



## 4-Nonylphenol (NP) in Food Contact Materials: Review, Analytical Methodology and Occurrence

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**4-Nonylphenol (NP) in Food Contact Materials: Analytical Methodology and Occurrence**

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## Abstract

Nonylphenol (NP) is a recognised environmental contaminant but it is unclear whether its occurrence in food arises only through environmental pathways or also during the processing or packaging of food, as there are reports that indicate that materials in contact with food such as rubber products and polyvinylchloride (PVC) wraps can contain NP. A review of the literature has highlighted the scarcity of robust analytical methodology or data on the occurrence of NP in packaging materials. This paper describes methodology for the determination of NP in a variety of packaging materials which includes plastics, paper and rubber. The method uses either Soxhlet extraction or dissolution followed by solvent extraction (depending on the material type), followed by purification using adsorption chromatography. Procedures were internally standardised using <sup>13</sup>C carbon-labelled NP and the analytes were measured by GC/MS. The method has been validated and data relating to quality parameters such as limits of detection (LODs), recovery, precision and linearity of measurement are provided. Analysis of a range of 25 food contact materials found NP at concentrations of 64-287 µg/g in some polystyrene and PVC samples. Far lower concentrations (<0.03 - 1.4 µg/g) were detected in the other materials. It is possible that occurrence at the higher levels has the potential for migration to food.

**Key Words** – 4-nonylphenol, food packaging, materials, GC-MS, migration, APEOs, TNPP

**Introduction**

Nonylphenol (NP) is a term commonly used to describe a complex commercial mixture of mainly nonyl-substituted phenol. The complexity arises from the production of the compound from commercial nonene that is used to alkylate the phenol. Commercial nonene is a complex mixture of C9 branched chain alkenes and the resultant product is predominantly 4-nonylphenol (>90%, because alkylation favours the *para*- position) with varying amounts of 2-nonylphenol (5% in the purer grades), and smaller amounts of dinonylphenol and decylphenol (Kirk 1992, KEMI 1994). Most NP produced commercially is thus 4-nonylphenol with varied alkyl chain branching.

NP is a recognised environmental contaminant, occurring principally as a hydrolytic breakdown product of the most widely used non-ionic surfactants - the alkylphenol ethoxylates (APEOs) (Thiele et al 1997). Recently, there have also been reports on the occurrence of NP in food (Fernandes et al 2003, Nemoto et al 2000, Guenther et al 2002) and although APEOs are used as dispersing or stabilising agents in food packaging plastics, rubber and papers, it is less clear whether NP in food might also derive from the breakdown of the compounds used in this way. Another potential source of NP in food may be hydrolysis of the antioxidant tris(nonylphenyl)phosphite (TNPP) which is used as a heat stabiliser in the manufacture of a range of polymeric food packaging materials such as, styrenes, vinyl polymers, rubber polyolefins, etc. TNPP is a phosphite stabilizer used to maintain the colour stability, processing stability and performance integrity of the polymers in which it is incorporated (TNPP product description). NP is used as a starting material in the manufacture of TNPP and so it may be present as an impurity as well as a hydrolysis product, formed either within the food or after gastric digestion.

NP has been shown to have biological effects in a number of animal studies. Reviewed data shows that the lowest aquatic toxicity (LC50) is in the range of 0.0033 mg/L to 0.41mg/L (freshwater algae) and 0.0074 mg/L to 0.31 mg/L (fresh and salt-water fish) (EU Risk Assessment on NP). However, it is concern about the estrogenic activity of these compounds that has resulted in many studies on endocrine disruption. The first observations were made in 1991 when a medical researcher observed a proliferation of breast cancer cells stored in plastic containers (Soto et al 1991, Renner

