Report FD 10/05

FSA PROJECT C01048

Determination of phthalates in foods and establishing methodology to distinguish their source

PROJECT INFORMATION

Report Number:	FD 10/05
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Date:	January 2012
Project title:	Determination of phthalates in foods and establishing methodology to distinguish their source
Sponsor:	Food Standards Agency Aviation House 125 Kingsway London WC2B 6NH
Sponsor’s Project Number:	C01048
Fera Project Number:	R6NN
Fera File Reference:	FLN 8904
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Distribution:	1. D. Mortimer (FSA) 2. Fera Library

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SUMMARY

This report describes the development and validation of methodology for the determination of 17 phthalate diesters (dimethyl phthalate (DMP), diethyl phthalate (DEP), diisopropyl phthalate (DiPP), diallyl phthalate (DAP), diisobutyl phthalate (DiBP), di-n-butyl phthalate (DBP), di-n-pentyl phthalate (DPP), di-n-hexyl phthalate (DHexP), benzyl butyl phthalate (BBP), dicyclohexyl phthalate (DCHP), di-(2-ethylhexyl) phthalate (DEHP), di-n-heptyl phthalate (DHepP), di-n-octyl phthalate (DOP), n-octyl-n-decyl phthalate (ODP), diisononyl phthalate (DiNP), diisodecyl phthalate (DiDP) and di-n-decyl phthalate (DDP)), 9 phthalate monoesters (monoisopropyl phthalate, monoisobutyl phthalate, mono-n-butyl phthalate, monobenzyl phthalate, monocyclohexyl phthalate, mono-n-pentyl phthalate, mono-2-ethylhexyl phthalate, mono-n-octyl phthalate and monoisononyl phthalate), phthalic acid and total phthalates in foods. The methods have been validated in-house and their broad applicability demonstrated by the analysis of high fat, high carbohydrate and high protein foodstuffs as well as combinations of all three major food constituents.

The methods were used to analyse UK total diet study (TDS) samples. The TDS samples comprise twenty food groups: bread, fresh fruit, fruit products, dairy products, oils & fats, milk, nuts, beverages, meat products, offal, green vegetables, eggs, miscellaneous cereals, fish, sugar & preserves, canned vegetables, poultry, carcass meat, other vegetables, and potatoes. In total 119 sub-categories of food are combined into the 20 groups.

- Diethyl phthalate was present in one food group (cereal) at a concentration of 13 µg/kg.
- Benzyl butyl phthalate was present in one food group (bread) at a concentration of 8 µg/kg.
- Diisobutyl phthalate was present in four food groups (bread, oils & fats, nuts and cereal) at respective concentrations of 17, 41, 49 and 81 µg/kg.
- Di-n-butyl phthalate was present in seven food groups (bread, oils & fats, nuts, meat products, cereal, fish and carcass meat) at respective concentrations of 16, 16, 28, 15, 14, 9 and 6 µg/kg.
- Di-(2-ethylhexyl) phthalate was present in eleven food groups (bread, dairy, oils & fats, nuts, meat products, cereal, fish, sugar & preserves, poultry, carcass meat and other vegetables) at respective concentrations of 125, 141, 106, 112, 329, 104, 789, 115, 322, 90 and 35 µg/kg.

After DEHP, the most prevalent phthalate was phthalic acid itself which was present in 11 food groups (bread, oils & fats, milk, nuts, green vegetables, eggs, sugar & preserves., canned vegetables, carcass meat, other vegetables and potatoes) at 27, 36, 66, 100, 89, 35, 110, 84, 36, 99 and 84 µg/kg, respectively.

The finding that the two most abundant diesters were DBP and DEHP was also reflected in the results for monoesters. Low levels of mono-n-butyl phthalate (MBP) and mono-(2-ethylhexyl) phthalate (MEHP) were detected in several of the TDS animal-based food groups. If post mortem metabolism can be discounted, this indicates exposure of the growing animals to the parent phthalate diesters had occurred as well as any possible contribution from e.g. processing and packaging of the foodstuffs.

To follow-up the TDS work, two hundred and sixty one retail foodstuffs were analysed for fifteen of the phthalate diester compounds (DMP, DEP, DiPP, DAP, DiBP, DBP, DPP, DHexP, BBP, DCHP, DEHP, DOP, DiNP, DiDP and DDP). The samples were categorised by the Food Standards Agency broadly along the lines of the TDS samples

into categories of: bread products (8 samples), dairy products - including milk (15 samples), oils & fats (24 samples), meat and meat products - including poultry (69 samples), liver – of fish and meat (12 samples), eggs (7 samples), miscellaneous cereal products (26 samples), fish and fish products (74 samples), infant food (10 samples) and infant formula (16 samples).

Phthalate diesters were present in 77 samples. DEHP was the most frequently detected (66 samples) however the highest levels were for the isomeric mixture DiNP in some samples. The tables below give an overview of the incidence per food category as well as the concentrations detected. The data given in these tables are for confirmed residues only. A response was observed in the GC-MS chromatograms of other samples and/or for other phthalates not listed in the tables. However in those cases the confirmation criteria were not met and as such it could not be confirmed unequivocally that the response was due to the presence of a phthalate diester.

Of the samples tested 75% of the bread products contained one or more phthalate diester, 53% of the dairy products, 33% of the oils & fats samples, 29% of the meat and meat products, 50% of the miscellaneous cereal products, 23% of the fish and fish products, 20% of the infant food samples and 19% of the infant formula samples.

The tables for the retail samples also include the diester results for the corresponding TDS food groups. A very detailed comparison is not appropriate because the TDS samples are prepared (e.g. cooked) to make ready-to-eat whereas the retail samples were simply unpacked and analysed. Also, the TDS food groups are made up of individual samples (e.g. beef, lamb, pork etc into the carcass meat group) in strict proportion to their importance to the national diet whereas the retail sampling was more ad hoc. Nevertheless, it is noteworthy that, with a few individual exceptions such as for food groups where the incidence and the concentrations in retail samples were low and so the phthalate would be averaged to below the detection limit in the TDS composites), the same phthalates were found in the retail samples as were found in the TDS samples.

Additional work was carried out to determine the source of the phthalates found in a small number of packaged foodstuffs. Phthalates may be present in foods as a result of migration from food contact materials and articles. Studies carried out in this project confirmed that phthalates in foods could be derived from migration from this source.

Bread products (8 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)	
		DiBP	DEHP
S09-017242	Granary loaf	67	
S09-017259	White bread rolls	17	92
S09-017293	Wholemeal pitta		94
S09-017307	Brown bread		
S09-017346	Rye wholemeal bread	108	
S09-017347	Milk brioche rolls		
S09-017382	Organic wholemeal bread		59
S09-017389	Sliced white plain bread		115
	incidence	**3**	**4**
	mean of all positive samples	**64**	**90**
	mean of all samples (non-detects set to 0)	**24**	**45**

	TDS Group Bread Also: DBP (16); BBP (8)	17	125

Dairy products including milk (15 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)			
		DEP	**DiBP**	**DEHP**	**DiNP**
S09-017245	Jersey milk			109	
S09-017246	British double cream			328	
S09-017250	Blue Stilton			496	3055
S09-017270	Cornish clotted cream		186	690	
S09-017271	Traditional Normandy camembert				
S09-017279	Fresh filtered whole milk				
S09-017280	Greek style natural probiotic yoghurt			78	
S09-017318	Lemon curd yogurt			102	
S09-017334	Grated Emmental	272		366	
S09-017348	Brie				
S09-017379	Extra thick real cream - UHT			222	
S09-017437	Organic whole milk				
S09-017454	Organic whole milk				
S09-017465	Welsh farm assured whole milk				
S09-017482	Whole milk				
	incidence	**1**	**1**	**8**	**1**
	mean of all positive samples	**272**	**186**	**299**	**3055**
	mean of all samples (non-detects set to 0)	**18**	**12**	**159**	**204**

	TDS Group Dairy	-	-	141	-
	TDS Group Milk	-	-	-	-

Oils & fats (24 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)				
		DiBP	DBP	BBP	DEHP	DiNP
S09-017247	Lard		141		119	
S09-017269	Dairy butter				347	1500
S09-017272	Goose fat					
S09-017276	Organic extra virgin olive oil	49	57	2084	6447	
S09-017286	Olive pomace oil blend				434	
S09-017290	Unsalted butter				2592	
S09-017295	Grapeseed oil					
S09-017298	Vegetable fat spread				160	
S09-017315	Pure vegetable oil					
S09-017330	Refined vegetable fat					
S09-017343	Beef dripping					
S09-017357	Sunflower oil					
S09-017404	Toasted sesame oil					
S09-017413	Groundnut oil				240	
S09-017448	Lard					
S09-017459	Extra virgin olive oil					
S09-017462	Extra virgin olive oil					
S09-017463	Lard					
S09-017474	Salted butter					
S09-017477	Lard					
S09-017481	Farmhouse butter					
S09-017484	Blended olive pomace oil					
S09-017489	Olive pomace oil					
S09-017497	Butter ghee				960	
	incidence	**1**	**2**	**1**	**8**	**1**
	mean of all positive samples	**49**	**99**	**2084**	**1412**	**1500**
	mean of all samples (non-detects set to 0)	**2**	**8**	**87**	**471**	**63**

