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Analysis of Food Taints and off-flavours - A review

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ANALYSIS OF FOOD TAINTS AND OFF-FLAVOURS– A REVIEW

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Abstract

Taints and off-flavours in foods are a major concern to the food industry. Identification of the compound(s) causing a taint or off-flavour in food and accurate quantification is critical in assessing the potential safety risks of a product or ingredient. Even when the tainting compound(s) are not at a level that would cause a safety concern, taints and off-flavours can have a significant impact on the quality and consumers' acceptability of products. The analysis of taints and off-flavour compounds presents an analytical challenge especially in an industrial laboratory environment because of the low levels, often complex matrices and potential for contamination from external laboratory sources. This review gives an outline of the origins of chemical taints and off-flavours and then looks at the methods used for analysis and the merits and drawbacks of each technique. Extraction methods and instrumentation are covered along with possible future developments. Generic screening methods currently lack the sensitivity required to detect the low levels required for some tainting compounds and a more targeted approach is often required. This review highlights the need for a rapid but sensitive universal method of extraction for the unequivocal determination of tainting compounds in food.

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3 **Keywords:-** Food taints; off-flavour; sensory; headspace; GC-O; SPME; SBSE; SDE;
4 chlorophenols; electronic-nose
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8 **Introduction**

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10 A taint in food results from contamination by a foreign chemical derived from an external
11 source (e.g. from packaging or storage), whereas an off-flavour is an atypical odour or taste
12 resulting from a compound formed by internal deterioration in the food, such as
13 microbiological spoilage or lipid oxidation. However, this distinction is seldom made,
14 particularly in consumer complaints, as both can be picked up by odour or taste and give the
15 impression of poor food quality. Previous reviews on food taints have discussed the origins of
16 food taints in detail (Mottram 1998; Whitfield 1998), but this review also considers the
17 analytical approach to the determination of both known and unknown tainting compounds
18 and includes methods introduced in recent years for taint analysis.
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28 Methods of analysis for the determination of compounds causing taints and off-flavours are
29 generally the same. The presence of a taint may cause a food to be unfit for consumption,
30 however, unlike most chemical contamination, where there are established validated
31 analytical procedures and maximum permitted levels, there are no set limits for tainting
32 compounds.
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38 The compounds responsible for taints are frequently only present at trace levels (low ng g⁻¹),
39 and hence rarely pose a health risk to the consumer. However, the first question that must
40 always be asked following the discovery of a chemical taint in food being discovered is
41 whether there is any risk to human health based on risk assessment. This requires rapid
42 accurate analysis to identify and quantify the chemical(s) responsible for the taint and would
43 then typically be followed by root cause analysis and risk reduction measures, such as a
44 product recall. In general although a food with the taint or off-flavour is often not a safety
45 risk to the consumer, the perception of low quality, brand damage and adverse publicity can
46 be extremely costly to the food industry. Therefore it is imperative that the most appropriate
47 approach is used to reliably identify and quantify the taint and its occurrence. In rare cases,
48 where a food taint is due to gross contamination from a chemical leak (such as a solvent or
49 refrigerant), outbreaks of illness can occur (Dworkin *et al.*,2004).
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Sensory aspects and threshold values

The first step in any taint investigation is sensory analysis. This will only be briefly described in this review and more details can be found in books (Baigrie 2003; Heymann and Lawless, 1999; Howgate, 1999) and numerous papers on the subject (Dijksterhuis and Piggott, 2000; Piggott, 2000; Piggott, 1995; Sidel and Stone, 1993). The flavour of food is defined by both its odour and taste and most food taints are detected by odour. Odour refers to both the volatile compounds released in the mouth and those perceived from the food when external to the body (aroma). The 'taste' of food is technically experienced in the mouth by the taste-buds and can be attributed to both volatile and non-volatile compounds. Some compounds can be detected at extremely low concentrations (Table 1) and individuals may be more sensitive to certain odours and compounds. The possibility of someone detecting a taint is concentration dependent and if the sensitivity to detection is plotted against the log of concentration then an s-shaped curve is obtained (Figure 1).

Threshold values are used for the sensory analysis of taints, and are generally defined as the probability of detection being 0.5, that is 50% of the general population will detect a taint at that level. However, care should be taken when using such values, as each individual will have a different threshold and most compounds are measured in air or water and this may not be representative of detection in a real food matrix.

The sensory descriptor of a taint can often be the key to performing targeted chemical analysis. Sensory panels are trained to give objective assessments and descriptions of taints and can provide an insight, when a public consumer has complained that the foodstuff tastes 'funny'. A control/reference sample should always be assessed alongside the problem sample to enable a comparison with the 'normal' flavour of the product. Descriptors associated with specific tainting compounds can be used from reference guides (Bairgrie, 2003; Saxby *et al.*, 1992; Saxby, 1993), or specialised websites (www.odour.org.uk and www.flavornet.org). Artificial taste sensors have been developed in an attempt to replace or support the use of human panellists and were discussed in a recent review (Citterio and Suzuki, 2008). They concluded that currently no absolute models can correlate the taste that a human perceives with the chemical composition of a sample.

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It may be that more than one compound is responsible for a taint or off-flavour in food and this further complicates the sensory descriptors, as in the case of fishy off-flavour in dried spinach (Masanetz *et al.*, 1998) caused by two compounds, neither of which possessed a fishy character as an individual compound.

The origin of food taints

Taints and off-flavours can originate from many sources, including microbiological degradation, migration from packaging, contaminated process-water, or an unsuitable storage environment of ingredients or finished products. Some common taints associated with these sources are discussed in this section and summarised (Table 2), although it should be noted that the list is not exhaustive as changes in practices and developments in processes can lead to previously unknown taints being formed. Mottram (1998) described the origins of some chemicals responsible for taints and off-flavours in foods and gives details of several specific incidents. Examples of the causes of taints investigated in our own laboratories concluded that 'musty' tea was due to the presence of tribromoanisole; a soapy taint in soup was from decanoic and octanoic acids; disinfectant taints in soft drinks and instant soup powder from di- and tri-chlorophenols and in fish sticks were due to chlorocresol, all of which were a direct result of cross contamination during processing or storage. The move towards a more global supply chain and the possibilities for joint storage or transport has the potential to increase taint incidents in the food industry.

Taints from packaging

Packaging, particularly for food and beverages is designed to ensure products remain unchanged on storage, retaining the flavour and odour of the product whilst preventing external contamination. It is therefore prudent to carefully select packaging and control processes to minimise the likelihood that the packaging itself can become the source of a food taint. The problems and causes of odours and taints originating from packaging have been reviewed previously (Tice, 1993; Lord, 2003).

Taints from packaging can occur through direct contact or by vapour phase transfer of substances from the packaging to the food. In general, foods with high fat content or dry foods with a high surface area are most vulnerable. For direct contact, more migration will

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3 occur with fatty foods, where the oil and fat components can penetrate into the packaging and
4 their low polarity makes them a good matrix to absorb many organic contaminants. Neutral
5 products like bottled water can also be more susceptible to organoleptic influences. The food
6 packaging industry carries out regular taint and odour tests as part of their quality assurance
7 programs. These sensory tests assess the odour intensity of the packaging and usually involve
8 a taint comparison using a test food (e.g. a triangle test, including at least one control sample,
9 not exposed to the packaging).
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17 A wide variety of materials are used in food packaging and odours can originate not only
18 from the principal components, but also from impurities, additives, reaction products formed
19 during manufacture, or environmental contamination. The origins of tainting substances
20 formed from packaging materials include; inappropriate or contaminated raw materials,
21 incorrect or poor control during processing, chemical reactions within the packaging material,
22 and storage and transport conditions. A good example of the investigations often required
23 was an instance in our own laboratory of taint in peanut butter, which was traced back to the
24 lacquer on storage drums, migrating through the plastic bags containing the product. This
25 also illustrates the importance of taking representative samples, as the taint was only
26 observed round the edges at the top of the drum.
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37 Inks used on the outer surfaces or materials used for secondary packaging may migrate into
38 the packaged product, either by direct contact or transfer in the vapour phase. Paper and
39 carton board materials often form part of a multilayer packaging with adhesives, varnishes
40 and plastics. Each component could provide a source of compounds that may result in food
41 tainting.
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48 The use of recycled or reuse of packaging can also lead to food taints, including consumer
49 misuse as illustrated in a study investigating contaminants in water from reusable PET bottles
50 (Widén *et al.* 2005).
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54 *Inks*

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56 Examples of taints originating from packaging include residual solvents from inks and
57 varnishes, which generally are a result of insufficient drying after printing. There are no
58 generally agreed maximum levels for residual solvents in food packaging as many factors
59 determine whether the residue will result in a taint in the food. UV-cured inks and varnishes
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3 are essentially solvent-less, but residual acrylate monomers, photoinitiators (Sagratini *et al.*,
4 2008), such as benzophenone, or reaction by-products from the polymerisation process, such
5 as benzaldehyde and alkyl benzoates, can lead to trace odours, that could migrate into the
6 food product. Mesityl oxide (4-methylpent-3-en-2-one), previously used as a solvent for
7 paints and lacquer coatings, can react with hydrogen sulphide (present naturally in many
8 foods) to form 4-mercapto-4-methylpentan-2-one, known to produce a catty odour (Mottram,
9 1998).