1997). This behaviour is normally observed only in the presence of an oestrogen. Subsequent investigative work showed that the proliferation was caused by NP. It has also been shown in laboratory studies that the no-observed effect concentration of NP for vitellogenin (a fish egg protein normally produced in large amounts by female fish) production in male rainbow trout is approximately 5 µg/L with inhibition of testicular growth at concentrations greater than 30 µg/L (Jobling et al 1996). Similar studies (Lye et al 1999, Gimeno et al 1996, Gray and Metcalfe 1997) have confirmed that alkylphenols are capable of inducing vitellogenin production and inhibiting testicular growth in a number of fish species. In rats, a single 50 ppm dose was seen to elicit a uterotrophic response (Lee et al 1996, CSTEE opinion 2000), and increased uterine vascular permeability was observed in ovariectomized mice at 70 mg/kg bodyweight (Milligan et al 1986, CSTEE opinion 2000). Similar effects such as increased uterine weight and acceleration in vaginal openings at 30-100 mg/kg bw/day have also been reported (Chapin et al 1998). Within the European Union, the risk assessment for alkylphenols and their ethoxylates is covered under the EU existing Substances Regulation (EEC 793/93). Based upon the Draft Risk Assessment Report (Draft EU Risk Assessment on NP), a specific opinion was issued on the evaluation and control of these chemicals (CSTEE 2000, 2001). EC Directive 2003/53/EC came into force on 17 January 2005 and significantly limited the use of nonylphenol/nonylphenol ethoxylates. In addition, the CSTEE opinion agreed with the view that there were low concerns for carcinogenicity arising from these compounds; rather, the focus of concern was interaction with the endocrine system.

The aim of this study was the development of methodology for the measurement of 4-NP in order to investigate occurrence in a range of commonly used food contact materials. The first stage of this process was the identification of suitable materials. It is possible that some of these materials could contain NP as a result of the presence of NP ethoxylate additives or in particular, TNPP. There have recently been reports (Ozaki and Baba 2003, TNO 2003, Inoue et al 2001, Noriko 1999, Nemoto et al 2000, Kawamura 2000, Loyo-Rosales et al 2004) of positive identification and quantification of NP in some materials either directly or as a result of migration studies. The results from these reports on the measurements of NP in materials that may come in contact with food are summarised in Table 1. However, it is difficult to anticipate exactly which materials may contain nonylphenols. For example, in a

Japanese study (Kawamura 2000) high levels of NP were found in a plasticised PVC cling film, even though PVC itself does not require an antioxidant stabiliser. The presence was attributed to the possible use of tris(nonylphenyl)phosphite to stabilise the plasticisers used. Similarly, NP was found in high-impact polystyrene (HIPS) (Kawamura 2000) but it was thought to be present from the use of alkylphenol ethoxylate detergents in the suspension-polymerisation of the butadiene rubber component of the HIPS. Consequently, for method development purposes, a wide range of food contact materials were considered.

Insert Table 1 here

**Selection of food-contact materials**

The range of materials used for food contact is wide. The following 10 types of materials are in general use and have the potential to contain NP:

- Polyolefins and co-polymers
- Polystyrene and co-polymers
- Polyesters and co-polymers
- Chlorinated polymers and co-polymers
- Polyamides
- Polycarbonate
- Cellulose and cellulose esters
- Thermoset phenolic polymers
- Paper
- Rubber products

The choice of materials for the development of the analytical methodology was based on the following criteria:

- Widespread current usage as food contact material.
- Potential to contain NP either because of the suspected presence of tris(nonylphenyl) phosphite or nonylphenol ethoxylates (NPEO).
- Availability of unused (uncontaminated by food) material.

On the basis of these criteria, 20 sub-types of the above listed materials were obtained. For 5 of the 20 sub-types another example was obtained (the same material sub-type obtained at a different time and from different sources) in order to

investigate variation. Of the material types listed above the first four polymeric groups cover the most common contact materials and therefore made up the majority of the sample set. Additionally there have been findings of NP in PVC (Inoue et al 2001, Noriko 1999) and in rubber products (Ozaki and Baba 2003). In a few cases it was not possible to obtain unused material and so external portions of the packaging that were not in contact with the food were used.

### Materials and methods

The identity of the materials obtained was confirmed either by the embossed recycling code or, where this was absent, by Fourier Transform-Infra Red spectroscopy of the material.