	TDS Group Oils & Fats	41	16	-	106	-

Meat and meat products including poultry (69 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)		
		DBP	DEHP	DiNP
S09-017241	Breast of lamb			
S09-017248	British Wiltshire cured thick cut gammon steak			
S09-017249	Lasagne			
S09-017251	British lean beef steak mince		256	
S09-017261	Shin of beef			
S09-017263	Diced lamb			
S09-017264	Boneless chicken breasts			
S09-017278	Lasagne			
S09-017289	Rosemary & garlic lamb chipolatas		163	636
S09-017291	Large roast chicken - whole		197	
S09-017300	Neck cutlets			
S09-017301	Chicken thighs - bone-in / skin on		65	1820
S09-017302	Beef topside			
S09-017303	Pork chops - bone-in / skin on			
S09-017305	Welsh breakfast sausage		166	
S09-017311	Lamb mince		190	
S09-017312	Turkey breast roast			
S09-017317	Diced chicken			
S09-017320	Free range chicken		32	
S09-017321	Rolled pork shoulder joint			
S09-017322	Beer & treacle back bacon			
S09-017328	Lorne sausage		71	
S09-017329	Smoked ham shank		114	
S09-017332	Half spring chicken			
S09-017338	Halal lamb carcase pieces			
S09-017339	Fresh British pork leg joint			
S09-017349	Sirloin steaks			
S09-017350	Pork sausage			
S09-017351	Lamb loin chops			
S09-017359	Turkey breast steaks			
S09-017362	Cunmbrian lamb hotpot			
S09-017370	Belly of pork (rind on)			
S09-017374	Sausages			
S09-017375	Prime gammon joint - unsmoked			
S09-017377	Spare rib of pork joint			
S09-017386	Turkey		56	
S09-017387	Turkey breast butter basted		60	
S09-017390	Beef sausages			

Meat and meat products including poultry (continued)

LIMS number	Sample description	Phthalate concentration (µg/kg)		
		DBP	DEHP	DiNP
S09-017392	Garnished turkey drumsticks	33	204	
S09-017393	Eye roast joint			
S09-017394	Frying steak			
S09-017396	Boneless pork chops			
S09-017397	Boneless stuffed chicken			
S09-017399	Streaky bacon			
S09-017408	Halal bone-in chicken thigh pieces			
S09-017409	British pork mince		47	
S09-017410	Organic bone-in lamb shoulder roast		238	
S09-017426	Breaded chicken pieces		76	
S09-017433	Free range belly pork			
S09-017439	British turkey mince			
S09-017449	Pork sausages			
S09-017450	English minced lamb			
S09-017451	Turkey leg			
S09-017452	Boneless leg of pork			
S09-017457	Organic British free range chicken thigh fillets			
S09-017458	British turkey lean mince			
S09-017460	Classic pork sausage - 16			
S09-017461	New Zealand farm assured lamb mince			
S09-017464	Free range turkey breast mince		127	
S09-017470	Minced lamb			
S09-017471	Minced beef			
S09-017472	Boneless leg of pork		20	
S09-017473	Minced beef			
S09-017475	Newmarket sausage			
S09-017478	Free range chicken legs			
S09-017479	Turkey mince		142	
S09-017480	Boneless leg of pork			
S09-017494	Minced beef		74	
S09-017496	British turkey drumsticks		74	
	incidence	**1**	**20**	**2**
	mean of all positive samples	**33**	**119**	**1228**
	mean of all samples (non-detects set to 0)	**0.5**	**34**	**36**

	TDS Group Carcass Meat	6	90	-
	TDS Group Meat products	15	329	-
	TDS Group Poultry	-	322	-

Liver (of fish and meat species; 12 samples)

No phthalate diesters present. Also, no phthalate diesters in TDS Group Offal

Eggs (7 samples)

No phthalate diesters present. Also, no phthalate diesters in TDS Group Eggs

Miscellaneous cereal products (26 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)			
		DEP	DiBP	DBP	DEHP
S09-017252	Chocolate sponge sandwich				
S09-017255	Cornflakes				
S09-017258	Bourbon creams			60	
S09-017273	Ripple roll				
S09-017275	Ginger biscuits				110
S09-017282	Matzos				
S09-017283	Egg pasta lasagne		30		
S09-017292	Belgian biscuit collection				219
S09-017294	Wheat cereal				49
S09-017297	Lasagne				
S09-017306	Gingerbread ghosties				91
S09-017308	Farmhouse fruit cake		46		245
S09-017345	Microwaveable porage oats - syrup swirl				
S09-017352	Pecan & maple crisp			33	
S09-017353	De-luxe muesli		83		228
S09-017356	Cream crackers				
S09-017363	Rice cereal				
S09-017364	Angel slices				
S09-017366	Cookies				83
S09-017371	Iced fairy cakes				
S09-017380	Wheat cereal				
S09-017402	Raspberry and coconut cakes				365
S09-017407	Honey cereal				
S09-017415	Rye crispbread		38		
S09-017417	Iced cake	44			98
S09-017424	Fruity malt loaf - sliced				
	incidence	**1**	**4**	**2**	**9**
	mean of all positive samples	**44**	**49**	**47**	**165**
	mean of all samples (non-detects set to 0)	**2**	**8**	**4**	**57**
	TDS Group Misc. Cereals	13	81	14	104

Fish and fish products (74 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)			
		DEP	DiBP	DEHP	DiNP
S09-017239	Scottish mussels in shell				
S09-017243	Sea bass fillets				
S09-017244	Salmon fillets				
S09-017260	Conger eel				
S09-017266	Red mullet				
S09-017267	Lemon sole				
S09-017268	Sprats				
S09-017277	Fish pie				
S09-017281	Jumbo salted cod cutlets				
S09-017284	Mackerel fillets in oil			85	
S09-017288	Organic cod fish fingers				
S09-017310	Wild local caught trout fillets				
S09-017313	Swordfish				
S09-017314	Fresh baby hake				
S09-017324	Dressed crab		62		
S09-017325	Sprats (large)			96	
S09-017326	Bloater				
S09-017327	Whole smoked eel				
S09-017333	Tilapia				
S09-017335	Fresh tuna				644
S09-017336	Salmon fillets				
S09-017340	Salmon in dill sauce - frozen steam meal				
S09-017342	Whitebait IQF				
S09-017354	Craster kipper fillets	340		271	11576
S09-017355	Salmon				
S09-017360	Wild Alaskan salmon fillets			314	
S09-017361	Fillets of anchovies				
S09-017372	Wild pink salmon				
S09-017373	Scottish smoked peppered mackerel				
S09-017376	Naturally smoked haddock fillets				
S09-017378	Smoked salmon slices			2176	
S09-017384	Wild Alaskan salmon fillets				
S09-017391	2 River cobbler fillets			49	
S09-017395	Herring fillets			69	
S09-017398	Trout fillet				
S09-017400	Organic Irish oak smoked salmon			850	
S09-017401	Traditionally smoked mackerel fillets				5303
S09-017405	South Pacific sardines in tomato sauce			81	
S09-017420	Halibut				
S09-017421	Trout				

Fish and fish products (continued)

LIMS number	Sample description	Phthalate concentration (µg/kg)			
		DEP	DiBP	DEHP	DiNP
S09-017422	Whitebait				
S09-017423	Trout				
S09-017427	Salmon en croute			204	
S09-017434	Yellowfin tuna				
S09-017435	Mackerel fillets				
S09-017436	Mackerel				
S09-017438	Sardines				
S09-017441	Dressed crab				
S09-017442	Kipper fillets				
S09-017443	Sea bream				
S09-017444	Herrings				
S09-017445	Red sea bream				
S09-017446	Farmed sea bass				
S09-017447	Mackerel				
S09-017453	Whole herring				
S09-017455	Sardines				
S09-017456	Whole kippers			97	
S09-017466	Dressed crab		18		
S09-017467	Mackerel				
S09-017468	IQF whitebait				
S09-017469	Whitebait				
S09-017476	Farmed sea bass fillets				
S09-017483	Fresh sardines				
S09-017485	Salmon steaks				
S09-017486	Sardines – fresh				
S09-017487	Smoked mackerel				
S09-017488	Sardines				
S09-017490	Smoked salmon				
S09-017491	Hebridean white crab meat				
S09-017492	Salmon			45	
S09-017493	Smoked mackerel				
S09-017495	Smoked salmon			52	1999
S09-017498	Wild salmon steaks				
S09-017499	Wild Solway salmon				
	incidence	**1**	**2**	**13**	**4**
	mean of all positive samples	**340**	**40**	**338**	**4881**
	mean of all samples (non-detects set to 0)	**5**	**1**	**59**	**264**

	TDS Group Fish Also: DBP (9)	-	-	789	-

Infant food (10 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)
		DEHP
S09-017254	Fisherman's bake	
S09-017257	Apple, peach & mango fruit pots	
S09-017274	Rice pudding	57
S09-017287	Lasagne	
S09-017367	Cauliflower & broccoli cheese	59
S09-017411	Organic beef stew with potatoes	
S09-017412	Strawberry dairy dessert	
S09-017416	Organic gingerbread men	
S09-017419	Cottage pie	
S09-017431	Biscotti	
	incidence	**2**
	mean of all positive samples	**58**
	mean of all samples (non-detects set to 0)	**12**