16 17 *Residual monomers*

18 In plastics packaging, residual monomers are one of the main sources of potential taints.
19 Styrene, for example, has a relatively low odour threshold and also can be formed from the
20 plastic packaging if excessive heat is used in processing. The detection of styrene taint in
21 food is very dependent on the type of food product (Gilbert and Startin, 1983; Linssen *et al.*,
22 1991). Contamination of cheese by styrene dibromide (used as a catalyst in polystyrene
23 manufacture up to the 1970s) has been reported following migration of leachate from
24 polystyrene cold storage insulation (Bendall, 2007). Monomers used in polyethylene
25 terephthalate (PET) packaging, although not particularly odorous, can form degradation
26 products, such as acetaldehyde, during the manufacturing process, which have been known
27 to cause taints in beverages (Lorusso, 1985). Similarly, although residual monomers present
28 in polyethylene, polypropylene and related copolymers are not generally responsible for
29 odours, oxidation compounds have been identified, such as 1-heptan-3-one and 1-nonenal
30 (Koszinowski and Piringer, 1986).

42 43 *Paper and board*

44 Odours can be present in paper and board packaging and can arise from bacteria, moulds,
45 auto-oxidation of residual resins, and the degradation of processing chemicals. Soderhjelm
46 and Eskelinen, (1985) gave a list of volatile compounds found in pulp samples, along with
47 odour descriptors. Decarboxylation and oxidation of lignin can produce vanillic acid and its
48 subsequent degradation causes the presence of guaiacol (Chatonnet *et al.*, 2004). If a
49 synthetic resin binder is used, particularly one based on styrene/butadiene, odorous volatile
50 by-products can be produced. Hexanal is often found in paper and board at low levels and
51 can also give rise to a taint. Metallic ions present in the pulp can act as catalysts for the
52 oxidation of lipids and give odorous volatiles, such as aldehydes, alcohols and esters (Tice
53 and Offen, 1994), but these compounds are usually present at too low a level to impart a
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3 noticeable odour. However, some paper and board can become more odorous on storage due
4 to such oxidation reactions and complexing agents are commonly added to reduce the level of
5 free metal ions, which can act as catalysts. Surface coatings on paper and boards can add
6 another potential source of taints and careful selection of inks and varnishes and control of
7 the printing and drying process is advisable to minimise taint incidents. Migration studies of
8 model compounds have shown that migration depends on the nature of the paper samples and
9 that more migration occurs from packaging into products with higher fat content
10 (Triantafyllou *et al.*, 2007). The use of recycled rather than virgin board for food contact
11 applications could also lead to potential contaminants from inks or previous use, if paper
12 sources and recycling processes are not strictly controlled and monitored.
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22 *Fungicides – halophenols*

23 One of the most commonly reported taints in foods is due to contamination by chlorophenols
24 and chloroanisoles. Chlorophenols have been used industrially as fungicides, biocides and
25 herbicide intermediates, most commonly in the treatment of wooden storage pallets.
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28 Chlorophenols can be microbially methylated by numerous organisms to the corresponding
29 chloroanisoles (Leonard *et al.*, 1974). Pallets made from soft wood that has been treated with
30 certain fungicides can therefore be responsible for taints due to the migration of
31 chlorophenols or chloroanisoles into ingredients or products during storage.
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38 Pentachlorophenol (PCP) is now rarely used in most countries due to concerns over toxicity
39 and as a consequence there are less taint incidents from trichloroanisole. However, the use of
40 bromophenols in place of chlorophenols can also lead to the formation of bromoanisoles
41 through microbial methylation. Brominated anisoles generally have lower sensory thresholds
42 than chlorinated anisoles. 2,4,6-Tribromoanisole in particular, has a very low sensory
43 threshold and has been linked to taints originating from treated wooden pallets. The use of
44 tribromophenol as a timber treatment can make an entire building unsuitable for food
45 production (Chatonnet *et al.* 2004). Halophenols can also be formed when phenols present in
46 wood/board from the decomposition of the lignin react with a source of bromine or chlorine
47 and similarly tribromophenol can be formed by the reaction of certain biocides with phenol.
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58 There have been several reports of the contamination of food with chlorophenols and anisoles
59 originating from packaging materials (Lord, 2003). The packaging affected included jute
60 sacks, multi-wall paper sacks (where PCP was used as a biocide in an adhesive used to glue

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3 the seams), fibreboard cartons and even wooden pallets on which carton board has been
4 stacked.
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7 ***Water as a source of taints***

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10 If food is produced using mains water that has been contaminated by tainting compounds,
11 then it is probable that the product will also be tainted. Water containing a source of phenol
12 (for example from peat soil), that is then chlorinated can easily produce chlorophenols.
13 Similarly if bromine is present then bromophenols can be produced. Tastes and odours in the
14 aquatic environment can originate from naturally occurring compounds derived from the
15 activity of micro-organisms in soil or water, or from oil or petroleum spills (Davis, Moffat
16 and Shepherd, 2002; Howgate, 1999). Most taints detected in fish originate from the aquatic
17 environment (Tucker 2000; Whitfield, 1999). Sulphur compounds formed from precursors,
18 such as plankton, can cause taints in fish. For example, a taint often described as petroleum
19 has been reported due to the presence of dimethyl sulfide (DMS) (Whitfield, 1999) and fish
20 and crustacean have been reported to have iodoform or iodine like taints, attributed to
21 bromophenols (Whitfield *et al.*, 1988). A common taint reported in water as earthy-musty is
22 due to geosmin, 2-methylisoborneol (MIB) and haloanisoles (Zhang *et al.*, 2005), and is
23 generally associated with micro-organisms, particularly bacteria (Watson *et al.*, 2003). Other
24 compounds reported to cause taint in water, include 2-isopropyl-3-methoxypyrazine (IPMP)
25 and 2-isobutyl-3-methoxypyrazine (IBMP), which are metabolites of *Actinomyces* and soil
26 bacteria. Various treatment processes have been developed to remove off-odours from
27 potable water (Suffet *et al.*, 1993).
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45 ***Cleaning products***

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47 A large number of reported taints each year originate from cleaning products or disinfectants
48 (Olieman, 2003). These taints can occur accidentally due to the transfer of volatiles or poor
49 rinsing, or from direct contact if 'no-rinse' products are used. Disinfectants based on active
50 chlorine, iodine or oxygen can react with food components (such as phenols) to form
51 additional compounds – for example halophenols and potentially haloanisoles. Methyl
52 ketones present in the majority of foods at low concentrations can react to form chloroform or
53 iodoform. These reactions can depend on the presence of other compounds, for example
54 sequestering agents for metals can be added to decrease metal-catalysed formation reactions,
55 whereas the presence of quaternary ammonium compounds can increase reactions (Olieman,
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2003). New polymer flooring, contaminated with traces of phenol, can react with chlorine-based disinfectants to produce chlorophenols (Mottram, 1998). If chlorine-based disinfectants are used on the same site as phenolic disinfectants then a reaction can occur – not only in the drain but also potentially in the atmosphere. The presence of microorganisms can lead to the formation of tribromoanisole, which has an extremely low sensory threshold and can lead to considerable taint problems in a factory environment.

Micro-organisms

The micro-organisms generally associated with off-favours in food, bacteria and fungi have been reviewed by Whitfield (1998). The food affected, includes meat, dairy products, fruit, vegetables and cereals, and a wide range of compounds with varied sensory descriptors can be produced (Whitfield, 2003; Springett, 1993). Examples include the production of guaiacol from vanillin (Perez-Silva *et al.*, 2006; Varez-Rodriguez *et al.*, 2003), a compound responsible for the vanilla flavour in products, such as ice cream, and an off-flavour produced by *Penicillium* species in margarine (Hocking *et al.*, 1998). Sorbic acid, used as a preservative in food, can be converted by mould to give pentadienes and 1,3-pentadiene causes taints in various foodstuffs (Loureiro and Querol, 1999). Pinches and Apps (Pinches and Apps, 2007) described the production in food of 1,3-pentadiene and styrene by *Trichoderma* species. The production of styrene in foods has been linked to the action of a specific yeast on cinnamaldehyde, although the presence of cinnamon or cinnamon flavours is not a prerequisite for styrene production (Spinnler *et al.*, 1992). Two bacterial species and their metabolites have been linked to the production of compounds, such as guaiacol, dibromophenol, geosmin and 2-methylisoborneol, in apple juice (Zierler *et al.*, 2004), leading to an off-flavour described as musty/earthy or medicinal-like.

Food reaction off-flavours

Thermal processing and the Maillard reaction are responsible for many food flavours and can also be responsible for some off-flavours in foods. Examples include the browning and flavour deterioration of fruit juices on storage, attributed to Maillard reaction products such as substituted furfurals, furans and pyrroles (Handwerk, and Coleman, 1988) and similarly the deterioration of UHT milk flavour during storage (Valero *et al.*, 2001). However, lipid oxidation is generally considered the main source of off-flavours in foods. There are several mechanisms for lipid oxidation, which have been reviewed by Saxby (Saxby, 1993) and Hamilton (Hamilton, 2003). Common compounds associated with the resultant 'rancid' off-

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flavours, include aldehydes, ketones, lactones and furans, carboxylic acids, alcohols and hydrocarbons.

Cork taint

One of the most well known food taints is the musty taint in “corked” wines, and many papers have been dedicated to the subject (Evans *et al.*, 1997; Ezquerro and Tena, 2005; Gomez-Ariza *et al.*, 2004a; Insa *et al.*, 2005; Juanola *et al.*, 2004; Juanola *et al.*, 2002; Martinez-Urunuela *et al.*, 2004a; Martinez-Urunuela *et al.*, 2004b; Martinez-Urunuela *et al.*, 2004c; Martinez-Urunuela *et al.*, 2005; Riu *et al.*, 2002; Taylor *et al.*, 2000; Zalacain *et al.*, 2004). Several compounds are thought to contribute to the ‘cork’ taint in wine and can originate from practices during wine production. (Soleas *et al* 2002). Chloroanisoles, in particular 2,4,6-trichloroanisole, due to its low sensory threshold, have been identified as a potential cause. The presence of chloroanisoles in cork can be due to the microbial degradation of chlorophenols (used in insecticides and herbicides) or chlorinated solutions used to bleach the cork. Other off-flavours in wine can originate from a number of sources, including fungal flora on the grape, formation by yeasts or bio-methylation of phenols (Boutou and Chatonnet, 2007). 2,4,6-Trichloroanisole has also been identified as causing a musty/muddy off-flavour in sake and was thought to originate from the wooden tools used in preparing rice koji for sake brewing (Miki *et al.*, 2005).