A procedure using internal standardisation was used for the analysis. NP was isolated from the material matrix either by sample dissolution (polystyrene and polyvinyl chloride) in cyclohexane or by Soxhlet extraction using methanol (all other materials). The extracts were purified by open column chromatography using deactivated neutral alumina. The purified extracts were concentrated and analysed by gas chromatography-mass spectrometry. A schematic representation of the methodology is given in Figure 1.

### Reagents and standards

4-Nonylphenol technical mixture was obtained from ChemService, Pa. USA,  $^{13}\text{C}_6$  labelled 4-n-NP (purity - 99%) and  $^{13}\text{C}_{12}$  labelled PCB 52 were from Cambridge Isotope Laboratories Mass, USA. The labelled standards were used as the internal standard and internal sensitivity standard, respectively. Methanol, cyclohexane, dichloromethane and petroleum ether (boiling range 40-60 °C), were from Rathburn Chemicals Ltd, Scotland, UK. Other reagents included alumina (Brockmann Grade 1, Fluka) sulphuric acid (98 %) and anhydrous sodium sulphate, from Fisher Chemicals Ltd.

### Method

Samples were reduced to around 1-3 mm particles by cutting, using dichloromethane-rinsed scissors or metal cutters. With the exception of PVC and polystyrene



(described below) the materials were extracted as follows. Approximately 2 g of sample was fortified with 50 µl of internal standard,  $^{13}\text{C}_6$  4-n-NP (1 ng/µl) and Soxhlet extracted for ~ 20 h in methanol. Prior to extraction the soxhlet thimbles used were similarly extracted in order to minimise any NP contribution from the thimble material. Method blanks were included with every analytical batch. The extracts were concentrated to around 50 ml using a Turbo Vap II<sup>TM</sup> (Zymark Corporation) apparatus. The extracts were acidified with four drops of concentrated sulphuric acid and partitioned into 65ml of cyclohexane by shaking for 3 min. The partitioning was repeated a further two times and the cyclohexane fractions were combined and concentrated to ~0.5 ml.

PVC film and polystyrene foam and film samples (~ 0.1 – 0.5 g) were extracted ultrasonically for 30 min at 40<sup>0</sup>C using 20 ml of cyclohexane giving either partial or complete dissolution. The extracts were partitioned against 25 ml of acidified methanol and the partitioning repeated two times. The combined methanol extract was partitioned into cyclohexane as described above, and concentrated to ~0.5 ml.

The crude extracts were purified chromatographically using alumina that was deactivated with 15 % (w/w) water after heating in a muffle furnace at 450 <sup>0</sup>C for a minimum of four hours. Glass columns (300 x 8mm pre- soaked in methanol for 3-4 hours and dried) were packed with 5.6 g of this reagent and topped with a 5 mm layer of anhydrous sodium sulphate. The columns were conditioned with 10 ml of cyclohexane. The concentrated extracts were eluted successively with 50 ml petroleum ether (discarded) followed by 50 ml dichloromethane. The dichloromethane fraction was evaporated down to a final volume of approximately 100 µl, with addition of the internal sensitivity standard (25 µl of  $^{13}\text{C}$  PCB 52).

Insert Figure 1 here

The extract was analysed by GC-MS carried out in selected ion monitoring mode (SIM), using a 0.22 mm x 60m DB-5 (5% phenyl methyl polysiloxane, 0.25µ) column. A full description of the GC-MS measurement procedure has been reported (Fernandes et al, 2003). In brief, 2 µl injections of the sample extracts were made using a Programmed Temperature Vaporisation (PTV) injector. The GC oven was

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3 programmed from 60 °C for 3 min to 160 °C at 25 °C /min for 2 min, then 0.8 °C /min  
4 to 180 °C, then 30 °C /min to 300 °C for 5 min. The mass spectrometer was operated  
5 in SIM mode and a number of fragment and molecular ions corresponding to NP, <sup>13</sup>C  
6 NP and <sup>13</sup>C PCB 52 were monitored in the same group.  
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## 10 11 **Results and Discussion**