	No corresponding TDS Group	

Infant formula (16 samples)

LIMS number	Sample description	Phthalate concentration (µg/kg)	
		DiBP	DEHP
S09-017256	Infant milk - ready to use		
S09-017285	Infant milk - ready to use		
S09-017296	Organic follow-on milk - ready to use		
S09-017316	Infant milk - ready to use		
S09-017331	Infant milk - formula	13	
S09-017365	Growing up milk for toddlers - ready to use		
S09-017381	Infant milk - formula		
S09-017383	Goat milk nutrition - formula		96
S09-017385	Soya infant formula		
S09-017403	Soya infant formula		
S09-017406	Infant milk - formula		
S09-017414	Infant milk - formula		
S09-017418	Organic infant milk - formula		125
S09-017425	Follow-on milk - ready to use		
S09-017429	Follow-on milk - ready to use		
S09-017430	Infant milk - formula		
	incidence	**1**	**2**
	mean of all positive samples	**13**	**111**
	mean of all samples (non-detects set to 0)	**1**	**14**

	No corresponding TDS Group		

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ABBREVIATIONS

BBP	Benzyl butyl phthalate
DAP	Diallyl phthalate
DBP	Di-n-butyl phthalate
DCHP	Dicyclohexyl phthalate
DDP	Di-n-decyl phthalate
DEHP	Di-(2-ethylhexyl) phthalate
DEP	Diethyl phthalate
DHepP	Di-n-heptyl phthalate
DHexP	Di-n-hexyl phthalate
DiBP	Diisobutyl phthalate
DiDP	Diisodecyl phthalate
DiNP	Diisononyl phthalate
DiPP	Diisopropyl phthalate
DMP	Dimethyl phthalate
DOP	Di-n-octyl phthalate
DPP	Di-n-pentyl phthalate
EFSA	European Food Safety Authority
FSA	Food Standards Agency
GC	Gas chromatography
LC	Liquid chromatogtaphy
LOD	Limit of detection
LOQ	Limit of quantification
MBnzP	Monobenzyl phthalate
MBP	Mono-n-butyl phthalate
MCHP	Monocyclohexyl phthalate
MEHP	Mono-2-ethylhexyl phthalate
MiBP	Monoisobutyl phthalate
MiNP	Monoisononyl phthalate
MiPP	Monoisopropyl phthalate
MOP	Mono-n-octyl phthalate
MPP	Mono-n-pentyl phthalate
MS	Mass spectrometry
ODP	n-Octyl-n-decyl phthalate
RASFF	Rapid alert system for food and feed
SOP	Standard operating procedure
TDI	Tolerable daily intake

ABBREVIATIONS

TDS	Total diet study
WHO	World Health Organisation

1. INTRODUCTION

1.1 Background

Phthalates are a group of compounds produced in large volumes that are widely used as additives in plastics and consumer products [1]. Although it has been reported that they are no longer used in plastic food packaging [2] in 2009 there were thirteen notifications issued through the EU rapid alert system for food and feed (RASFF) [3] several of which reported the migration of phthalates from lids used to seal glass jars. Phthalates may also be used in adhesives or inks applied to such materials.

The annual production of phthalates was estimated by the World Health Organisation (WHO) to approach 8 million tonnes in 1991 [4]. They are ubiquitous in the environment and have been detected in environmental samples including rain water, water, soil and sediments, indoor air/dust and aquatic systems [5]. Hubert *et al*. estimated the release of di-(2-ethylhexyl) phthalate (DEHP) to the environment to be about 1.8% of the annual production [6]. Phthalates have also been detected in foods [7-11], in particular fatty foods, and phthalate metabolites have been observed in breast milk [12] and urine [13].

There is a large volume of literature on the toxicology of phthalates. The European Food Safety Authority (EFSA) has issued opinions on several phthalates in recent years and has revised the Tolerable Daily Intake values (TDIs) for some. New restrictions for the use of these phthalates in food contact plastics have been accepted and have been included in Directive 2007/19/EC, the 4th amendment to Directive 2002/72/EC. These restrictions relate specifically to migration from food contact materials but do not take into account any contribution from any environmental sources. Given that the potential for contamination of phthalates from environmental sources exists, the European Commission expressed an interest in controlling phthalates under the Contaminants in Foods Regulations.

The toxicity of the phthalate diesters has recently been described in a background paper for discussion within the UK Food Standards Agency Committee on Toxicology [14].

The metabolites of some phthalates are also suspected of producing teratogenic and endocrine-disrupting effects and studies have shown that di-n-butyl phthalate (DBP) and DEHP were metabolised to bioactive phthalate monoesters [15-17]. Moreover, monobutyl phthalate (MBP) and monoethylhexyl phthalate (MEHP) have been detected in consumer milk and infant formula in the ranges 0.6-3.9 µg/L (MBP) and 5.6-9.9 µg/L (MEHP) which suggests that the cows producing these products have been exposed to phthalates themselves [18]. Animal origin food is likely to contain these compounds and, therefore, a suitable analytical method is needed to determine them in food. The phthalate metabolites have been determined in humans for assessing exposure to phthalate diesters. The main matrix studied is urine [13, 19-22] but determination of monophthalates has also been carried out in serum or breast milk [12, 18, 23].

1.2 Analysis of phthalates

1.2.1 Phthalate diesters

Several analytical methods have been reported for the determination of these substances in foodstuffs and these have utilised both gas chromatograpy-mass spectrometry (GC-MS) and liquid chromatogtaphy- mass spectrometry/mass spectrometry (LC-MS/MS) analysis to quantify the levels present. The available methods were summarised in an EU report published in 2009 (EUR 23682 EN – 2009) [24]. Although both analytical methods provide good detection limits – literature data gives values of < 10 µg/kg, phthalates are ubiquitous and therefore elimination of all background traces during the analytical procedure is not possible. As a result reporting limits are higher than the detection limits quoted and are dependent on the measures taken to minimise background contamination.

1.2.2 Phthalate monoesters

The methods applied for the determination of phthalate monoesters usually entail solvent extraction, SPE clean-up and determination by LC-MS. Most of the methods reported are for the determination of these substances in biological matrices where the monoesters have been used as biomarkers to phthalate exposure. Methodology for the determination of phthalate monoesters in milk have been reported [18] but not for other foodstuffs.

1.2.3 Total phthalate determination

By converting all phthalates (including metabolites) to dimethyl phthalate the total phthalate concentration in a foodstuff can be determined. Methodology for the determination of the total phthalate concentration in foodstuffs has been described previously [e.g. 7-8].

1.3 Occurrence in foodstuffs

There have been few surveys to determine phthalate levels in foods. The last one in the UK, to determine these substances in infant formula, was reported almost 10 years ago. The majority of work in Europe has been carried out by the UK [7-9] and Danish authorities [10-11]. Up-to-date data on the levels of these substances in the UK diet is required to allow the overall dietary exposure of the UK population to be determined.

1.4 Sources of phthalate contamination

In the mid to late 1990s, three surveys were carried out at CSL, funded by MAFF, to determine the concentrations of phthalates in infant formula and foodstuffs [7-9]. A fourth survey [25], also conducted by CSL, measured the levels of selected phthalates in paper and board packaging materials. The foodstuffs in contact with the paper and board samples found to contain the highest levels of phthalates were also analysed. The concentrations of the phthalates in some foods were greater than the worst case migration (i.e. even if all of the phthalates detected in the packaging had transferred to the food they would not account for the levels detected). The rate of transfer from the packaging to the food would have been expected to be similar for the two phthalates measured (DBP and DEHP). This was not the case for many samples. Finally when the phthalate concentrations at the surface of the foodstuff were compared with those at the core they were similar in approximately half of the samples analysed. If the source of these substances was the packaging then a concentration gradient would be expected to be observed with higher levels at the surface of the foodstuff. This distribution of migrants within foods has been demonstrated within the EU project FOODMIGROSURE [26]. A concentration profile (highest in the layer of food in direct contact with the packaging) was obtained for all but the most volatile substances where migration can occur through the gas phase. Therefore questions were raised following this study [25] as to whether or not the phthalates detected were present as a consequence of migration from the food contact materials and articles or if they would have been present at the same levels even if the foodstuff had not been packaged, i.e. from some other source of contamination.

2. AIMS

The aims of this project were to provide data on the exposure of the UK population to phthalates, firstly through the analysis of the UK total diet study (TDS) samples and secondly through the analysis of unpackaged and packaged foodstuffs. TDSs are an important part of the Food Standards Agency's (FSA) surveillance programme for chemicals in food. TDS samples have been collected on an annual basis since 1966 (by JFSSG and MAFF prior to the establishment of the FSA). Results from TDSs are used to estimate dietary exposures of the general UK population to chemicals in food, such as nutrients and contaminants, to identify trends in exposure and to make assessments on the safety and nutritional quality of the food supply. The TDS samples comprise twenty food groups. In total 119 sub-categories of food are combined into the 20 groups. The relative proportion of each food category within a group reflects its importance in the average UK household diet and is based on an average of three previous years of consumption data from the National Food Survey. Foods are grouped so that commodities known to be susceptible to contamination (e.g. offals, fish) are kept separate, as are foods which are consumed in large quantities (e.g. bread, potatoes, milk).

