester	methyl salicylate	30	222
Stearic acid, esters with ethylene glycol		30 (T)	>400
Sulphosuccinic acid dioctyl ester, Na salt *		none	360

Tetrakis (2,4-di-*tert*-butylphenyl)-4,4'-biphenylene diphosphonite	Irgafos P-EPQ	18	Mp 95
Thiodipropionic acid didodecyl ester	Didodecylthio dipropionate	5 (T)	Not found

* these 3 additives are no longer listed the most recent revision of [CEU 2008]. See text.

(T) – denotes where two or more related substances are covered by a (Total) specific migration limit.

Table 3. Recovery of substances subjected to an OM evaporation procedure

Substance	Simulant [a]	% Recovery [b]
Monomers		
11-Aminoundecanoic acid	3% acetic	126 ± 5 [c]
	3% acetic + PA	121 ± 2 [c]
Bisphenol A	95% EtOH	89 ± 4
Caprolactam	H_2O	0
	95% EtOH	10 ± 3
Diethylene glycol	H_2O	0
	95% EtOH	6 ± 7
1,2-Dihydroxybenzene (catechol)	H_2O	0
	95% EtOH	8 ± 7
4,4'-Dihydroxy- benzophenone	95% EtOH	92 ± 2
Dimethylaminoethanol (dimethylethanolamine)	H_2O	0
	95% EtOH	5 ± 6
	Isooctane	2 ± 3 (n=6)
Ethylene glycol (monoethylene gylcol)	H_2O	0
	95% EtOH	3 ± 3
Hexamethylene tetramine	H_2O	14 ± 25
	95% EtOH	4 ± 4
Maleic acid	H_2O	72 ± 15
	95% EtOH	41 ± 10
2,4,6-Triamino-s-triazine (melamine)	3% acetic acid	76 ± 5
Additives		
2,4-Bis-(octylthiomethyl)-6-methylphenol (Irganox 1520)	Isooctane	91 ± 2
	95% EtOH	96 ± 5 (n=6)
	Isooctane + PE	101 ± 2
	Isooctane + PP	65 ± 1
tert-Butyl-4-hydroxyanisole (BHA)	95% EtOH	14 ± 11
Diethyleneglycol monobutylether	H_2O	2 ± 1
	95% EtOH	4 ± 3
	Isooctane	7 ± 5

for footnotes, see Table end

Table 3 (cont.). Recovery of substances subjected to an OM evaporation procedure

Substance	Simulant [a]	% Recovery [b]
Dioctadecyl disulphide	Isooctane	102 ± 3
	Isooctane + PP	92± 7
2-Ethoxy-2'-ethyloxanilide (Tinuvin 312)	95% EtOH	95 ± 3
	95% EtOH + PVC	104 ± 7
	95% EtOH + PA	104 ± 3
Gallic acid, propyl ester (propyl gallate)	95% EtOH	94 ± 3
Hexadecyltrimethyl ammonium bromide (CTAB)	H_2O	116 ± 15
	95% EtOH	106 ± 3
	95% EtOH + PVC	101 ± 0
2-(2-Hydroxy-3-*tert*-butyl-5-methyl-phenyl)-5-chlorobenzotriazole (Tinuvin 326)	Isooctane	108 ± 7
	Isooctane + PVC	101 ± 5
	Isooctane + PE	90 ± 3
	Isooctane + PP	89 ± 8
	Isooctane + PS	99 ± 5
Monooctyltin tris-2-ethylhexyl mercaptoactate	Isooctane	56 ± 1
	95% EtOH	52 ± 1
	Isooctane + PVC	82 ± 3
	95% EtOH + PVC	75 ± 3
Octadecyl 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate (Irganox 1076)	Isooctane	98 ± 4
	Isooctane + PVC	99 ± 3
	Isooctane + PE	100 ± 3
	Isooctane + PP	97 ± 6
	Isooctane + PS	108 ± 7
Oxalic acid	H_2O	9 ± 4
	95% EtOH	3 ± 3
2-Phenylindole	95% EtOH	74 ± 11
	95% EtOH + PVC	95 ± 0
Phthalic acid, diethyl ester (diethylphthalate)	Isooctane	6 ± 6 (n=9)
	95% EtOH	4 ± 4
Ricinoleic acid	95% EtOH	94 ± 5