### 12 13 *Methodology considerations*

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15 Two specific stages of the analytical process were considered for development - the  
16 analyte isolation or extraction stage, and the extract purification stage. The  
17 measurement stage by GC-MS used the same procedures developed earlier for the  
18 determination of NP in food (Fernandes et al, 2003). The methodology developed  
19 here uses internal standardisation with <sup>13</sup>C labelled 4-n-NP to validate the  
20 measurement of each individual sample analysis.  
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28 Samples were size reduced by cutting, using scissors or metal cutters in order to  
29 minimise the possibility of degradation of either the materials or the analytes by the  
30 use of electric grinding equipment or filing that could raise the temperature of the  
31 material. The reduction in particle size also resulted in maximisation of the surface  
32 area available for extraction. This, combined with the use of methanol and the higher  
33 temperature (65°C) resulting from the Soxhlet extraction promoted effective  
34 extraction. The use of methanol for NP extraction due to the strong solubility of the  
35 compounds in this solvent has been reported in the literature (Thiele et al 1997,  
36 Fernandes et al 2003, Marcomini and Giger 1987) and it was therefore used for the  
37 extraction of most of the contact materials.  
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48 Polystyrene and PVC gave high levels of co-extractives which interfered with the  
49 measurement stage. They were therefore extracted ultrasonically using cyclohexane in  
50 which they dissolved either completely (PS, HIPS) or partially (PVC). Smaller  
51 amounts of sample were taken for analysis (~2 g) because the detection limit of the  
52 GC-MS method used for identification of the NP was sufficiently low. More  
53 importantly however, the large amount of co-extractives such as plasticizers, from  
54 bigger sample sizes have the potential to modify the efficacy of the later purification  
55 stages. For sample types where dissolution or partial dissolution of the sample was  
56 observed (expanded EPS, HIPS and PVC samples) it was necessary to take a yet  
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smaller sample size (0.1 – 0.5 g), in part due to the level of co-extractives observed, and in part due to the levels of NP detected in some of these samples.

The concentrated extracts were purified prior to GC-MS analysis because the potential chemical background arising from co-extractives could obscure or interfere with analyte signals. Deactivated alumina has been used successfully in the past (Lye et al 1999, Fernandes et al 2003, Van Ry et al 2000) for purification of food and environmental samples. However methodology that has been used to investigate levels of NP in packaging material has generally not used (or used minimal) further purification, probably because the LODs for these methods has generally been higher – of the order of  $\mu\text{g/g}$  levels, rather than the sub- $\mu\text{g/g}$  levels achieved in this work. As observed in previous work on food matrices (Fernandes et al 2003), good recoveries were observed using alumina deactivated to 15%, compared to other adsorbents, with generally low level of chemical interference from co-extractives. (Lye et al, 1999 and Fernandes et al, 2003)

The basic principle of the extraction and purification procedure described here is similar to other procedures described in the literature for the analysis of NP and NPEOs (Lye et al 1999, Fernandes et al 2003, Ozen and Floros 2001) where good recoveries were obtained for both NP and NPEOs, demonstrating the stability of these analytes to the extraction and purification techniques used. Recoveries for NP using this procedure are described in the following section.

The GC-MS method has been described in detail (Fernandes et al, 2003). A number of ions corresponding to major mass fragments of the analytes were monitored for quantification or confirmation purposes. These ions correspond to cleavage of the branched nonyl chain and depending on the degree of branching, different ions are observed, e.g.  $m/z$  135 corresponds to the loss of the  $\text{C}_6\text{H}_{13}$  group,  $m/z$  149 to loss of  $\text{C}_5\text{H}_{11}$  group, etc. Quantification was based on the molecular mass ions  $m/z$  220 for NP, and  $m/z$  226 for the  $^{13}\text{C}$  Carbon labelled internal standard. Although these ions give a lower response than some of the fragment ions, the higher mass is more selective and thus less prone to interference. Ions corresponding to  $^{13}\text{C}$  labelled PCB52 -  $m/z$  302 and  $m/z$  304 as internal sensitivity standard were also monitored in order to estimate analytical recovery.