In addition to the determination of the exposure of the UK population to these chemicals, this project aimed to establish the source of these substances in foodstuffs.

3. PROJECT OBJECTIVES

The project objectives defined in the scope of work were:

Objective 01. Method development and in-house validation

Objective 02. TDS sample receipt, logging and storage

Objective 03. Analysis of 20 TDS samples for phthalates

Objective 04. Screening of the empty TDS containers for phthalates

Objective 05. Interim report describing the results of the analysis of the 20 TDS samples and containers

Objective 06. Establishing methods to differentiate between environmental sources of phthalates and contamination arising as a result of migration from food contact materials

Objective 07. Interim report describing the results of the investigation into the applicability of the proposed methods to determine the likely source(s) of phthalates in foods

Objective 08. Perform a satisfactory sample check exercise

Objective 09. Sample receipt, logging and storage

Objective 10. Analysis of retail food samples

Objective 11. Interim spreadsheets

Objective 12. Final report

4. METHOD DEVELOPMENT AND IN-HOUSE VALIDATION

Methodologies for the determination of phthalate diesters, phthalate monoesters, phthalic acid and total phthalates in foodstuffs were developed.

4.1 Analytes

Based on information provided by the Food Standards Agency and reported concentrations of phthalate diesters in foodstuffs the following analytes were selected:

- Dimethyl phthalate (DMP)
- Diethyl phthalate (DEP)
- Diisopropyl phthalate (DiPP)
- Diallyl phthalate (DAP)
- Diisobutyl phthalate (DiBP)
- Di-n-butyl phthalate (DBP)
- Di-n-pentyl phthalate (DPP)
- Di-n-hexyl phthalate (DHexP)
- Benzyl butyl phthalate (BBP)
- Dicyclohexyl phthalate (DCHP)
- Di-(2-ethylhexyl) phthalate (DEHP)
- Di-n-heptyl phthalate (DHepP)
- Di-n-octyl phthalate (DOP)
- n-Octyl-n-decyl phthalate (ODP)
- Diisononyl phthalate (DiNP)
- Diisodecyl phthalate (DiDP)
- Di-n-decyl phthalate (DDP)

The monoesters selected were those that could be purchased commercially:

- Monoisopropyl phthalate (MiPP)
- Monoisobutyl phthalate (MiBP)
- Mono-n-butyl phthalate (MBP)
- Monobenzyl phthalate (MBnzP)
- Monocyclohexyl phthalate (MCHP)
- Mono-n-pentyl phthalate (MPP)
- Mono-2-ethylhexyl phthalate (MEHP)
- Mono-n-octyl phthalate (MOP)
- Monoisononyl phthalate (MiNP)

Phthalic acid was also included.

4.2 Method development and validation – phthalate diesters

4.2.1 Sample extraction

The extraction protocol was optimised to allow all seventeen phthalate diesters to be analysed in the same method. Despite the differences in size and polarity this was achieved using a mixture of acetonitrile and dichloromethane as the extraction solvent, evaporation to dryness and reconstitution in acetonitrile (to remove any traces of fat remaining in the extract). Care had to be exercised in the concentration step to ensure that the smaller, more volatile phthalates were not lost. Further clean-up steps were avoided due to risk of contamination of the sample with those ubiquitous phthalates (see Section 3.1.3).

4.2.2 Analysis

Both GC-MS and LC-MS/MS methods have been described for the determination of phthalate diesters [24]. GC-MS was chosen here. Chromatographic conditions were optimised to provide a single multi-analyte method of analysis. Stock solutions of each analyte and internal standard prepared at a concentration of 1 mg/ml were analysed with the mass spectrometer operated in full scan mode monitoring from 40 to 450 amu. The retention times of each analyte and the masses to analyse were selected from the analysis of these solutions (Table 1). Deuterated internal standards were used.

Analysis of the n-octyl-n-decyl phthalate revealed three peaks in the total ion chromatogram. These were identified (from their retention times and full scan mass spectra) as di-n-octyl phthalate, n-octyl-n-decyl phthalate and di-n-decyl phthalate. The percentage of the response derived from the n-octyl-n-decyl phthalate was calculated to be 58%. This was subsequently taken into account when determining the performance characteristics for these analytes.

4.2.3 Minimising background levels of phthalate diesters

The problems associated with the analysis of phthalates are well known. In particular analytical background levels of those phthalates that are ubiquitous need to be minimised. The measures taken included:

- A laboratory was dedicated to the analysis of all samples (away from where the analytical standards were prepared and from where the control sample spiking was carried out).
- All solvents were screened for phthalates prior to analysis and the ‘cleanest’ ones used throughout the study. Further clean up of the solvents used was performed by the addition of muffled aluminium oxide to the solvent bottles for at least 24 hours prior to use.
- All glassware was solvent rinsed and/or baked prior to use.
- PTFE vial septa were used throughout.
- A Merlin Microseal GC injector was used.
- A GC-MS instrument was dedicated to this analysis.

To establish the background levels procedural blank samples were taken through the analytical method. The SIM chromatograms obtained from the analysis of the procedural blank samples and the procedural blank samples overspiked with each phthalate diester at a nominal concentration of 0.05 mg/kg are shown in Figures 1 to 17. All measured ions are provided to detect any interferences in any of the channels of interest derived from the extraction procedure.

Low responses were observed in the m/z 149 channel for DiBP and DBP and for the m/z 149 and 167 channels for DEHP. The peak area ratios for the procedural blank samples were compared with the same samples overspiked at a nominal concentration equivalent to 0.05 mg/kg in the foodstuff. This data confirmed that the background contamination derived from the analytical procedure was < 1 µg/kg for the DiBP and DBP. DEHP could not be separated from DHepP in the chromatograms and therefore quantification uses the m/z 279 ion which is unique to this phthalate. No background response was detected for this concentration in the m/z 279 channel.

4.2.4 Validation of the methodology for foodstuffs

In order to demonstrate that the method developed was suitable for phthalate diester determination in a wide range of foodstuffs then the method performance characteristics (limit of detection and limit of quantification, linearity of the analysis, repeatability, in-house reproducibility and recovery) were derived. The performance of the method was assessed by the analysis of three foods (cheese – high fat, orange juice – high carbohydrate and pork mince – high protein) spiked with all 17 phthalate diesters.

4.2.4.1 Limit of detection and limit of quantification

The target LOQ (10x the signal:noise ratio) was set as 0.05 mg/kg for each of the single phthalates and 1 mg/kg for the isomeric mixes (DiNP and DiDP). Figures 18 to 33 show the SIM chromatograms (only the ion selected for quantification is shown) obtained when the three foods were analysed after spiking with the analytes at these target LOQ values.

Where no response was observed in the blank (unspiked) foodstuff then the LOD and LOQ in the foodstuff were calculated as 3 and 10 x the signal:noise ratio respectively.

Where a response was observed at the retention time of the analyte of interest in the blank (unspiked) foodstuff this approach was not applicable. In these cases the LOD and LOQ were calculated as 3 and 10 x the signal:noise ratio obtained from the procedural blank and overspiked samples. These are likely to be underestimations of the detection limits when the foodstuff is present to affect the partitioning.

Where a response was observed at the retention time of the analyte of interest in the blank (unspiked) foodstuff and in the procedural blank (i.e. DiBP and DBP) the LOD and LOQ were calculated as 3 and 10 x the concentration detected in the procedural blank samples.

The LODs and LOQs determined are given in Table 2.

The target LOQ was met for DMP, DEP, DiPP, DAP, DiBP, DBP, DPP, DHexP, BBP, DCHP, DEHP, DHepP, DOP, ODP and DDP in all three food matrices.

DHepP and DEHP could not be resolved using the GC column and temperature programme described. As a result the ions that were unique to these two analytes were used for quantification purposes. These ions give a much lower response than the m/z 149 ion and as a result the LOD and LOQ are lower but are still less than the target LOQ.

The target LOD and LOQ was not met for DiNP and DiDP in the high fat foodstuff (cheese). DiNP and DiDP are highly fat soluble and therefore are not readily extracted from the cheese. Here even the high concentration spiked (1 mg/kg) was not detected. Although LC-MS/MS analysis may be expected to improve the sensitivity of the analysis this approach will not allow us to compare isomeric profiles as effectively as by GC-MS as is required for the investigations into the source(s) of the phthalates in the foods. Therefore this higher LOD/LOQ was considered acceptable for the purpose of this study.

4.2.4.2 Linearity of the method

The calibration was linear for DMP, DEP, DiPP, DAP, DiBP, DBP, DPP, DHexP, BBP, DCHP, DEHP, DHepP, DOP, ODP and DDP at concentrations equivalent to 0 to 0.5 mg/kg in the foodstuff and for DiNP and DiDP at concentrations equivalent to 0 to 6.5 mg/kg in the foodstuff using calibration standards prepared by two analysts on different days. The data generated by the two analysts is combined in the calibration graphs shown in Figures 34 to 50. The correlation coefficients (r^2) were better than 0.99 in all cases.