Methods of chemical analysis

As the majority of taints are detected through odour (inside or outside the mouth) , most of the compounds that cause taints in food are volatile. As discussed earlier, sensory thresholds mean that extremely low levels can give rise to a taint – which presents a challenge to the analyst trying to identify the chemical compound(s) responsible. Following sensory analysis, the identification of the compound causing the taint is necessary to determine the cause and prevent re-occurrence. If the compound is known, then targeted analysis can be performed. However, often this is not the case and a more investigative approach is required. The description of the taint provides key information to the analyst, as any potential compound identified in the sample must have the same taste and odour characteristics as those described from sensory analysis. It is often necessary to predict what the compound might be from sensory descriptors and background information before starting the chemical analysis.

Requirements

The determination of taints and off-flavours in foods often involves two approaches as illustrated in the schematic in Figure 2. The initial procedure to identify differences in the volatile profile of the tainted sample compared to a 'good' control sample, followed by chromatographic analysis to enable the identification and quantitation of any compounds against standards. The sampling procedures employed are very important as a chemical causing a taint may not be evenly distributed throughout a product or ingredient. This is particularly the case for gross chemical contamination, such as solvents or with compounds migrating from packaging, where 'hot spots' can occur.

If the initial tests suggest a potential suspect then a targeted extraction can be employed. For a true screening method, where the cause of the taint is unknown, a wider more universal method is required than for targeted extraction and analysis. The tainting compound, however, may be present at very low levels and will need to be isolated from high concentrations of matrix components. Sometimes large sample sizes are needed to obtain a high enough concentration to enable detection, therefore the removal of matrix interferences without the loss of the compound(s) of interest presents a challenge to the analyst. As the majority of compounds responsible for taints are volatile, care must be taken to avoid losses during sampling and analysis, in particular during any solvent removal step, particularly if concentrating to small volumes (Ferreira *et al.*, 1998, Jakobsen *et al.* 2003)

Determination of chemicals causing food taints is a not an easy procedure and care must be taken to avoid all possibilities of contamination from external laboratory sources (including perfumes and personal care products used by the analysts). A dedicated area is preferred and all control and suspect samples, and reference standards should be handled and stored separately. Whereas initial identification of a compound can be predicted using library spectral searches (such as NIST mass spectral library), the use of analytical standards are essential in the unequivocal identification of a chemical compound. Extreme care should be taken with identification of 'extra' peaks observed in the chromatographic profile of the suspect sample and results should always be compared with sensory analysis and other available information to ensure an accurate diagnosis is made.

Extraction methods

There are several methods for the extraction of flavour volatiles (Marsili, 1996; Wilkes *et al.*, 2000), including liquid-liquid extraction (Weurman, 1969), simultaneous steam distillation solvent extraction (SDE) (Nickerson and Likens, 1966), static headspace (Chialva *et al.*, 1983), dynamic headspace (Chatonnet *et al.*, 2004), direct thermal desorption (Hoffmann and Sponholz, 1994) solid-phase microextraction (SPME) (Yang and Peppard, 1994) and more recently headspace sorptive extraction (HSSE) (Lorenzo *et al.*, 2006) and stir bar sorptive extraction (SBSE) (Nakamura *et al.*, 2001). Miniaturised techniques have more recently been employed, such as headspace liquid phase microextraction (HS-LPME) for chlorophenols (Hui *et al.*, 2007) and geosmin (Bagheri, and Salemi, 2006) in water. Closed loop stripping techniques (CLSA) have also been used for odorants in water (Hassett and Rohwer, 1999; Zander and Pingert, 1997). The choice of extraction method will depend upon the matrix and the predicted cause of the taint. The sensory data should give an indication of the compounds responsible for the taint and therefore the sensitivity of technique required. Sample preparation methods for chlorophenols in environmental, enological and biological samples were recently reviewed by Quintana and Ramos (Quintana and Ramos, 2008) who highlighted the need for different approaches for different matrix types. A 'fit for purpose' approach should be taken, considering both identification and quantification requirements.

Solvent extraction

Some methods have been reported for taints and off-flavours in foods that use direct solvent extraction. Indole and skatole have been associated with a taint in meat from male pigs and methods using direct solvent extraction, followed by HPLC, have been reported (Regueiro and Rius, 1998). In this example, fluorescence detection provided selectivity, but generally further clean-up stages are required. By performing several liquid-liquid partitions, and using pH adjustment it is possible to obtain a fraction containing the problem odour, but for complex matrices, such as foods, several matrix components may still be present, making accurate identification and quantitation difficult. Solvent extraction has been used for the analysis of chlorophenols and chloroanisoles in cork (Juanola *et al.*, 2002) and tribromoanisole in wine (Chatonnet *et al.*, 2004).

Solvent extraction methods generally require a subsequent concentration of the solvent by rotary evaporation or the use of solid phase extraction (SPE), but this can lead to a loss of analytes (Ezquerro and Tena, 2005; Riu *et al.*, 2002). Juanola *et al.* (Juanola *et al.*, 2002)

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3 used a 'shake-flask extraction' followed by silica column clean-up for the analysis of 2,4,6-
4 trichloroanisole in corks and compared the results to the use of Soxhlet and ultrasound
5 extraction methods. In all the methods a concentration step using a rotary evaporator and then
6 drying under a nitrogen flow was necessary. Procedures using SPE as a clean-up step can be
7 developed if sensory analysis can provide clues to the target compounds. SPE methods have
8 been reported for chloroanisoles (Insa *et al.*, 2005; Soleas *et al.*, 2002) and for both
9 chloroanisoles, and chlorophenols with derivatisation (Martinez-Urunuela *et al.*, 2005).

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12 Other solvent extraction methods include supercritical fluid extraction for 2,4,6-
13 trichloroanisole (TCA) in cork (Taylor *et al.*, 2000) and androsterone and skatole in pigs
14 (Zabolotsky *et al.*, 1995), Soxhlet extraction for analysis of trichloroanisole from corks
15 (Juanola *et al.*, 2002) as well as microwave extraction and pressurized fluid extraction
16 (Ezquerro *et al.*, 2006; Gomez-Ariza *et al.*, 2005).

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19 However, for true unknowns, isolation from matrix components and concentration can be a
20 challenge. Therefore direct solvent extraction is generally only used for targeted taint analysis
21 when the compound responsible for the taint is known and is present at a relatively high
22 concentration.

23 24 25 *Steam Distillation and SDE*

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28 As the majority of compounds that cause a taint or off-flavour are volatile, steam distillation
29 can be used for extraction from the non-volatile food components. The distillate can then be
30 further extracted or concentrated. Distillation has been used for the analysis of
31 trichloroanisole in wine (Juanola *et al.*, 2002). For thermally labile compounds, the
32 distillation can be performed under vacuum using lower temperatures. Microwave assisted
33 steam distillation has also been employed for tainting compounds, such as the extraction of
34 geosmin and methylisoborneol from catfish (Conte *et al.*, 1996; Lloyd and Grimm, 1999) and
35 chlorophenols from solid samples, such as soil and wood (Ganeshjeevan *et al.*, 2007).

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38 Combined steam distillation and solvent extraction (SDE) is one of the most widely used
39 techniques for the extraction of volatile tainting compounds and has been reported for the
40 analysis of trichloroanisole in wines (Hill *et al.*, 1995). SDE can avoid the extraction of major
41 matrix components as described (Landy *et al.*, 2004) in a study on odour-active compounds
42 in packaging. The use of SDE was required to enable the identification of compounds

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3 following spectral interferences from the high concentration of hydrocarbons using other
4 techniques. The original apparatus was first described by Likens and Nickerson (Likens and
5 Nickerson, 1964). A recent review of the technique (Chaintreau, 2001) describes some
6 changes and variations. The sample is placed in one flask (with water) and the extracting
7 solvent in the other. Both are boiled and the vapours mix and condense in a central chamber,
8 with the condensates returning to their original flasks. Volatile compounds distil out of the
9 sample with the steam, are extracted into the solvent in the central chamber and are
10 transferred to the solvent flask. Large sample sizes can be used as only the volatile
11 components are extracted and as volatilisation, condensation and extraction form a cyclic
12 process, a minimal amount of extracting solvent can be used. For some compounds, where
13 ultra-trace levels can be responsible for a taint, a further concentration step may still be
14 required. The method is matrix and analyte dependent and samples with high fat/lipid content
15 can reduce recoveries. However, for most matrices, good recoveries can be obtained, and
16 adjustment of pH can be made to encourage the extraction of certain compounds, such as 2,6-
17 dibromophenol (Whitfield *et al.*, 1988).
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32 One disadvantage of SDE is the potential break down of labile compounds and the possibility
33 of the formation of extra compounds either thermally or by oxidation (Chaintreau 2001,
34 Siegmund 1997). Vacuum SDE has been shown to reduce artefact formation by enabling
35 extraction at lower temperatures (Chaintreau, 2001) although a relatively non-volatile
36 extracting solvent should be used to avoid losses during the extraction.
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42 The advantage of SDE is that it can be used for a wide variety of food matrices and produces
43 a clean extract of volatile components. Large sample sizes can be taken and with the
44 inclusion of a concentration step excellent sensitivity is achievable (sub $\mu\text{g}/\text{kg}$ (ppb) levels).
45 The major disadvantage of this technique is the need for specialist glassware and the
46 possibility of cross contamination and losses on concentration. It is important to analyse both
47 a 'control' sample and suspect sample using each set of glassware, to enable identification of
48 genuine differences.
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54 *Thermal desorption*