### *Method validation*

A number of steps were taken to ensure validity of the developed methodology. The results of these are summarised in Table 2.

The analytical methodology described here has considerable potential to be compromised due to NP background levels, as the chemical may be commonly used as a stabiliser in standard laboratory equipment such as tubing, plastic shelving, detergents, etc. Careful selection of solvents and conditioning of the reagents together with scrupulously clean glassware has resulted in low blank levels (0.01 – 0.02 µg/g). The average LOD (defined as mean + 3 times SD of blank determinations) thus achieved was 0.03 µg/g.

The linearity of NP response to the SIM GC-MS conditions was investigated particularly with respect to the ions used for quantification (molecular ions for NP). Measurements covering a practical range from ~0.01 µg/g – 1000 µg/g showed a linear GC-MS response (regression analysis value of 0.9995) for NP normalised to the <sup>13</sup>Carbon labelled internal standard.

Analytical recovery was assessed for each sample as a measure of the efficacy of the methodology used and gives an indication of the losses incurred during the extraction, purification and measurement stages. The recovery calculated relative to the internal sensitivity standard (<sup>13</sup>Carbon labelled PCB 52) gave an average value of ~75% and typically ranged from ~ 50 to 100% (Table 3). As the methodology uses internal standardisation, concentration values reported for the materials are corrected for recovery. Analyte recovery from the sample matrix was confirmed by analysing a fortified (with native NP) sample. PET was chosen as the matrix as preliminary investigations showed no detectable levels of NP. The measurement showed that 94 % of the added NP was recovered and also confirmed that the analyte was not degraded during the extraction and purification process.

A number of replicate sub-samples of PVC and HIPS were analysed to investigate the reproducibility of the method. The two sets of analyses were separated in time by a few weeks. The precision (% CV) of the analysis was ~8% for the HIPS and ~10% for the PVC. The % CV was also estimated from the individual recovery measurements

for the replicates and these were 12% and 13% respectively. Although the latter set of figures (recoveries) are based on external calibration they nonetheless reflect the precision of measurement of replicates of the same sample measured at the same time. This approach was therefore used to estimate precision as %CV across the whole range of materials from recovery data for each of the individual materials. This value was estimated to be of the order of 24%. As this was an externally calibrated measurement the %CV for the internally standardised method would be expected to be lower than this value (as observed in the case of HIPS and PVC).

Precision was also investigated by analysing 4 materials in duplicate. As the replicate analyses described above were based on materials where higher concentrations were detected, materials that showed lower concentrations (PP, HDPE, PC and latex) were investigated. Good agreement was observed between duplicates with differences ranging from 0 – 16%.

**Analytical Results**

The results are given in Table 3 and have been corrected for the blank contribution. As the measurement was internally standardised the concentrations are inherently corrected for recovery losses. The table also shows data for the 5 duplicate sub-types (these were the same material sub-types but obtained at a different time and from different sources).

The levels detected in the 20 material sub-types ranged from <0.03 - 287 µg/g with the majority of samples (84%) showing sub-µg/g concentrations. The materials therefore fall into 2 broad categories – those in which NP was detected at high µg/g levels (64 – 287µg/g) and those in which NP was either not detected or was detected at around or below the 1 µg/g level. A study of the duplicate sub-types shows large variation between the levels of NP detected. This is particularly true for the PS butadiene co-polymer, where the ice-tray sample contained 64µg/g compared to the filter coffee holder where NP was not detected. This may indicate the use of different ingredients or different processes in the production of the same material sub-type.

There are a limited number of reports in the literature that discuss concentrations or identification of NP in similar materials to those investigated here and a comparison with Table 1 shows that the results of these studies accord well with those presented here, particularly in the types of material that show the presence of NP. There are also similarities between the concentrations detected in this study and those detailed in Table 1, although the differences in the units used for reporting concentrations (concentration/area) make comparison difficult in some cases. The studies listed in Table 1 also record considerable variation in NP content even within material sub-types. This agrees well with the variation seen between the duplicate sub-types listed in Table 2 and supports the view that this variation could arise from the use of different ingredients or different processes in the production of the same material sub-type.