4.2.4.3 Repeatability and reproducibility

The repeatability of the analysis of the phthalate diesters was determined in the three selected food matrices. The foods were spiked at nominal concentrations of 0.05 and 0.3 mg/kg for DMP, DEP, DiPP, DAP, DiBP, DBP, DPP, DHexP, BBP, DCHP, DEHP, DHepP, DOP, ODP and DDP and at nominal concentrations of 1 and 5 mg/kg for diisononyl phthalate and diisodecyl phthalate. The results obtained are summarised in Tables 3 to 19. The concentrations reported have been normalised to the actual spiking levels for clarity. The repeatability was acceptable for all analytes spiked at these levels with the concentrations detected in the spiked samples giving relative standard deviations of < 20%.

When determining the repeatability, the phthalate concentrations in the unspiked food were not determined and therefore the replicate results for Analyst 1 in Tables 3 to 19 have not been corrected for any background levels present in the foodstuffs.

The average concentrations detected in the spiked foods in analysis 1 (n = 8) and analysis 2 (n = 3) showed good agreement (reproducibility). With the exception of the DEHP, where different batches of the foods were used containing different background levels (as mentioned above, the concentrations measured in the repeatability samples have not been corrected for the background levels) the values were within 30% of one another.

4.2.4.4 Recovery

The analytical recoveries relative to the solvent calibration standards are shown in Tables 3 to 19 for each matrix. Recoveries were analyte and food matrix specific and therefore overspiking and recovery correction is required for the quantification of the phthalate diesters in foods.

The resulting standard operating procedure (SOP) is provided as Annex 1.

4.3 Method development and validation – phthalate monoesters

4.3.1 Sample extraction

The extraction protocol was optimised to allow all nine phthalate monoesters to be analysed in the same method. Samples were extracted with acidified acetonitrile:dichloromethane (1:1) and concentrated/cleaned-up prior to analysis.

4.3.2 Analysis

LC-MS/MS was used as the phthalate monoesters are polar compounds and are therefore more amenable to this technique. Chromatographic conditions were optimised to provide a single multi-analyte method of analysis. Labelled internal standards were used. Individual stock solutions of each analyte and each internal standard were prepared at a concentration of 10 µg/mL and were infused into the mass spectrometer in full scan mode to determine parent ion m/z values. The mass spectrometer was then used in MS/MS mode to determine and optimise MRM transitions. For each monoester the channel with the largest response was used as the quantification channel and the second largest was selected for confirmation. Individual stock solutions at a concentration of 1 µg/mL were then injected into the LC-MS/MS to determine individual retention times. For each analyte the selected transitions and cone voltages are shown in Table 20. The MiBP and MBP co-eluted in the chromatogram. One transition was identified that was specific to MBP and therefore to quantify the concentration of MBP derived from this response was subtracted from the concentration derived from the combined response.

4.3.3 Validation of the methodology for foodstuffs

In order to demonstrate that the method developed was suitable for phthalate monoester determination in a wide range of foodstuffs then the method performance characteristics (limit of detection and limit of quantification, linearity of the analysis, repeatability, in-house reproducibility and recovery) were derived. The performance of the method was assessed by the analysis of three foods (cheese – high fat, orange juice – high carbohydrate and pork mince – high protein) spiked with the nine phthalate monoesters.

4.3.3.1 Limit of detection and limit of quantification

The target LOQ (10x the signal:noise ratio) was set as 0.05 mg/kg for each phthalate monoester. Figures 51 to 59 show the MRM chromatograms (only the transition selected for quantification is shown) obtained when the three foods were analysed with the addition of the internal standards and after spiking with the analytes at this target LOQ.

Where no response was observed in the blank (unspiked) foodstuff then the LOD and LOQ in the foodstuff were calculated from the overspiked response as 3 and 10 x the signal:noise ratio respectively.

For the mono-2-ethylhexyl phthalate a response was observed at the retention time of the analyte of interest in both the blank (unspiked) foodstuff and the procedural blank samples. For this analyte the LOD and LOQ were calculated as 3 and 10 x the concentration calculated for the response detected in the procedural blank samples.

The LODs and LOQs determined are given in Table 21. The target LOQ was met for all analytes.

4.3.3.2 Linearity of the method

The calibration was linear for all analytes at concentrations equivalent to 0 to 0.1 mg/kg in the foodstuff. The calibration graphs are shown in Figures 60 to 68. The correlation coefficients (r^2) were better than 0.99 in all cases.

4.3.3.3 Repeatability and reproducibility

The repeatability of the analysis was determined in the three selected food matrices. The foods were spiked at nominal concentrations of 0.05 mg/kg for each analyte. The results obtained are summarised in Tables 22 to 30. For all analytes the repeatability was acceptable with the concentrations detected in the spiked samples giving relative standard deviations of < 19%.

The average concentrations detected in the spiked foods in analysis 1 (n = 6) and analysis 2 (n = 3) showed good agreement (reproducibility). The measured concentrations in the two analyses were within 30% of one another for all nine phthalate monoesters.

4.3.3.4 Recovery

The analytical recoveries relative to the solvent calibration standards are shown in Tables 22 to 30 for each matrix. Recoveries were in the range 65 to 125%. Recoveries were analyte and food matrix specific and repeatable within the batch and reproducible between batches. For example monoisopropyl phthalate spiked into pork mince gave 65% recovery with an RSD of 12% in analysis 1 and 60% recovery with an RSD of 0.9% in analysis 2. Therefore overspiking and recovery correction is required for the quantification of the phthalate monoesters in foods.

The resulting SOP is provided as Annex 2.

4.4 Method development and validation – phthalic acid

4.4.1 Sample extraction

Samples were extracted with acidified acetonitrile:dichloromethane (1:1) and concentrated/cleaned-up prior to analysis.

4.4.2 Analysis

LC-MS/MS was used for this polar compound. Labelled phthalic acid was used as an internal standard. Individual stock solutions of the analyte and internal standard were prepared at a concentration of 10 µg/mL and were infused into the mass spectrometer in full scan mode to determine parent ion m/z values. The mass spectrometer was then used in MS/MS mode to determine and optimise MRM transitions. The largest response was used as the quantification channel and the second largest was selected for confirmation. Individual stock solutions at a concentration of 1 µg/mL were then injected into the LC-MS/MS to determine individual retention times. The retention time was 2.5 minutes, the quantification transition was 164.75 -> 120.81 (cone voltage = 10 eV) and the confirmation transition was 164.75 -> 76.95 (cone voltage = 15 eV).

4.4.3 Validation of the methodology for foodstuffs

In order to demonstrate that the method developed was suitable for phthalic acid determination in a wide range of foodstuffs then the method performance characteristics (limit of detection and limit of quantification, linearity of the analysis, repeatability, in-house reproducibility and recovery) were derived. The performance of the method was assessed by the analysis of three foods (cheese – high fat, orange juice – high carbohydrate and pork mince – high protein) spiked with phthalic acid

4.4.3.1 Limits of detection and quantification

The target LOQ (10x the signal:noise ratio) was set as 0.05 mg/kg. Figure 69 shows the MRM chromatograms (only the transition selected for quantification is shown) obtained when the three foods were analysed with the addition of the internal standards and after spiking with the phthalic acid at this target LOQ. In all cases a response was observed at the retention time of the analyte of interest in the blank (unspiked) foodstuff. The LOD (5.3 µg/kg) and LOQ (17.7 µg/kg) were calculated as 3x and 10x the average response in the blank samples. The target LOQ was met.

4.4.3.2 Linearity of the method

The calibration was linear at concentrations equivalent to 0 to 0.1 mg/kg in the foodstuff. The calibration graph is shown in Figure 70. The correlation coefficient (r^2) was 0.999.

4.4.3.3 Repeatability and reproducibility

The repeatability of the analysis was determined in the three selected food matrices. The foods were spiked at nominal concentrations of 0.05 mg/kg. The results obtained are summarised in Table 31. For all foods the repeatability was acceptable with the concentrations detected in the spiked samples giving relative standard deviations of < 12%.

The average concentrations detected in the spiked foods in analysis 1 (n = 6) and analysis 2 (n = 3) showed good agreement (reproducibility). The measured concentrations in the two analyses were within 20% of one another.

4.4.3.4 Recovery

The analytical recoveries relative to the solvent calibration standards are shown in Table 31. Recoveries were in the range 87 to 116%.

The resulting SOP is provided as Annex 3.

4.5 Method development and validation – total phthalates

In previous studies differences were observed in the total phthalate concentration (calculated by converting all phthalates to dimethyl phthalate) and the sum of the individual phthalate diesters measured in foods. It was proposed that the difference in these two values could be due to the presence of phthalic acid and phthalate monoesters in the food samples tested as these substances would also be converted to dimethyl phthalate and therefore included in the total phthalate measurement as well as the presence of any phthalate diesters that were not included in the analysis.