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56 For solid samples, direct thermal desorption can be used including, for example, the
57 determination of trichloroanisole in corks (Caldentey *et al.*, 1998). Thermal decomposition
58 GC/MS of food packaging has been successful in identifying off-odour components in
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3 packaging material as well as in the original polymer (Hartman, 2007; Woodfin and George,
4 2003). This technique is only suitable for solid samples and requires the contaminant to be at
5 a level that can be detected above matrix components. Quantitation methods also need to be
6 optimized to replicate sample analysis. For complex matrices and unknown taints, direct
7 static headspace is more commonly used.
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10 11 12 13 *Direct static headspace*

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15 Static headspace is very useful for the general profiling of volatiles and can be used as a first
16 step to detect differences between 'good' and 'bad' samples. Examples include the quality
17 control of aromatic herbs (Chialva, 1983), musty taints from packaging (Mcgorrin *et al.*,
18 1987) and the determination of off-flavours in infant formula (Romeu-Nadal *et al.*, 2004). If
19 the tainting compound is present at a relatively high level then the additional
20 chromatographic peaks in a "bad" sample can be identified using a mass spectral library.
21 Standards should always be run under the same conditions for confirmation of retention time
22 and mass spectra. For accurate quantitation, the method of standard additions is
23 recommended, or if possible, the use of an internal standard (ideally an isotopically labelled
24 analogue). Although static headspace allows for a representative sample to be taken for
25 flavour analysis, often it only detects the most intense compounds. It is useful as an initial
26 screening method for detecting differences between control (untainted) samples and those
27 contaminated with a tainting compound. Recent developments in software that can allow for
28 chromatographic subtraction and difference analysis can be employed to aid the analyst in
29 differentiating complex volatile profiles. It is often the first step in a taint investigation and
30 can be used for most food types (or packaging), however, the sensitivity of the technique may
31 still be inadequate for some taints and techniques that include a concentration step (such as
32 headspace-SPME) are increasingly being used.
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48 49 *Dynamic headspace*

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51 Some tainting compounds will illicit an adverse olfactory response at extremely low levels
52 and can be difficult to detect using direct static headspace, particularly where the cause of the
53 taint is unknown. So-called dynamic headspace techniques, such as purge and trap, enable
54 concentration from the sample headspace and can improve sensitivity. In the determination of
55 bromophenols in water with *in situ* acetylation (Blythe *et al.*, 2006) the analytes were trapped
56 on a very small quantity of activated carbon (1.5 mg Grob tube) and eluted using 20-30 µl of
57 solvent prior to GC-MS analysis. Purge and trap systems using Tenax traps have also been
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3 reported for odorous compounds in water (Salemi *et al.*, 2006) and volatile compounds from
4 cork (Caldentey *et al.*, 1998).
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9 A recently reported technique comparable to a dynamic headspace method is pervaporation
10 (Gomez-Ariza *et al.*, 2004a) based on evaporation and diffusion through a membrane which
11 helps to minimise matrix effects and prevent water vapour interferences. It can be used online
12 with GC (Gomez-Ariza *et al.*, 2004b) and to achieve better sensitivity the technique can be
13 used with a solid phase trap (Gomez-Ariza *et al.*, 2006) or packed inlet liner (Gomez-Ariza *et*
14 *al.*, 2004c). Dynamic headspace techniques are rarely used for food taint analysis and the
15 traditional purge and trap devices can have problems with carry over. Although dynamic
16 headspace provides a concentration step, for complex matrices such as food, matrix volatiles
17 are also concentrated and thus the technique provides little advantage over direct static
18 headspace for most applications.
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26 27 *Solid-phase microextraction (SPME)* 28

29 Solid-phase microextraction can be used to increase the selectivity and sensitivity for some
30 volatile compounds. Initially SPME was used to quickly obtain volatile profiles of a wide
31 range of foodstuffs, including fruits, vegetable oils, coffee and milk (Yang and Peppard,
32 1994; Marsili, 1999). Yang and Peppard, (1994) compared direct immersion and headspace
33 sampling for 25 common flavour compounds. More recently, headspace-SPME extraction has
34 been increasingly used for flavour volatiles and Steffen and Pawliszyn, (1996) described the
35 quantitative analysis of some flavour volatiles in orange juice. A number of papers have
36 reported the use of HS-SPME for chloroanisoles and chlorophenols (Ezquerro and Tena,
37 2005; Bianchi *et al.*, 2003; Insa *et al.*, 2005; Juanola *et al.*, 2005; Martinez-Urunuela *et al.*,
38 2004b; Riu *et al.*, 2002; Riu *et al.*, 2006) and other compounds responsible for musty-earthly
39 off-odours (Prat, 2008) in cork (Figure 3).
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50 SPME has been employed for the determination of iodinated trihalomethanes in water
51 (Cancho *et al.*, 1999), 2-methylisoborneol and geosmin in environmental waters (Saito *et al.*,
52 2008) and off-flavours in milk (Marsili, 1999). The selectivity of SPME sampling means that
53 although some compounds will not be adsorbed by the fibre (Yang, and Peppard, 1994),
54 generally the background will be less than using direct static headspace (Marsili, 1999).
55 However, it should be noted that for very volatile compounds, direct headspace often gives a
56 better response than SPME (Zhang *et al.*, 1994) and matrix effects in SPME can be a
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3 problem. Consideration should also be made for the sample type, as for oil based samples the
4 matrix can decrease the sensitivity of headspace SPME sampling and higher temperatures
5 may be required (Yang and Peppard, 1994). As SPME is an equilibrium technique, the results
6 depend strongly on the experimental conditions and sample matrix.
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11 External calibration methods are generally not suitable for quantitation and the use of a
12 labelled internal standard or the method of standard additions may be required for accurate
13 quantitation. Boutou and Chatonnet, (2007) used HS-SPME for wine off-flavours with
14 labelled internal standards for quantitation. Similarly McCallum *et al.* (2008) used deuterated
15 geosmin and 2-methylisoborneol for the determination of the native compounds in water
16 Vlachos *et al.*, (2007), used HS-SPME GC-ECD for the analysis of 2,4,6-trichloroanisole in
17 wine and cork soaks, employing 2,3,6-trichlorotoluene as an internal standard for
18 identification. However, due to matrix affects when more than 3 corks were extracted,
19 external calibration and the method of standard additions was necessary for accurate
20 quantification.
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31 The sample matrix can be modified to increase the recovery of the target compounds, such as
32 acidification for extraction of phenols or the addition of salt (Riu *et al.*, 02). However, Evans
33 *et al.*, (1997) reported that the addition of salt did not increase the response for the analysis of
34 2,4,6-trichloroanisole in wines. For some analytes, such as the detection of limonene in
35 aqueous systems (Yang and Peppard, 1994), it can have a negative effect. Derivatisation can
36 also be used in SPME, either in the matrix solution prior to extraction (Martinez-Urunuela *et*
37 *al.*, 2004b) or on-fibre after analyte absorption (Pizarro *et al.*, 2007b).
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46 Fibres can be chosen to suit the analyte properties. Yang and Peppard concluded that
47 polyacrylate fibres suited higher polarity compounds compared to PDMS (Yang and Peppard,
48 1995) and Adams *et al.* (Adams *et al.*, 1999) used polyacrylate fibres for the determination of
49 bromophenols in water and model systems. A PDMS/DVB fibre has been reported to give the
50 best sensitivity for chloroanisoles (Carasek *et al.*, 2007), and was also chosen for
51 determination of geosmin and 2-methylisoborneol (McCallum *et al.*, 1998).
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58 Multiple headspace SPME has been used to study the volatiles in cork (Ezquerro, and Tena,
59 2005), haloanisoles and chlorophenols in wine (Martinez-Urunuela *et al.*, 2005; Pizarro *et al.*,
60 2007a). Using repeated consecutive extractions from the same sample, this technique enables

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3 an estimate of the complete extraction of the analyte, overcoming problems with matrix
4 affects. Juanola *et al.*, (2004) compared sensory and instrumental analysis using HS-SPME
5 and results showed the ability of sensory measurement to predict trichloroanisole content in
6 wine.
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12 Zhang *et al.*, (2005) used SPME with cool inlet PTV injection, to improve sensitivity for
13 several odorous compounds in water. A recent development in SPME – that of cold fibre
14 SPME, (CF-SPME), which allows for the simultaneous cooling of the fibre coating whilst
15 heating the sample, has also been employed for the determination of chloroanisoles in cork
16 (Carasek *et al.*, 2007). This technique was compared to normal HS-SPME and was shown to
17 give improved quantification limits, with recoveries >90% providing almost exhaustive
18 extraction.
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26 SPME is used widely for flavour profiling, and is increasingly being employed for targeted
27 taint analysis. However, the need to optimise the technique for each matrix limits its use as a
28 screening method for unknown taints . The technique can be used where the compound
29 responsible for the taint is known and can provide relatively low detection limits for specific
30 applications. For accurate quantitation, the method of standard additions is often required, or
31 the use of a suitable internal standard. It has been used successfully for a range of tainting
32 compounds (Boutou and Chatonnet, 2007) and provides superior sensitivity compared to
33 direct headspace analysis, but to date no screening method has been reported for
34 determination of unknown taints.
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43 *Stir bar sorptive extraction (SBSE)*