### ***Migration potential***

The detection of NP in some of the materials studied here raises the possibility that the chemical could migrate to food contained in these materials.

Migration depends on the concentration of NP available in a material coupled with the nature of the food in contact and the contact conditions of temperature and duration. (e.g. as NP is lipophilic, it is likely to migrate more strongly into material with a high lipid content). It should also be noted that in this work, the combination of methanol as extracting solvent and soxhlet extraction on finely divided material (in order to exploit larger surface areas) used here to analyse for NP represents a fairly aggressive extraction, particularly when compared to the conditions likely to be prevalent when most migration to food occurs. In this context, the levels detected in the materials probably represent an extreme migration potential.

Inoue *et al* (2001) reported, not surprisingly, that NP migrates more strongly (62.5%) into n-heptane than into water (0.23%) or acetic acid (4%). Investigating cooked rice wrapped in PVC film (containing NP up to 3300 µg/g) and reheated in a microwave for 1 min or left at room temperature for 30 min, they found maximum concentrations up to 410 ng/g in the surface of the rice sample and 171.8 ng/g for the whole sample. This value corresponded to the film with the highest NP concentration - 3300 µg/g. Since the concentration in the food is 5% of the initial concentration in the film, even



assuming 100% migration, the tests clearly used at least 5g of film per 100g portion of rice. This is unrealistic since at a typical film thickness of 20  $\mu\text{m}$  this means that the ratio would have been 1700  $\text{cm}^2$  film contacting 100 g rice. Their dietary intake estimation from this data, of 0.7  $\mu\text{g}$  /kg bw/day, gave a safety margin of 70000, based on a NOAEL of 50mg/kg bw/day (Cunney et al 1997, Chapin et al 1999)

Although not directly studying migration into food, a similar study (Ozaki and Baba 2003) found that NP in rubber products that could come in contact with food such as spatulas, migrated more strongly into n-heptane (6.6%) when compared to 20% ethanol (0.7%) and water (0.03%). The results indicated that migration of NP to food can occur during cooking and that food is affected at different rates depending on its hydrophobic content; e.g. if 60ml fatty food were cooked with a spatula with an NP migration level of 1.5  $\mu\text{g}/\text{ml}$  it would lead to 1.5  $\mu\text{g}/\text{g}$  intake per 60-kg adult per day. The study concluded that the migration amounts observed could be regarded as having no influence on an adult's endocrine system.

The Greenpeace study (TNO 2003) referred to above also looked at migration from poultry and cheese wrappers into the top layer of the food that they were in contact with. Although only 8 samples were studied, (with undetected levels of NP in 3 of the wraps), it appeared that in most cases, more NP was detected in foods that were in contact with wraps that showed higher NP content. The report concluded that migration of NP from the wrapper to food does take place, and indicated that the migration level of NP to the outer layer of food contained in the wraps was roughly 1% for meat products and 0.1 % for cheese products. Nemoto *et al* 2000, studied the migration of NP from wrapping film, HIPS trays and expanded polystyrene trays (as described above), as well as the concentrations of NP present in the fish that were contained in these packaging materials. Their results suggested that most of the NP detected in the fish was due to migration from the wrapping film.

It would be useful to supplement the literature described above with some more systematic studies of migration from a range of food contact materials into foods under realistic conditions of use.

## Conclusions

The analytical methodology developed and validated for the determination of NP has proved to be robust enough to investigate the occurrence of this chemical in a range of food contact materials. NP was detected at concentrations of up to 1.4µg/g in 85% of the materials studied. However 3 of the material sub-types, two polystyrenes and one PVC, showed concentrations of NP in the range 64 – 287µg/g. The results of this study are in broad agreement with the limited literature available. It is possible that the higher levels of NP detected in some of the materials have the potential for migration to food and some migration testing into foods would be informative.