4.5.1 Sample extraction

Samples were extracted with acidified acetonitrile:dichloromethane (1:1) and the phthalates extracted were converted to DMP by heating with methanolic potassium hydroxide solution and then with boron trifluoride/diethyl ether complex. These extracts were then fractionated by size exclusion chromatography and analysed GC-MS.

4.5.2 Analysis

As DMP was the analyte to be measured then the same approach as described for the phthalate diesters (Section 4.2.2) was used.

4.5.3 Validation of the methodology for foodstuffs

In order to demonstrate that the method developed was suitable for total phthalate determination in a wide range of foodstuffs then the method performance characteristics (limit of detection and limit of quantification, linearity of the analysis, repeatability, in-house reproducibility and recovery) were derived. The performance of the method was assessed by the analysis of three foods (cheese – high fat, orange juice – high carbohydrate and pork mince – high protein). Validation was achieved through the analysis of replicate spiked samples containing 200 µg/kg each of diethyl phthalate, diisobutyl phthalate and di-n-decyl phthalate in the food. This is equivalent to 420 µg/kg of dimethyl phthalate in the final measurement. These phthalates were chosen as they represent a low, medium and high molecular weight phthalate diester.

4.5.3.1 Limits of detection and quantification

Figure 71 shows the selected ion chromatograms obtained when the three foods were analysed with the addition of the internal standards and after spiking with the analytes. The LOD and LOQ in the foodstuff were calculated from the overspiked response as 3 and 10 x the signal:noise ratio respectively. The LODs and LOQs determined are given in Table 32.

4.5.3.2 Linearity of the method

The calibration was linear for dimethyl phthalate at concentrations equivalent to 0 to 1000 µg/kg in the foodstuff. The calibration graph is shown in Figure 72. The correlation coefficient (r^2) was 0.9995.

4.5.3.3 Repeatability and reproducibility

The repeatability of the analysis was determined in the three selected food matrices. The foods were spiked at nominal concentrations 200 µg/kg each of diethyl phthalate, diisobutyl phthalate and di-n-decyl phthalate in the food. This is equivalent to 420 µg/kg of dimethyl phthalate in the final measurement. The results obtained are summarised in Table 33. The repeatability was acceptable with the concentrations detected in the spiked samples giving relative standard deviations of < 12%.

The average concentrations detected in the spiked foods in analysis 1 (n = 6) and analysis 2 (n = 3) showed good agreement (reproducibility). The measured concentrations in the two analyses were within 8% of one another.

4.5.3.4 Recovery

The analytical recoveries relative to the solvent calibration standards are shown in Table 33 for each matrix. Recoveries were in the range 88 to 100%.

The resulting SOP is provided as Annex 4.

5. TDS SAMPLE ANALYSIS

5.1 Samples

The TDS samples were received on 03/07/2008. On receipt in the laboratory each was assigned a unique sample number using the Fera LIMS system. Sample details are provided in Table 34. Samples were stored frozen (-20℃) until required for analysis.

5.2 Analysis

All TDS samples were analysed according to the SOPs provided in Annexes 1 to 4.

5.3 Results

The phthalate diester, phthalate monoester, phthalic acid and total phthalate concentrations detected in the twenty TDS samples are given in Tables 35 to 38.

5.3.1 Phthalate diesters

The concentrations of the phthalates detected in the TDS samples are given in Table 35.

In many cases no response was observed or the response was < 3 x the signal to noise ratio in the selected ion chromatogram used for quantification. These are reported in Table 35 as < LOD. The LOD was calculated as 3 x signal to noise ratio using the response of the lowest concentration overspiked samples. The LODs were sample/analyte specific and are also given in Table 35.

In some cases a response was observed in the chromatogram but its peak height was < 10 x the signal to noise ratio in the selected ion chromatogram used for quantification. These are reported in Table 35 as < LOQ (LOQ = limit of quantification). The LOQ was calculated as 10 x signal to noise ratio using the response of the lowest concentration overspiked samples. LODs were sample/analyte specific and are also given in Table 35. The samples reported as < LOQ are highlighted in blue. All concentrations determined from the response observed were less than the calculated LOQ value. For these samples it was not confirmed whether or not the response was due to the analyte of interest or from a co-eluting interference (whole or in-part).

For those samples in which the response was > 10 x the signal to noise ratio in the selected ion chromatogram used for quantification and all confirmation criteria were met the recovery corrected concentration was calculated. For the quantified results recoveries were in the range 75 to 116%. Relative retention times and ion ratios were used to confirm that the responses observed were due to the phthalate diesters.

The following phthalates were detected and confirmed in the TDS samples:

- Diethyl phthalate in one sample at a concentration of 13 µg/kg
- Diisobutyl phthalate in four samples at concentrations in the range 17 to 81 µg/kg
- Di-n-butyl phthalate in seven samples at concentrations in the range 6 to 28 µg/kg
- Benzyl butyl phthalate in one sample at a concentration of 8 µg/kg

- Di-(2-ethylhexyl) phthalate in eleven samples at concentrations in the range 35 to 789 µg/kg

These are highlighted in green in Table 35.

Where responses were observed but the confirmation criteria were not met the calculated concentration is provided. These responses could not be attributed to the phthalate diester. This may be due to the response being too low such that the confirmatory ions were close to the limit of detection or the response being due to a co-eluting interference (whole or in-part) giving a response in the target or qualifying ions.

5.3.2 Phthalate monoesters

The concentrations of the phthalate monoesters detected in the TDS samples are given in Table 36.

In many cases no response was observed or the response was < 3 x the signal to noise ratio in the multiple reaction monitoring (MRM) chromatogram used for quantification. These are reported in Table 36 as < LOD (LOD = limit of detection). The LOD was calculated as 3 x signal to noise ratio using the response of the overspiked samples. The LODs were analyte specific and are given in Table 36.

In some cases a response was observed in the chromatogram but its peak height was < 10 x the signal to noise ratio in the selected ion chromatogram used for quantification. These are reported in Table 36 as < LOQ (LOQ = limit of quantification). The LOQ was calculated as 10 x signal to noise ratio using the response of the overspiked samples. LOQs were analyte specific and are given in Table 36. The samples reported as < LOQ are highlighted in blue. All concentrations determined from the response observed were less than the calculated LOQ value. For these samples it was not confirmed whether or not the response was due to the analyte of interest or from a co-eluting interference (whole or in-part).

For those samples in which the response was > 10 x the signal to noise ratio in the MRM chromatogram used for quantification and all confirmation criteria were met, the recovery corrected concentration was calculated. For the quantified results recoveries were in the range 90 to 114%. Relative retention times and ion ratios were used to confirm that the responses observed were due to the phthalate monoesters.

The following phthalate monoesters were detected and confirmed in the TDS samples:

- Mono-n-butyl phthalate in three samples at concentrations in the range 29 to 52 µg/kg
- Mono-(2-ethylhexyl) phthalate in one sample at 54 µg/kg

These are highlighted in green in Table 36. All samples that the monoesters were detected in were animal/fish based wherein metabolism of any diesters to which they have been exposed may be expected.

Procedural blank samples were analysed alongside the foodstuffs. In all cases the phthalate monoester concentrations detected in the procedural blank samples were equivalent to concentrations of < 15 µg/kg in the foodstuff.

5.3.3 Phthalic acid

The concentrations of the phthalic acid detected in the TDS samples are given in Table 37.

In one case no response was observed in the multiple reaction monitoring (MRM) chromatogram used for quantification. This is reported in Table 37 as < LOD (LOD = limit of detection). The LOD was calculated as 3 x signal to noise ratio using the response of the overspiked samples.

In two cases a response was observed in the chromatogram but its peak height was < 10 x the signal to noise ratio in the selected ion chromatogram used for quantification. These are reported in Table 37 as < LOQ (LOQ = limit of quantification). The LOQ was calculated as 10 x signal to noise ratio using the response of the overspiked samples. The concentrations determined from the response observed were less than the calculated LOQ value. For these samples it was not confirmed whether or not the response was due to the analyte of interest or from a co-eluting interference (whole or in-part).

For those samples in which the response was > 10 x the signal to noise ratio in the MRM chromatogram used for quantification and all confirmation criteria were met the recovery corrected concentration was calculated. For the quantified results recoveries were acceptable. Relative retention times and MRM channel ratios were used to confirm that the responses observed were due to the phthalic acid.

5.3.4 Total phthalates

The total phthalate concentrations detected in the TDS samples are given in Table 38. All samples gave measurable total phthalate levels. When comparing the total phthalate concentrations with the sum of the individual diesters, monoesters and phthalic acid differences were observed. The measurements derived from the targeted analysis of the individual substances are considered to be the most accurate as the analysis is more direct and straightforward compared to the uncertainties associated with the complex methodology of the total phthalate determination.

6. PHTHALATE SOURCE STUDIES

Phthalates may be present in foods as a result of migration from food contact materials and articles. Although it is reported that phthalates are no longer used in the production of food contact plastics they may be used as carriers for pigments in printing inks and in adhesives applied to a plastic food contact material or article. Further, phthalates are commonly found in paper and board food contact materials, in particular when the material has been recycled. As a result of their widespread use and their moderate resistance to degradation phthalates are also ubiquitous in the environment. Therefore they may be also be present in foods as a consequence of environmental contamination.