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45 Chloroanisoles and chlorophenols in cork have been studied by Hayasaka *et al.*, (2003) and
46 Callejon *et al.*, (2007), using an initial liquid-solid extraction of the corks followed by SBSE.
47 By adjustment of the pH, migration of the phenols into the non-polar PDMS extracting phase
48 was enhanced (Chatonnet *et al.*, 2004; Zalacain *et al.*, 2004). Alternatively *in-situ*
49 derivatisation can be used as described by Kawaguchi *et al.*, (2005) for the determination of
50 chlorophenols in river water and urine. Derivatisation is commonly used for determination of
51 chlorophenols due to the poor GC response and tailing peaks obtained for these compounds
52 (Figure 4). SBSE has also been used for the determination of 2,4,6-trichloroanisole in sake
53 (Miki *et al.*, 2005) and benzophenone and derivatives in river water (Kawaguchi *et al.*, 2006).
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3 Similarly to SPME, SBSE can also be used to selectively extract volatiles from the headspace
4 above a sample after heating, known as headspace sorptive extraction (HSSE). Marsili and
5 Laskonis, (2006) compared SBSE and HSSE for the determination of off-flavour chemicals
6 in beer and concluded that SBSE detected more odour active compounds and provided the
7 most accurate quantitation. HSSE has been used for the determination of chloroanisoles in
8 cork (Lorenzo *et al.*, 2006), enabling a non-destructive method to be developed (Figure 5).
9 The larger volume of coating compared to SPME, means that analytes are extracted into the
10 bulk phase and this allowed higher temperatures to be used to enable extraction of the
11 contaminants from the cork matrix.
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21 SBSE and HSSE are not exhaustive extraction techniques and as with all equilibrium based
22 techniques internal standards or the method of standard additions are generally employed for
23 quantitation. Both techniques provide the high concentration factors which are necessary for
24 detecting trace level tainting compounds, but to date have only been employed for targeted
25 analysis.
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31 ***Instrumentation***

32 *GC-MS*

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34 As the majority of flavour compounds (and therefore off-flavours and taints) are volatile, the
35 analytical instrumentation of choice is invariably GC-MS. In order to allow for mass spectral
36 matches with libraries to identify unknown compounds the most common instrumentation is a
37 single quadrupole instrument using electron impact ionisation (EI (+)) at 70 eV. Ion trap
38 instruments (Insa *et al.*, 2005) and more recently time of flight (TOF) instruments (Carasek *et*
39 *al.*, 2007; Marsili and Laskonis, 2006), offer full spectra information and can also provide
40 adequate sensitivity for quantitation. The automation of the sample preparation step now
41 enables on-line extraction, including headspace systems and SPME with direct injection or
42 automated thermal desorption in SBSE.
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53 *GC-Olfactometer (GC-O)*

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55 A GC-O or 'sniffer port' can be used alongside a traditional GC detector to allow an analyst
56 to identify the odour of a peak as it elutes from the GC column. The GC effluent is mixed
57 with humid air and a trained panellist records the time, intensity and descriptor of the odour,
58 producing an 'odourgram' of retention time vs sensory response. The GC-O detector can be
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3 coupled (via a splitter) with FID for quantitation or more commonly now with MS to provide
4 identification of the odour causing compounds. The sensory response can be overlaid with the
5 GC-MS chromatogram. GC-O can be useful in correlating odours to compounds, and can be
6 used as an initial screening of volatile compounds or to confirm the presence of a specific
7 taint compound. Quantification can be performed subsequently using instrumental techniques
8 such as GC-MS.
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15 Individual compounds can be quantified using GC-O, either by using extract-dilution analysis
16 (AEDA) (Grosch, 1993) or combined hedonic and response measurements (CHARM)
17 methods (Acree *et al.*, 1984). Dilution analysis, as the name suggests, involves the trained
18 assessors analysing successive dilutions of the sample until no odour is perceived, providing
19 a semi-quantitative measurement useful for profiling. CHARM methods compare only the
20 magnitude of each odour, by recording the concentration when the sensory threshold is
21 exceeded and then when it is no longer detected. Generally these approaches are used for
22 profiling the entire volatile profile of a food and the relative importance of each compound,
23 rather than as quantification methods for taint analysis.
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33 As with all analytical techniques, the use of reference standards with GC-O is important both
34 for matching retention times and odour characteristics (Molyneux and Schieberle, 2007).
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38 A review on GC-olfactometry in aroma analysis was published in 1999 (Feng and Acree,
39 1999) and more recently, Plutowska and Wardencki reviewed the use of GC-O in the analysis
40 and quality assessment of alcoholic beverages (Plutowska and Wardencki, 2008).
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45 *The electronic nose*

46 Digital aroma technology like the 'electronic nose' is designed to mimic the function of
47 sensory panels and can therefore offer an objective method for the detection and
48 measurement of some odours. In most electronic nose systems an array of sensors, with
49 different surface properties, is used, and the volatile compounds are absorbed and desorbed at
50 the surface of the sensors, causing a change in electrical resistance (Arnold and Senter, 1998).
51 The odours are classified based on previous readings. It should be noted that it is the total
52 odour of the sample headspace that is being analysed and individual volatile compounds are
53 not separated as in GC instruments. As the headspace vapour crosses the array of sensors, an
54 odour profile similar to a fingerprinting technique is produced.
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5 The electronic nose has been used to detect tainting compounds in raw and treated portable
6 water (Stuetz, 2007), in ham (Otero *et al.*, 2003) and pork (O'Sullivan *et al.*, 2003) and to
7 monitor lipid oxidation in nuts (Pastorelli *et al.*, 2007). Stuetz, (2007) described a semi-
8 quantitative analysis for a range of tainting compounds in water, although it was noted that
9 the background matrix influenced the response pattern and for any environmental analysis,
10 seasonal variations in matrix background would need to be considered. Cimato *et al.*, (2006)
11 used both SPME-GC-MS and the electronic nose for the analysis of olive oil defects (off-
12 flavours) and Esposto *et al.*, (2006) concluded that discrimination of virgin olive oils was
13 possible using both techniques.
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23 However, Berna *et al.*, (2008) compared a sensor electronic nose (metal oxide) and MS
24 electronic nose with the GC-MS method and concluded that performance of the electronic
25 noses did not approach the sensitivity accuracy or specificity of GC-MS when analysing wine
26 for 4-ethylphenol and 4-ethylguaiacol. The sensors were unable to predict spoilage accurately
27 when a range of wines were analysed due to the variation in other volatile components, even
28 when an additional drying step was used in an attempt to minimise interferences from
29 ethanol. An electronic nose metal oxide sensor device gave good correlation compared to
30 SPME, as a screening tool for monitoring lipid oxidation in nuts (Pastorelli *et al.*, 2007).
31 Applications using the electronic nose for quantitative measurement are limited and follow-
32 up confirmatory analysis is nearly always required. The technique is often seen more as a
33 screening technique to replace olfactory analysis by human sensory panels – which can
34 produce varying results and can be expensive and time-consuming.
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47 **Future developments**

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49 Developments in software for pattern recognition and background subtraction are allowing
50 better profiling of food samples and enable a more rapid comparison of 'good' and 'bad'
51 samples using techniques, such as principal component analysis (Kallithraka *et al.*, 2001;
52 Pigani *et al.*, 2009; Rodríguez-Delgado *et al.*, 2002; Rudnitskaya, 2009). This will allow for
53 more rapid identification of the tainting compound, particularly in samples with very
54 complex volatile profiles containing trace level contamination. Developments in sorptive
55 extraction techniques, such as SBSE and cold fibre SPME, are leading to more rapid methods
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3 that can achieve the necessary sensitivity to determine compounds even with extremely low
4 sensory thresholds in the presence of large matrix components.
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9 Other extraction techniques, still under development include droplet or dispersive extraction,
10 which to date have only been applied to aqueous solutions (Rezaee *et al.*, 2006; Yangcheng *et*
11 *al.*, 2006; Zhou, *et al.*, 2008). The use of GC x GC (d'Acampora Zellner *et al.*, 2007) and
12 TOF-MS for profiling is likely to lead to the use of such techniques in taint analysis.
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14 However, currently quantitation down to low levels is a problem and suitable software is not
15 available for many applications. As with most screening or multi-residue methods, where
16 selective sample preparation cannot be used for targeted analysis, instrumentation and
17 adequate data processing must be relied upon to provide the unequivocal identification and
18 sensitivity that is required for accurate quantitation.
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27 **Conclusions**

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29 The prevention of taints and off-flavours in foods by controlling processes, packaging and
30 storage conditions is paramount to ensure food quality and potentially food safety. Risk
31 management and reduction measures should be considered for areas where potential taints
32 can occur.
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39 As this paper illustrates, for the investigation and analysis of taints and off-flavours a flexible
40 approach needs to be taken. Each case must be viewed individually, gathering as much
41 background information as possible. The analytical methods employed will depend on many
42 factors, including instrument availability and analyst experience. If targeted analysis can be
43 performed then several techniques may be suitable, but for unknown taints, the choice is
44 more limited. An example approach is given in Figure 2, which illustrates some of the steps
45 involved in deciding which method is fit for purpose. An experienced taint analyst may
46 follow a more targeted approach to provide a more rapid response, as it is frequently critical
47 to the food industry to obtain early identification of a tainting compound.
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57 When a taint or off-flavour is detected, accurate methods of analysis are required to rapidly
58 identify and quantify the compounds responsible to enable consumer safety risk assessments
59 and help identify the origins of the taint. Current extraction methods for taint analysis fall into
60 two categories. Those that are more generic and are therefore useful for screening but may