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Table 1: Concentrations of nonylphenol reported in potential food contact materials

Reference	Material	Concentration detected	Method limit of detection	Notes
Inoue <i>et al</i> (2001)	PVC film used as food wrap	<500 to 3300 µg/g	500 µg/g	Retail samples
Noriko (1999)	PVC food wrap	0.19 – 0.63 µg/g	0.05 µg/g	Concentration/g or concentration shown to migrate?
	PVdC, nylon, polyolefin Polyethylene, polypropylene,	Not detected	0.05 µg/g	Food wraps
Greenpeace (TNO2003)	PVC and non PVC food wraps	0.05 to 62 µg/g	-	Food wraps used for poultry and cheese
Nemoto <i>et al</i> (2000)	EPS trays	< 0.4 to 83 ng/cm <sup>2</sup>	0.7ng/cm <sup>2</sup>	Retail samples – trays used to pack fish
	HIPS trays	120 to 270 ng/cm <sup>2</sup>		
	EPS film	70 to 931 ng/cm <sup>2</sup>	0.7ng/cm <sup>2</sup>	Retail samples – wrapping film
	HIPS film	7 to 873 ng/cm <sup>2</sup>		
Ozaki & Baba (2003)	Rubber products	< 2.0 to 439 µg/g	2.0 µg/g	Range of products with food contact use
Kawamura <i>et al</i> (2000)	PVC	10 to 2600 µg/g	10.0 µg/g	Wrapping film
	Polystyrenes, Polycarbonate, Polypropylene	17 to 499 µg/g		Range of food contact products & infant/nursing ware

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Table 2: Summary of Method Validation Parameters.

Parameter	Value
Method Limit of detection across all materials	0.03 µg/g
Measurement Linearity. Regression analysis value across the range 10 ng/g – 100 µg/g	0.9995
Average recovery across all materials	78%
Recovery from Fortified Sample Material (PET)	94%
Precision as % Coefficient of Variation for individual materials (PVC and HIPS)	~ 10%
Precision as % Coefficient of Variation across all materials	24%

Table 3: Concentrations of NP detected in material sub-types

Material Type	Sub-Type	Example Obtained	Concentration $\mu\text{g/g}$	Recovery (%)
Polyolefins and co-polymers	PE (HDPE)	Dairy drink container	< 0.03	102
	hard PP	Baby first years cup	0.05	100
	PE (LDPE)	Clingfilm (non-PVC)	0.06	102
	clear PP	Snack pots	0.05	95
Polystyrene and co-polymers	HIPS	Fish trays, Hard PS film	265	80
	EPS	Fish trays, Expanded PS	0.07	48
	PS buta co-poly	Ice-tray (butadiene co-polymer)	64.2*	112*
	PS buta co-poly	Filter coffee holders duplicate	< 0.03	72
Polyesters and co-polymers	hard PET	Water container	< 0.03	53
	film PET	Roasting bag	< 0.03	50
Chlorinated polymers and co-polymers	PVC film	All purpose clingfilm	287	107
	PVdC	Hard pvc	< 0.03	85
	Vinyl Acetate	Bread bag	1.42*	177*
	Vinyl Acetate	Wine stopper duplicate	0.50	60
Polyamides	Nylon	Fish container	0.11	73
	Nylon	Roasting bag duplicate	0.07	93
Polycarbonate	PC	Baby feeding bottle	0.31	88
Cellulose and cellulose esters	RCF	Fudge wrapper	0.09	50
	RCF	Cough sweet wrapper duplicate	0.25	95
Thermoset phenolic polymers	MF	Melamine	< 0.03	71
Rubbers	rubber latex	Baby natural latex teats	0.39	73
	synthetic r	Wine stopper duplicate	0.36	98
	synthetic r	Silicone teats	0.06	82
Papers	paper	Greaseproof paper,	0.04	98
	coated paper	Paper coffee cup	0.04	98

\*Estimated value due to co-extracted interference on  $^{13}\text{C}$  internal standard. Likely to be underestimate