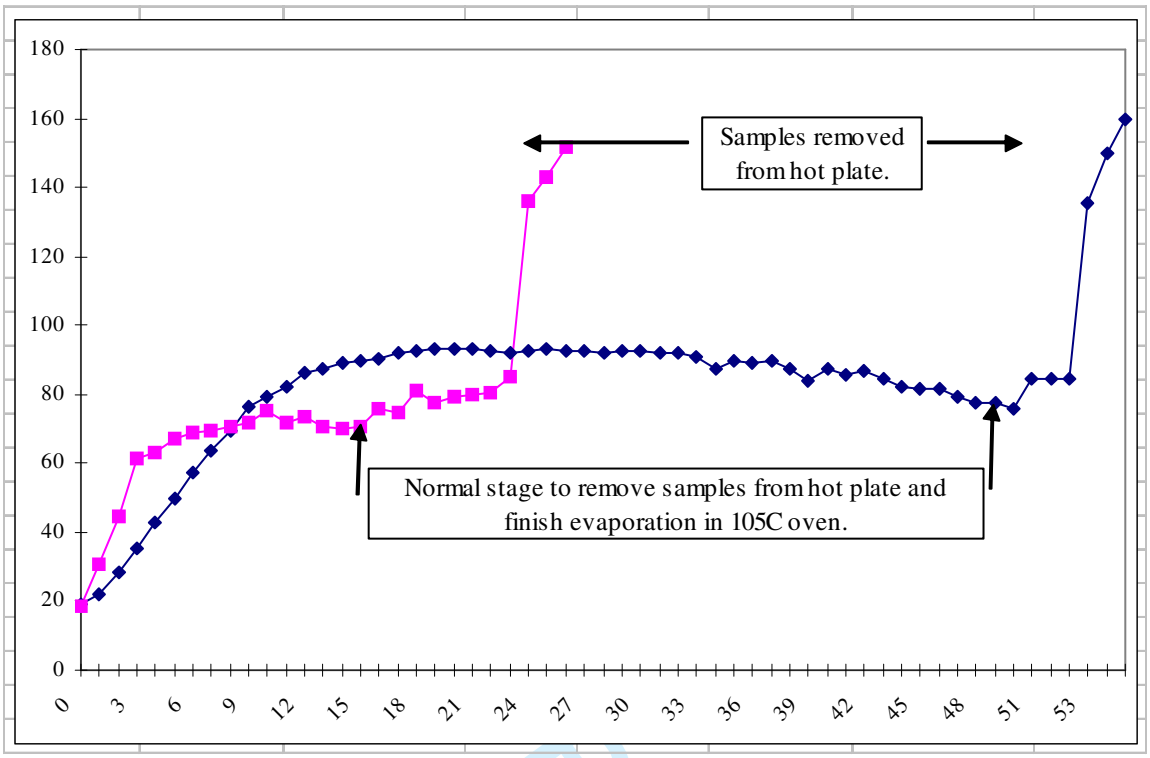
for footnotes, see Table end

Table 3 (cont.). Recovery of substances subjected to an OM evaporation procedure

Substance	Simulant [a]	% Recovery [b]
Salicylic acid, methyl ester (methyl salicyclate)	Isooctane	1 ± 1
	95% EtOH	10 ± 9 (n=6)
Stearic acid, esters with ethylene glycol	Isooctane + PE	96 ± 1
	Isooctane + PP	93 ± 2
	Isooctane + PS	97 ± 1
	Isooctane + PVdC	97 ± 2
	Isooctane + ABS	92 ± 0
Sulphosuccinic acid, dioctyl ester Na salt (dioctylsulphosuccinate)	Isooctane	96 ± 3
	95% EtOH	102 ± 2 (n=6)
	Isooctane + PVC	96 ± 1
Tetrakis (2,4-di-*tert*-butylphenyl)-4,4'-biphenylene diphosphonite (Irgafos P-EPQ)	Isooctane	41 ± 5
	95% EtOH	38 ± 1
	Isooctane + PE	78 ± 1
	Isooctane + PP	69 ± 7
	Isooctane + PS	77 ± 1
Thiodipropionic acid, didodecyl ester (didodecylthiodipropionate)	Isooctane	91 ± 4
	Isooctane + PP	93 ± 5
	Isooctane + PS	97 ± 1

[a] Simulant used, with/without co-extractives from the polymer indicated.
[b] Tests in triplicate unless stated otherwise.
[c] This recovery greater than 100% is attributed to salt formation from the amine.

Fig 1 Temperature profile of water simulant samples subjected to an OM evaporation procedure, with extended heating past dryness



x-axis = time in minutes for the evaporation procedure

y-axis = temperature in ºC of the inner surface of the base of the evaporation dish (placed on a hotplate)

Squares = 10 ml volume tested

Diamonds = 50 ml volume tested

Note. The evaporation dishes would normally be taken off the hotplate just before going completely to dryness. This point is marked on the figure. In this test, the samples were deliberately left past this point to show the effect of leaving too long. The temperature runs away once the cooling effect of evaporation is lost as the sample dries completely. Clearly, at these higher temperatures there is the risk of loosing the migrate by reaction (charring) or volatilisation.