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3 not have the required sensitivity for some analytes, and those developed for more targeted
4 analysis that will only be useful for certain known compounds. The rate limiting step is
5 sample extraction and many of the more generic techniques based on liquid extraction are
6 time consuming and still require a solvent concentration step.
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12 Direct static headspace can often lack the sensitivity required or if dynamic systems are used
13 then matrix effects can be a problem with some foodstuffs. Headspace techniques that
14 incorporate a selective concentration step, such as SPME are increasingly being used, but
15 may not be applicable to all analytes. SBSE and HSSE offer some selectivity and high
16 concentration factors, and have been applied to specific tainting compounds.
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23 Once a taint has been detected then the course of action will depend on several factors, such
24 as whether the product is already on the market, the number of batches affected and whether
25 the contamination poses a potential risk to human health. If there is a consumer safety risk
26 then a public recall must be considered, but even where the tainting compound represents no
27 risk to consumers, a silent recall may be undertaken to minimise brand damage or perception
28 of poor quality.
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37 This review highlights the need for a rapid universal method of extraction for determination
38 of taints in food to enable detection of compounds at trace and ultra-trace levels in foods (sub
39 ng g⁻¹).
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7 Figure 1: Variations in taste thresholds

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9 (reproduced with kind permission of Springer Science and Business Media, from
10 Food taints and off-flavours Ed M J Saxby (1993)
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14 Figure 2: An example analytical approach for investigation of food taints.
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19 Figure 3: Chromatogram obtained by HS–SPME–GC–MS for the VOC determination
20 in a cork stopper in full scan and Selected Ion Storage (SIS) mode.
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22 (Reproduced from Ezquerro and Tena, (2005) *J. Chromatogr. A* 1068: 201-208, with
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28 Figure 4: Comparison of chromatogram of chlorophenols subjected to SBSE with in
29 situ derivatization with that subjected to SBSE without derivatization. (10 ml of
30 chlorophenol standard solution (10 ng ml^{-1}) stirring for 60 min at $25 \text{ }^\circ\text{C}$).
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40 Figure 5: (a) Spiked natural cork stopper chromatogram analysed by headspace stir
41 bar sorptive extraction (HS-SBSE) with gas chromatography–mass spectrometry.
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43 (b) Overlaid selected ion chromatograms of the six target compounds at 25 ng g^{-1} in
44 spiked cork stoppers; internal standard (I.S.); (1) 2,4,6-trichloroanisole (TCA); (2)
45 2,3,4,6-tetrachloroanisole (TeCA); (3) 2,4,6-tribromoanisole (TBA); (4) 2,4,6-
46 trichlorophenol (TCP); (5) pentachloroanisole (PCA); (6) 2,3,4,6-tetrachlorophenol
47 (TeCP).
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Table 1: Sensory threshold values for some common tainting compounds
 [Main source – “Index of chemical taints” Leatherhead Foods RA 1992, and “Food Taints and off flavours” Saxby (Ed)]

Compound	Taint descriptor	Odour Threshold $\mu\text{g/l}$ (ppb)	Taste Threshold $\mu\text{g/l}$ (ppb)
4-cresol	Phenolic, horse manure	200	2
2-bromophenol	Disinfectant, phenolic	0.1 (18 $\mu\text{g/kg}$ reported in oil)	0.03 (2 $\mu\text{g/kg}$ reported in prawns)
2-chlorophenol	Disinfectant, medicinal	3 $\mu\text{g/l}$	0.1 $\mu\text{g/l}$ (2 $\mu\text{g/kg}$ in milk)
Chlorophenol		1.2ppm	0.006ppm
6-chloro-o-cresol	Disinfectant, medicinal, TCP		0.05 $\mu\text{g/kg}$ in blancmange, 0.03 $\mu\text{g/l}$ in tea, 2 $\mu\text{g/kg}$ in margarine
2,6-dibromophenol	Iodoform		0.0005 (0.06 $\mu\text{g/kg}$ reported in prawns)
2,4-dichlorophenol	Phenolic, chemical	200 ng g^{-1}	0.3
2,6-dichlorophenol	Phenolic, chemical	3	0.2, (0.5 $\mu\text{g/l}$ in beer)
2,4,6-trichlorophenol	Disinfectant	300 $\mu\text{g/l}$	2 $\mu\text{g/l}$
Dimethyl sulphide	Cabbage, sweet, repulsive	0.33	6 $\mu\text{g/l}$ in milk, 60 $\mu\text{g/l}$ in beer
Decanoic acid	soapy		0.02%
Decanal	green	0.1	7
Ethyl acrylate	Acrid		67 $\mu\text{g/l}$
Geosmin	Earthy, musty, muddy	0.02 (0.007)	0.05 $\mu\text{g/l}$ (6 $\mu\text{g/kg}$ in fish)
2,4,6-tribromophenol	Iodoform		0.6
Guaiacol	Smoky, phenolic, medicinal	21 (70 $\mu\text{g/l}$ in paraffin oil, 20 $\mu\text{g/l}$ in wine)	13 $\mu\text{g/l}$ (50, 21)
Hexanal	Rancid	0.19-30 ng g^{-1} (ppb)	0.2-10
Indole	Faecal	0.3 mg/kg (ppm)	0.5 mg/kg (ppm)
Methyl methacrylate	Plastic	0.2 mg/kg (ppm) in air	
2,4,6-trichlorophenol	Disinfectant	300	2
1-octen-3-ol	Mouldy, musty, metallic	10 $\mu\text{g/l}$	1 $\mu\text{g/l}$
Oct-1-en-3-one	Oily, green, metallic, mushroom, apple, cardboard	0.09 $\mu\text{g/l}$ 80 $\mu\text{g/l}$ in oil	1 $\mu\text{g/kg}$ in butterfat, 10 $\mu\text{g/kg}$ in skimmed milk
Cis-Oct-2-enal	Sour, rancid	3 $\mu\text{g/l}$	83 $\mu\text{g/l}$ in oil/water emulsion
2,4-dichloroanisole	Musty, sweet, fruity, scented	0.4	-
2,6-dichloroanisole	Musty, medicinal, phenolic	0.04	
2,4,6-tribromoanisole	Musty	0.000 008 (8 pg/l (ppq))	
2,4,6-trichloroanisole	Musty, earthy	0.000 03 $\mu\text{g/l}$ (0.03 ng/l (ppt) in water	0.02 $\mu\text{g/l}$ in water 0.01 $\mu\text{g/l}$ in wine 2.4 $\mu\text{g/kg}$ in egg yolk
pentachloroanisole	Musty, earthy	4 $\mu\text{g/l}$	2.8 mg/kg (ppm) in egg yolk
Styrene	Hydrocarbon, Plastic, Acrid	0.7 mg/kg in water, 50 $\mu\text{g/l}$ in air	37 $\mu\text{g/l}$ (or 22 ppb, 0.022 ppm) in water 0.2 tea 0.5 yoghurt, 1.2 whole milk, 5 $\mu\text{g/kg}$ (ppb) in sour cream
Skatole	Faecal, animal, nauseating	10 $\mu\text{g/l}$ in water 0.0012 mg/l in air	50 $\mu\text{g/l}$ in water
Trans-1,3-pentadiene	Plastic, paint, paraffin, kerosene	2.5 ml/l in 10% brine	4 mg/kg in cheese
Terpineol	Musty, Piney		2 mg/l (ppm) in orange juice
2-pentylfuran	Beany, rancid-greasy	6 $\mu\text{g/l}$	1 mg/l (ppm)

Note: Thresholds in water unless stated otherwise (Values as reported in the literature, therefore, more than one for some compounds).

Table 2 – Examples of Taints and their possible origins.

(Main source – “Index of chemical taints” Leatherhead Foods RA 1992, and “Taints and off flavours in food” Baigrie (ed))

Odour descriptor	Compounds	Possible origin
Acrid	Acrolein	Formed microbiologically in distillery mashes.
Acrid/plastic	Ethyl and methyl acrylate	Industrial chemicals.
	Methyl methacrylate	Industrial chemical.
Almond	Heptane-2-one	Oxidation of oils (rancid coconut), light-induced oxidation of fats.
	1,4-Dichlorobenzene	Drain cleaners and moth-proofing agents.
	Benzaldehyde	Packaging – reaction by product
Apple	Damascenone	Microbiological - produced by <i>Actinomyces</i> . ?
	Oct-1-en-3-one	Autooxidation of fats and sometimes found in plastics containing diisooctyl phthalate.
	Acetaldehyde	Over production in milk cultures or yoghurt (also described as green) Also can be a degradation product of PET packaging.
Brine/seaside	Bromocresol (2-bromo-4-methylphenol)	Associated with corresponding bromophenol/anisole.
	Dbromocresol (2,6-dibromo-4-methylphenol)	Associated with corresponding bromophenol/anisole.
Cabbage	Dimethyl sulphide	Reactions with methionine and the cause of off flavour in beer.
	Diphenyl sulphide	Photoinitiator for cationic inks.
Cardboard	2,4-Nonadienal	Autooxidation of oils and fats.
	Oct-1-en-3-one	Autooxidation of fats and sometimes found in plastics containing diisooctyl phthalate.
	Hexanal	Lipid degradation associated with paper (decarboxylation and oxidation of lignin).
Catty/ cats urine	4-Mercapto-4-methylpentan-2-one	Reaction of hydrogen sulphide (in foods) with mesityl oxide (solvent impurity found in some paints/varnishes).
Chemical	Chlorobenzene	Used as an antifungal agent in some glues.
	2,4- or 2,6-Dichlorophenol	Fungicides, biocides and herbicide intermediates. Found in packaging - wood pulp that has been treated and cardboard.
Cucumber	trans-2-cis-6-Nonadienal	Algae in water.
Disinfectant	6-Chloro-o-cresol (2-methyl-6-chlorophenol)	Disinfectants and drain cleaners or impurity in some herbicides.
	2-Chlorophenol	Chlorination of phenol (associated with 2-methyl-6-chlorophenol) . E.g. from water containing phenol (eg from peat soil) that is chlorinated
	2,3-Dichlorophenol	Fungicides, biocides and herbicide intermediates. Or from water containing phenol (eg from peat soil) that is chlorinated
	2,4,6-Trichlorophenol	Found in packaging - wood pulp that has been bleached and cardboard and polyvinyl acetate glues.