# Analysis of Nonylphenol in Food Contact materials

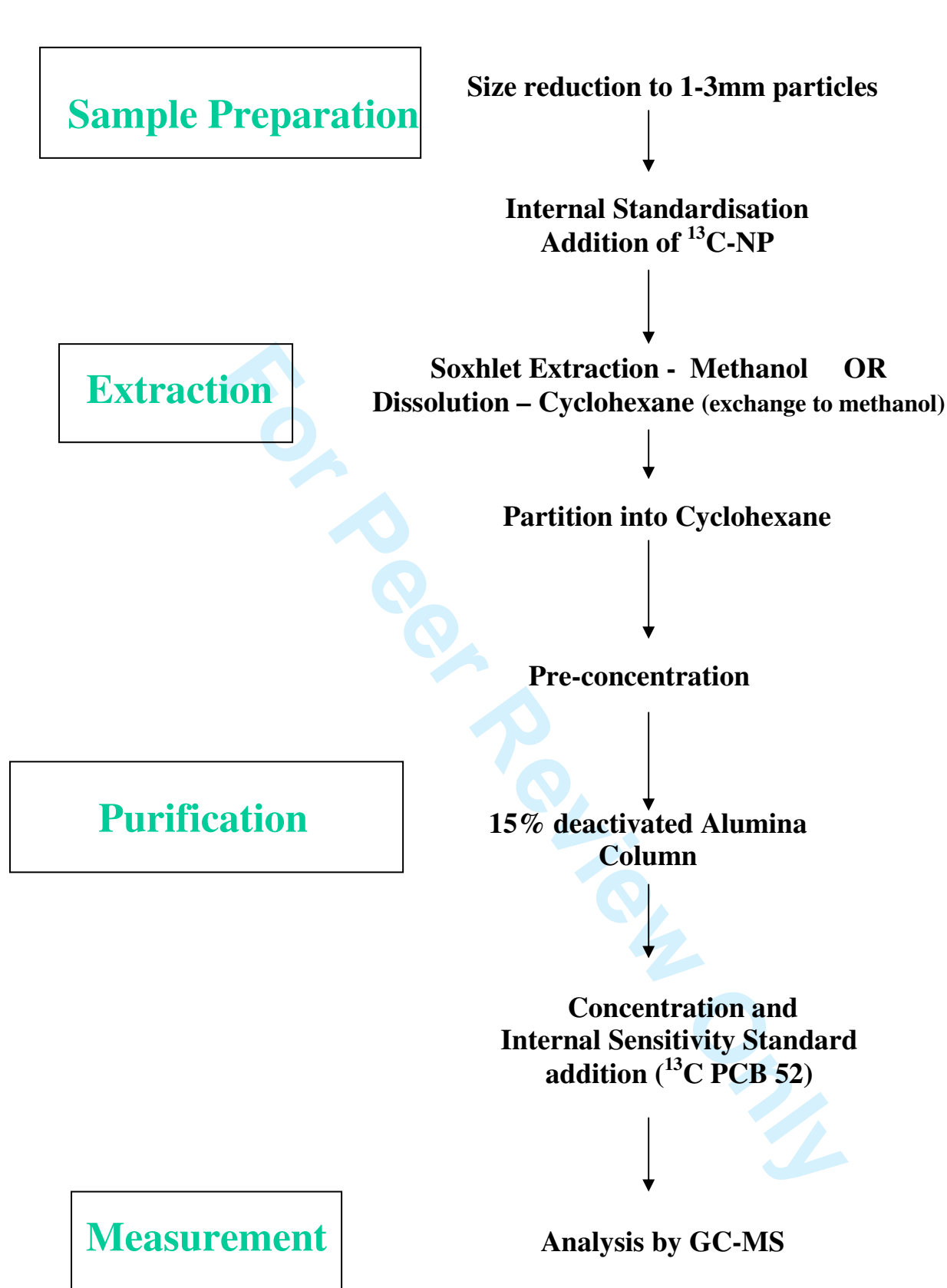


Figure 1 Schematic representation of NP analysis