For a solid foodstuff it may be envisaged that environmental and migration sources of phthalates may be differentiated in three ways:

(a) Which phthalates are present in the food and the packaging?

For any phthalates to be present in food as a result of migration from food contact materials it would be expected that the phthalates present in the packaging materials would also be detected in the foods, i.e. the identity would be the same.

(b) What are the ratios of the phthalate concentrations in the foods and in the packaging?

As phthalates are structurally similar it would be expected that they would migrate at a similar rate so the ratio of the concentrations of the individual phthalates to one another in the packaging would be expected to be similar to that in the foods.

(c) What is the concentration depth profile of the phthalates in the food?

For a solid foodstuff the concentration of any phthalates migrating from a food contact material into a food would be expected to be greater at the food contact surface compared to within the bulk of the foodstuff. Similarly any phthalates 'picked-up' by the food during processing would expect to be at higher concentrations at the foods surface than in the centre. By cutting slices of the food at defined distances from the packaging material and analysing each 'slice' separately a concentration depth profile should be observed if the source of the phthalates in the foodstuff is migration. If this is not the case then the phthalates will be distributed homogeneously throughout the foodstuff.

For a liquid foodstuff, e.g. milk, only (a) and (b) above would be relevant as mixing of the foodstuff would readily occur and therefore it would not be expected that a concentration depth profile would be observed. This is also expected to be true, to a certain extent, for semi-solid foods such as yoghurt.

6.1 Sample details

Twenty nine packaged foodstuffs were purchased from retail outlets in the north of England. Each was assigned a unique sample code using the Fera LIMS system. Sample details are provided in Table 39. These samples were selected for screening purposes to establish the presence (or absence) of phthalates in the packaging materials.

6.2 Extraction and analysis

6.2.1 Gaskets

Gasket (100 mg), cut from the lid of the test sample using a hexane rinsed scalpel, was dissolved in tetrahydrofuran (10 mL). The sample was sonicated to effect the dissolution of the polymer. Ethanol (20 mL) was added dropwise to precipitate the polymer. The mixture was centrifuged and the solvent decanted into a clean glass vial. The sample was evaporated to dryness on a heating block set at 50℃ (no nitrogen). The dry residue was reconstituted in acetonitrile (2 mL) prior to analysis by GC-MS.

One sample was prepared as described.

Duplicate samples were prepared as described but with the addition of 150 µl of a mixed labelled phthalate internal standard solution to the gasket prior to dissolution in the solvent. The mixed labelled internal standard solution contained d_4-dimethyl phthalate, d_4-diethyl phthalate, d_4-dipropyl phthalate, d_4-diisobutyl phthalate, d_4-di-n-butyl phthalate, d_4-di-n-pentyl phthalate, d_4-di-n-hexyl phthalate, d_4-benzylbeutyl phthalate, d_4-dicyclohexyl phthalate, d_4-di-2-ethylhexyl phthalate, d_4-di-n-heptyl phthalate, d_4-di-n-octyl phthalate and d_4-di-n-nonyl phthalate each at a nominal concentration of 100 µg/mL.

Duplicate overspiked sample were prepared as described but with the addition of the internals standards (as above) and with the addition of 175 µl of a mixed phthalate standard solution to the gasket prior to dissolution in the solvent. The mixed phthalate standard solution contained dimethyl phthalate, diethyl phthalate, diisopropyl phthalate, diallyl phthalate, diisobutyl phthalate, di-n-butyl phthalate, di-n-pentyl phthalate, di-n-hexyl phthalate, benzylbeutyl phthalate, dicyclohexyl phthalate, di-2-ethylhexyl phthalate, di-n-heptyl phthalate, di-n-octyl phthalate, diisononyl phthalate, diisodecyl phthalate and di-n-decyl phthalate each at a nominal concentration of 150 µg/mL.

Quantification was achieved by the analysis of calibration standards containing 150 µl of the 100 µg/mL mixed labelled phthalate internal standard solution and 0, 45, 90, 175, 350, 700 and 1000 µL of the 150 µg/mL mixed phthalate standard solution in a total volume of 2 mL of acetonitrile (the final volume of the reconstituted extract).

All extracts and calibration standards were analysed following the analytical procedure given in Annex 1.

6.2.2 Packaging

An area of 0.5 dm^2 was cut from each packaging material and the mass was recorded. The entire packaging material was then cut into small pieces (~ 1 x 1 cm), the pieces were mixed and a mass equivalent to an area of 0.5 dm^2 was weighed into a glass vial, the packaging was extracted with dichloromethane (20 mL) by shaking for 4 hours at room temperature. The solvent layer was decanted into a clean glass vial and the extract was evaporated to dryness on a heating block set at 50℃ (no nitrogen). The dry residue was reconstituted in acetonitrile (1 mL) prior to analysis by GC-MS.

One sample was prepared as described.

Duplicate samples were prepared as described but with the addition of 25 µl of a mixed labelled phthalate internal standard solution to the packaging material prior to extraction. The mixed labelled internal standard solution contained d_4-dimethyl phthalate, d_4-diethyl phthalate, d_4-dipropyl phthalate, d_4-diisobutyl phthalate, d_4-di-n-butyl phthalate, d_4-di-n-pentyl phthalate, d_4-di-n-hexyl phthalate, d_4-benzylbeutyl phthalate, d_4-dicyclohexyl phthalate, d_4-di-2-ethylhexyl phthalate, d_4-di-n-heptyl phthalate, d_4-di-n-octyl phthalate and d_4-di-n-nonyl phthalate each at a nominal concentration of 100 µg/mL.

Duplicate overspiked sample were prepared as described but with the addition of the internals standards (as above) and with the addition of 25 µl of a mixed phthalate standard solution to the gasket prior to dissolution in the solvent. The mixed phthalate standard solution contained dimethyl phthalate, diethyl phthalate, diisopropyl phthalate, diallyl phthalate, diisobutyl phthalate, di-n-butyl phthalate, di-n-pentyl phthalate, di-n-hexyl phthalate, benzylbutyl phthalate, dicyclohexyl phthalate, di-2-ethylhexyl phthalate, di-n-heptyl phthalate, di-n-octyl phthalate, diisononyl phthalate, diisodecyl phthalate and di-n-decyl phthalate each at a nominal concentration of 100 µg/mL.

Quantification was achieved by the analysis of calibration standards containing 25 µL of the 100 µg/mL mixed labelled phthalate internal standard solution and 0, 5, 10, 25, 50, 100 and 250 µL of the 100 µg/mL mixed phthalate standard solution made up in dichloromethane to achieve a total volume of 20 mL. The calibration standards were evaporated to dryness (alongside the samples) and reconstituted in acetonitrile (1 mL) prior to analysis by GC-MS.

All extracts and calibration standards were analysed following the analytical procedure given in Annex 1.

6.2.3 Foodstuffs

Foods were analysed as described in Annex 1.

6.3 Results

6.3.1 Phthalate diesters in gaskets and other packaging materials

Phthalates were detected in nine of the 29 packaging materials at concentrations in excess of the limit of quantification (LOQ). The concentrations detected are shown in Table 40.

In general the levels present were low. The worst case migration was calculated, assuming 100% transfer from the packaging to the foodstuff taking into account the packaging area/total mass of gasket and the mass of the foodstuff. The results of these calculations are shown in Table 41.

6.3.2 Phthalate diesters in foods

Taking into account the food contact conditions, the presence of any packaging layers that may act as total or partial barriers to migration, the fact that typically up to 10% of a substance will migrate and the analyte limits of detection sample S09-003808 (Lasagne sheets) was selected for analysis.

Sample S09-003808 (Lasagne sheets) is a solid foodstuff and is therefore suitable for testing the three hypotheses given above:

(a) Which phthalates are present in the food and the packaging?

Dimethyl phthalate, diisobutyl phthalate, di-n-butyl phthalate and di-(2-ethylhexyl) phthalate were detected in the packaging.

(b) What are the ratios of the phthalate concentrations in the foods and in the packaging?

(c) What is the concentration depth profile of the phthalates in the food?

The two outer lasagne sheets, the next two sheets and two sheets from the centre of the stack were selected as the three layers of the foodstuff.

Triplicate samples of the lasagne sheets were purchased. From each the two outer lasagne sheets, the next two sheets and two sheets from the centre of the stack were tested. The results obtained are shown in Table 42. Recoveries were acceptable, in the range 85 to 110%. Results have not been corrected for recovery. Only diisobutyl phthalate was detected in the foodstuff. This was not unexpected as this phthalate was present at the high concentration in the packaging, consistent with hypothesis (a). As only one phthalate was detected in the foodstuff then hypothesis (b) could not be tested but the absence of the other phthalates in the food is at least consistent, because pro-rata they would be expected to be present at levels of less than the limits of detection. Figure 73 shows the concentration depth profiles for the diisobutyl phthalate for the three samples tested. The highest concentration of the diisobutyl phthalate was in the lasagne sheet that comes into direct contact with the cartonboard packaging. Although only one example this result provides clear evidence that the source of the diisobutyl phthalate in the food was the packaging.