	2-Bromophenol	Present in algae (major portion of the diet of prawns). Also can be formed by reactions – e.g. has been found as a taint in fish that has been bleached with hydrogen peroxide, treated with brine (containing a bromide impurity) in the presence of trace levels of phenol (in oak storage barrels).
Drains	2,6-Dimethyl-3-methoxypyrazine	Produced by certain bacteria.
Earthy	Geosmin (trans-1, 10-Dimethyl-trans-9-decalol)	Microorganisms – particularly bacteria. Produced by actinomycetes ? blue-green algae and cyanobacteria (can contaminate water supplies or soil).
	Pentachloroanisole	Microbial methylation of the corresponding chlorophenols – particularly in wood/pallets treated with a chlorophenol preservative.
	2,3,4,6-Tetrachloroanisole	Microbial methylation of the corresponding chlorophenol – particularly in wood/pallets treated with a chlorophenol preservative or in corks treated with chlorophenol. Can be formed by degradation of pentachloroanisole.
	2,3,6- and 2,4,6-Trichloroanisole	Microbial methylation of the corresponding chlorophenols – particularly in wood/pallets treated with a chlorophenol preservative or in corks treated with chlorophenol.
	2-Methylisoborneol	Water contaminated with actinomycetes ? or cyanobacteria.
Faecal	Indole (2,3-benzopyrrole)	Rotting potatoes and also associated with boar taint in male pigs.
	Skatole (3-methylindole)	Bacterial metabolite of amino acids, found in mammalian faeces and has been associated with taint in meat from male pigs.
Fruity	Acetaldehyde	Over production in milk cultures or yoghurt (also described as green). Also can be a degradation product of PET packaging.
	2,4-Dichloroanisole	Microbial methylation of 2,4-dichlorophenol.
	Ethyl butanoate, ethyl hexanoate, ethyl octanoate	Microorganisms in foods including dairy, fish and meat.
Geranium	cis-Octa-1,5-dien-3-one	Autooxidation of butterfat.
	Benzophenone	Packaging – photo-initiator in UV inks and varnishes.
Green	Decanal	Autooxidation of fats.
Iodine	2-Bromophenol	Present in algae (major portion of the diet of prawns). Also can be formed by reactions – e.g. has been found as a taint in fish that has been bleached with hydrogen peroxide, treated with brine (containing a bromide impurity) in the presence of trace levels of phenol (in oak storage barrels).
Iodoform	2,6-Dibromophenol	Aquatic environment - seafood, also can be present in some fungicides, biocides and herbicide intermediates (wood treatment).
	2,4,6-Tribromophenol	Seafood, or reaction of biocide/bromination of phenol.
Kerosene	1,3-Pentadiene	Degradation of sorbate by the <i>Penicillium</i> species (products treated with sorbic acid as a mould inhibitor).
Medicinal	2-Chlorophenol	Chlorination of phenol (associated with 2-methyl-6-chlorophenol). e.g from water

		containing phenol (eg from peat soil) that is chlorinated
	6-Chloro-o-cresol	Disinfectants and drain cleaners or impurity in some herbicides.
	2,6-Dichloroanisole	Microbial methylation of corresponding chlorophenol.
	Guaiacol	Microbiological degradation of vanillin/degradation product of lignin.
	2-Iodo-4-cresol	Reaction of p-cresol (used in some flavours) with iodised salt.
	Dichlorobenzene	Disinfectants, drain cleaner, fumigants.
Metallic	1-Octen-3-ol	Fungal growth, autooxidation of fats, natural component of clover and fresh mushrooms.
	Oct-1-en-3-one	Autooxidation of fats and sometimes found in plastics containing diisooctyl phthalate.
	cis-Octa-1,5-dien-3-one	Autooxidation of butterfat.
Mouldy	1-Octen-3-ol	Fungal growth, autooxidation of fats, natural component of clover and fresh mushrooms.
	Geosmin (trans-1, 10-dimethyl-trans-9-decalol)	Produced by actinomycetes and blue-green algae (can contaminate water supplies or soil).
Musty	Pentachlorophenol	Used as a biocide in wood treatment and adhesive glues
	Pentachloroanisole	Microbial methylation of the corresponding chlorophenols – particularly in wood/pallets treated with a chlorophenol preservative.
	2,3,4,6-Tetrachloroanisole	Microbial methylation of the corresponding chlorophenol – particularly in wood/pallets treated with a chlorophenol preservative or in corks treated with chlorophenol. Can be formed by degradation of pentachloroanisole.
	2,3,6- and 2,4,6-Trichloroanisole	Microbial methylation of the corresponding chlorophenols – particularly in wood/pallets treated with a chlorophenol preservative or in corks treated with chlorophenol.
	2,4- and 2,6-Dichloroanisole	Microbial methylation of corresponding chlorophenol.
	Geosmin (trans-1, 10-dimethyl-trans-9-decalol)	Produced by actinomycetes and blue-green algae (can contaminate water supplies or soil).
	2-Methylisoborneol	Water contaminated with actinomycetes or cyanobacteria.
	2,4,6-Tribromoanisole	Reaction of some biocides with phenol, followed by microbial methylation to form the anisole.
	1-Octen-3-ol	Fungal growth, autooxidation of fats, natural component of clover and fresh mushrooms.
	Octa-1,3-diene	Metabolite of <i>Anabaena oscillarioides</i> and autooxidation of fats.
	α -Terpineol	Disinfectants.
	4,4,6-Trimethyl-1,3-dioxan	Reaction of 2-methyl-2,4-pentanediol in packaging film with formaldehyde during storage.
	Trimethylanisole	Contaminant in rubber seals.
Paint	Heptane-2-one	Oxidation of oils and fats.
	trans,trans-Hepta-2,4-dienal	Autooxidation of fats.

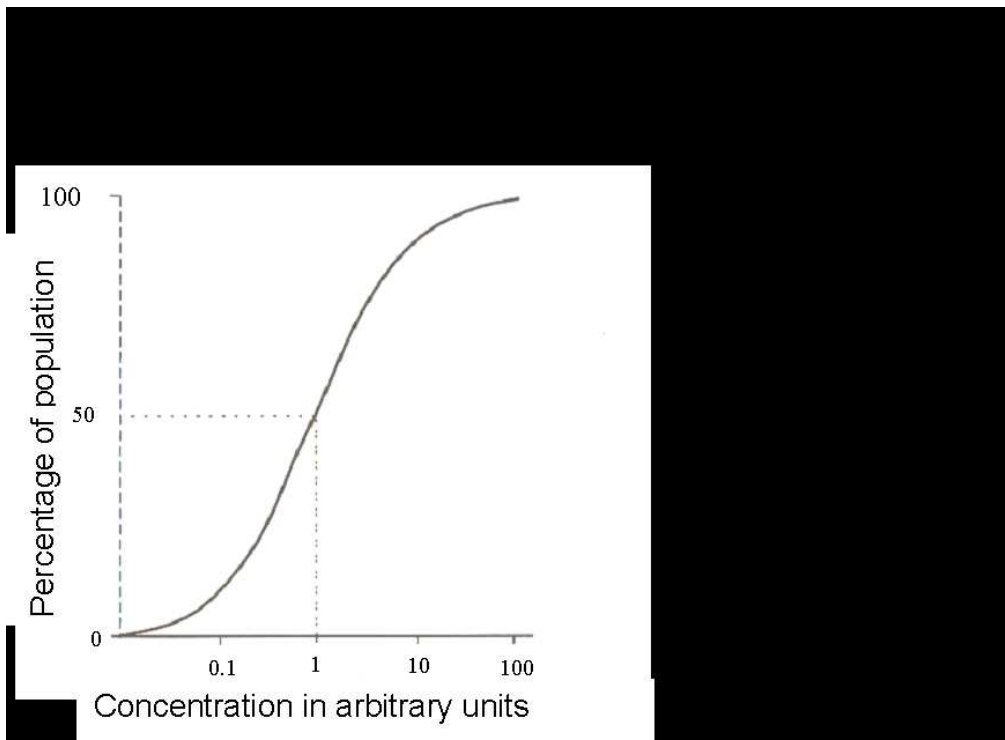
	trans-1,3-Pentadiene	Degradation of sorbate by the <i>Penicillium</i> species (products treated with sorbic acid as a mould inhibitor).
Paraffin	trans-1,3-Pentadiene	Degradation of sorbate by the <i>Penicillium</i> species (products treated with sorbic acid as a mould inhibitor).
Pear-like	Acetaldehyde	Degradation product sometimes formed during processing of PET packaging.
	Butyl acetate	Printing inks.
Petroleum	Dimethylsulphide	Formed from sulphur containing precursors in the aquatic environment such as plankton.
	Xylenes	Residual solvents from varnishes/lacquers – can migrate through packaging.
Phenolic	2-Bromophenol	Present in algae (major portion of the diet of prawns). Also can be formed by reactions – e.g. has been found as a taint in fish that has bleached with hydrogen peroxide, treated with brine (containing a bromide impurity) in the presence of trace levels of phenol (in oak storage barrels).
	p-Cresol (4-methylphenol)	Microbiological degradation.
	2,4- or 2,6-Dichlorophenol	Impurities in herbicides and in packaging from bleaching of wood pulp. Or from water containing phenol (eg from peat soil) that is chlorinated.
	2,6-Dichloroanisole	Microbial methylation of corresponding chlorophenol.
	Guaiacol	Microbiological degradation of vanillin/degradation product of lignin.
Piney	α -Terpineol	Disinfectants.
Plastic	Styrene	Migration from polystyrene containers or formed from cinnamaldehyde (in cinnamon).
	Benzothiazole	Butyl rubbers.
	trans-1,3-Pentadiene	Degradation of sorbate by the <i>Penicillium</i> species (products treated with sorbic acid as a mould inhibitor).
Rancid	cis-Oct-2-enal	Metabolite of <i>Anabaena oscillarioides</i> and autooxidation of fats.
Smoky	Guaiacol	Microbiological degradation of vanillin/degradation product of lignin.
	4-Vinylguaiacol	Degradation product in orange juice.
Soapy	Decanoic acid	Lipolysis of lipids (palm kernel oil, coconut oil).
	Lauric acid (dodecanoic acid)	Lipolysis of lauryl glycerides (palm kernel oil, coconut oil, butter).
Sulphury	Methanethiol (methyl mercaptan)	Degradation of sulphur-containing proteins.
Sweet	2,4-Dichloroanisole	Microbial methylation of 2,4-dichlorophenol.
	Cyclohexane	Screen-printing solvent.
TCP	6-Chloro-o-cresol	Disinfectants and drain cleaners or impurity in some herbicides
Turpentine	para-Cymene (1-isopropyl-4-methylbenzene)	Degradation product of lemon oil and limonene and γ -terpinene in soft drinks.
	Nonan-2-one	Rancid coconut.
Urine	5 α -Androst-16-en-3-one	Meat from uncastrated male pigs.

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Woody	1,4-Dichlorobenzene	Drain cleaners and also used in moth-proofing agents.
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For Peer Review Only

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146x107mm (150 x 150 DPI)

View Only

Taint or off-flavour in food reported

Sensory descriptors
(consumer vs panel?)

Background information provided
(suspected compounds /causes of taint)

Is there sufficient information to predict the identity of the compound(s) responsible for the taint?

No

Yes

Screening "Generic" method required as first step.
GC-O, Headspace GC-MS (scan acquisition)
comparing control and suspect/complaint sample.
Additional peaks identified in suspect sample?

Targeted extraction and analysis.
Method depends on sensitivity required i.e. level of
sensory threshold / Predicted levels in samples?
Low (ppb/ ppt): SDE / SPME / SBSE
High (ppb/ ppm): Headspace /SPME /solvent
extraction

Yes

No

Tentative
identification of peaks
using spectral library
(+ sensory)

Run with
alternative GC
column.
Additional peaks?

Yes

No

Run reference
standard and confirm
retention time and
spectra (SIM).
Perform quantitation
(consider standard
additions depending
on matrix)

Yes

More sensitive
method required,
SDE with GC-MS
and/or GC-HRMS
(scan)
Additional peaks?

No

No

Yes

Taint compound
tentatively identified?

Use more sensitive
method or follow
screening procedure

Run reference standard
and confirm retention
time and spectra (SIM).
Perform quantitation
(consider standard
additions depending on
matrix)

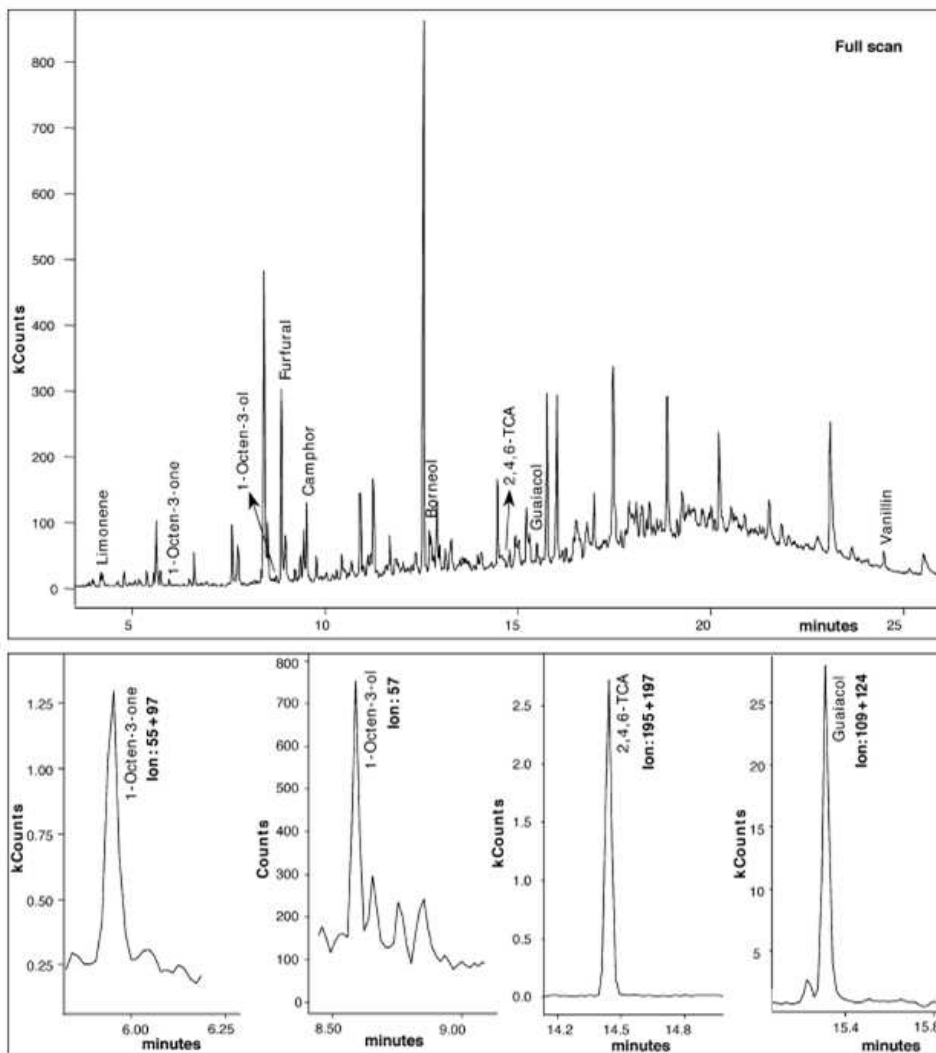
Re-interrogate
background information
(sensory data/suspected
compounds).
Follow targeted analysis
approach for possible
'known' tainting
compounds.

Check compounds identified and levels
match sensory descriptors

Risk assessment

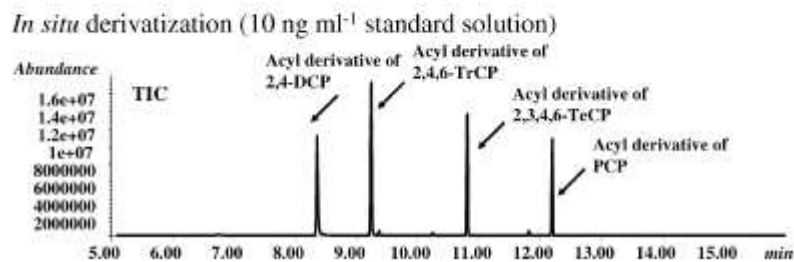
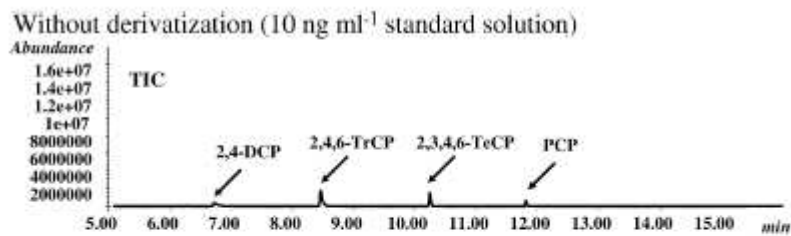
Risk management/ reduction
(Follow up root cause, possible further analysis)

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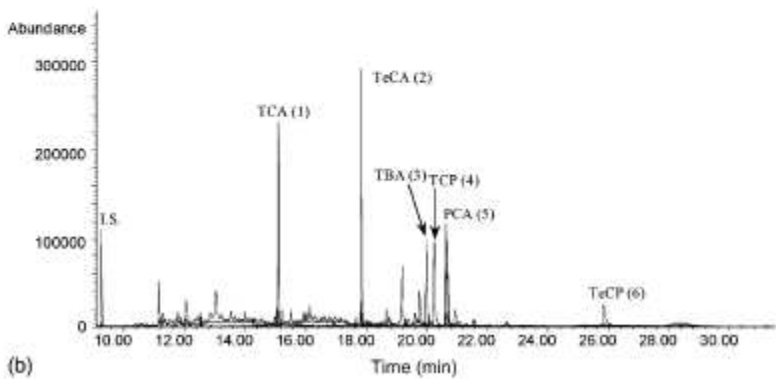
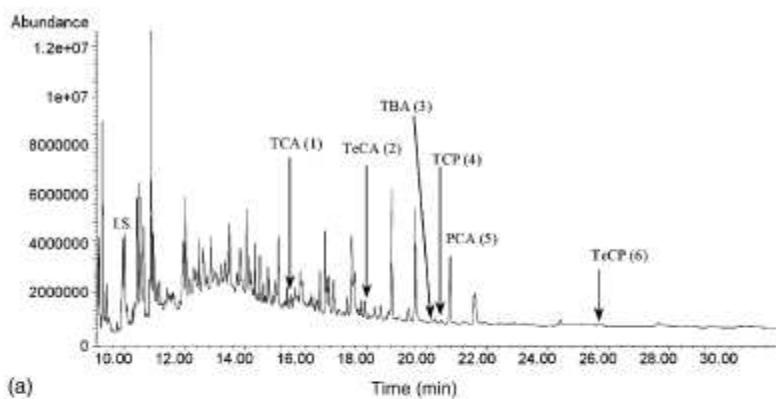
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117x72mm (96 x 96 DPI)

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