7. PHTHALATE DIESTERS IN RETAIL FOODSTUFFS

Fifteen of the seventeen phthalate diesters for which the methodology was validated were selected for analysis: DMP, DEP, DiPP, DAP, DiBP, DBP, DPP, DHP, BBP, DCHP, DEHP, DOP, DiNP, DiDP and DDP.

7.1 Samples

Two hundred and sixty one foodstuffs were received from Ventress Technical Ltd. Fera LIMS numbers and the sample description are given in Table 43.

7.2 Extraction and analysis

On receipt in the laboratory the foodstuff was removed from the packaging and homogenised. Precautions were taken to minimise contamination (as described previously, Section 4.2.3). The samples were analysed according to the SOP given in Annex 1 but with the omission of the DHepP (excluded as it co-elutes with DEHP and its inclusion resulted in a higher LOQ for this more common phthalate diester) and ODP.

7.3 Results

The concentrations of the phthalate diesters measured in the samples are shown in Table 44. For those analytes for which there was no response in the procedural blank the limits of detection and quantification were calculated as three and ten times the signal to noise ratio of the overspike response. For those analytes for which a response was observed in the procedural blank the limits of detection and quantification were calculated as three and ten times the concentration measured in this sample. The limits of detection and quantification have not been verified. For those samples in which the response was > 10 x the signal to noise ratio in the selected ion chromatogram used for quantification and all confirmation criteria were met the recovery corrected concentration was calculated and this is reported in Table 44. Relative retention times and ion ratios were used to confirm that the responses observed were due to the phthalate diesters. In some cases the criteria defined in the SOP (Annex 1) were not met. For this data the recovery corrected concentration is given but the failure to confirm is noted. This may be due to the response being too low such that the confirmatory ions were close to the limit of detection or the response being due to a co-eluting interference (whole or in-part) giving a response in the target or qualifying ions. For some analyte/matrix combinations no data is present. This may be because of an interference derived from the foodstuff from which the phthalate diester could not be separated or it may be due to failure of the phthalate diester to partition into the organic solvent as a consequence of its solubility in the foodstuff.

In general those samples for which the presence of one or more phthalate diester was confirmed contained low levels of one or more of DBP, DiBP or DEHP. Sample S08-17276, an organic virgin olive oil, contained the highest phthalate concentrations (DiBP = 49 µg/kg, DBP, 57 µg/kg, BBP = 2084 µg/kg and DEHP = 6446 µg/kg).

8. DISCUSSION

This report describes the development and in-house validation of methodology for the determination of phthalate diesters, phthalate monoesters, phthalic acid and total phthalates in foods. The methods encompass a wide range of analytes and have been validated and applied to high fat, high carbohydrate and high protein foodstuffs as well as combinations of all three. TDS sample analysis detected low levels of DEP, DiBP, DBP, BBP and DEHP and these same phthalate diesters were detected when 261 retail foodstuffs were analysed for these compounds. Low levels of MBP and MEHP were detected in several of the TDS animal/fish based samples wherein metabolism of any diesters to which they have been exposed may be expected. Phthalates may be present in foods as a result of migration from food contact materials and articles. Studies carried out in this project confirmed that phthalates in foods could be derived from migration from food packaging materials.

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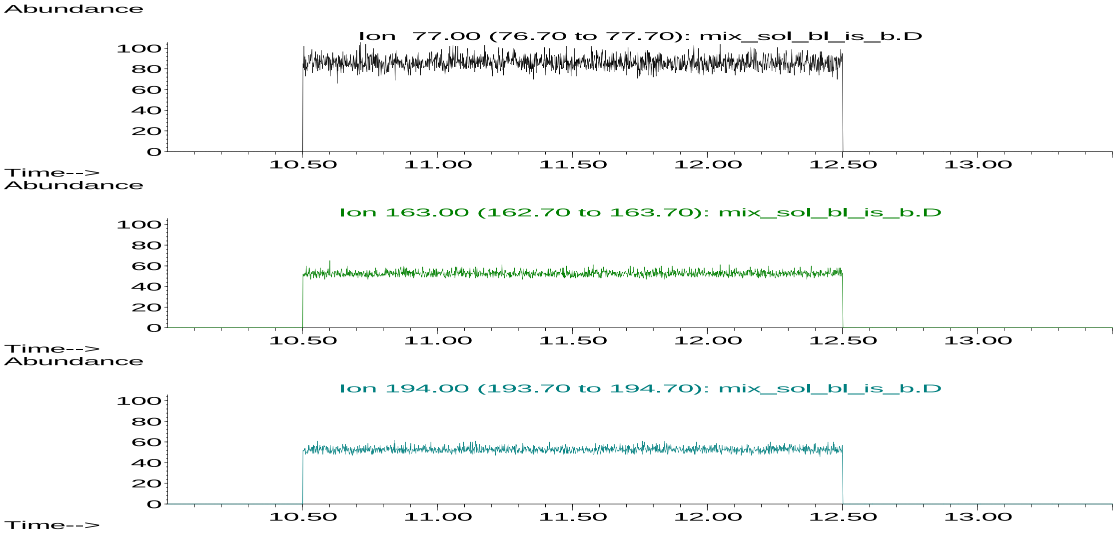
Figure 1. Dimethyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

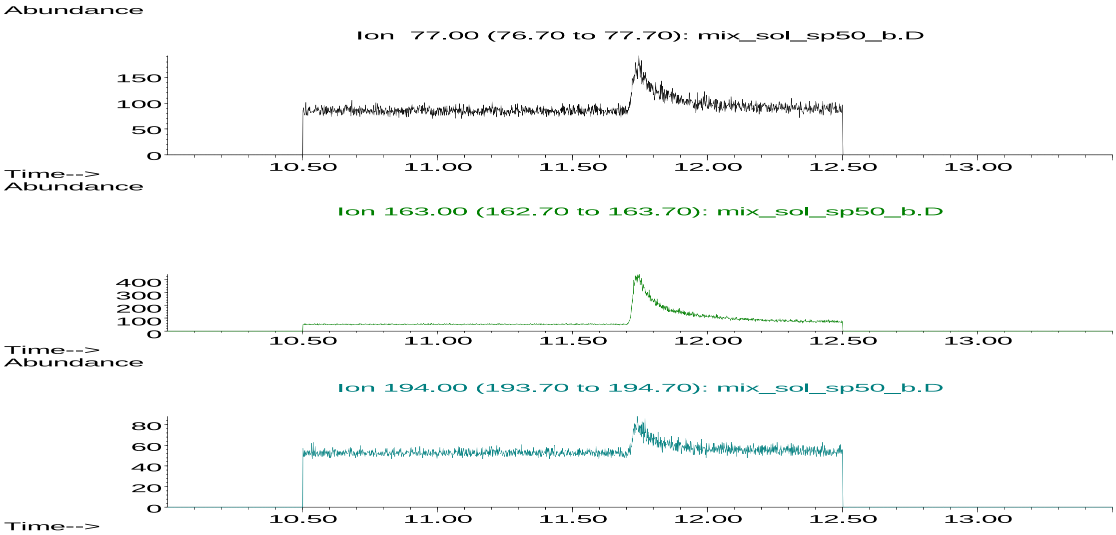
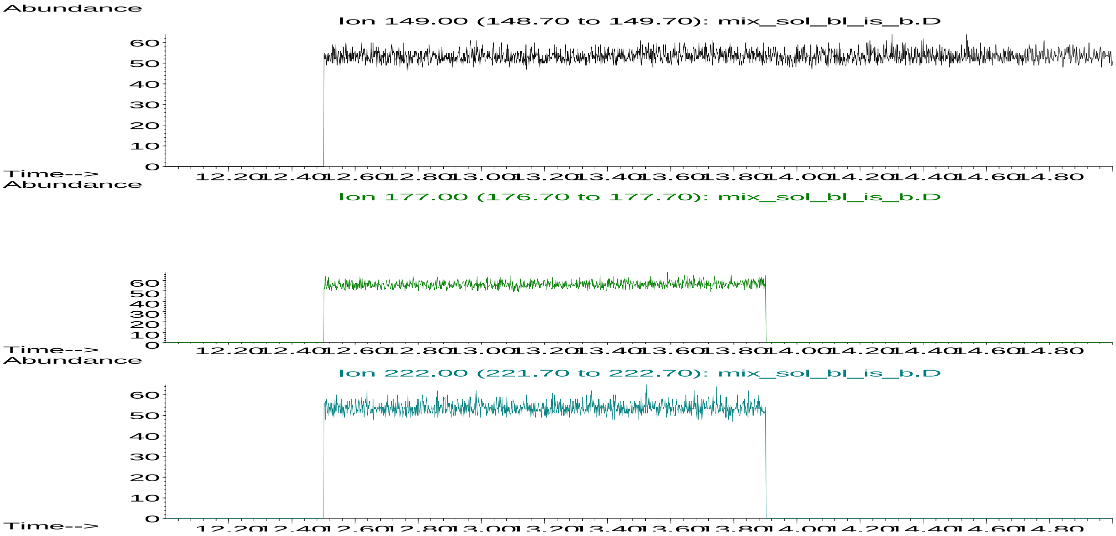


Figure 2. Diethyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

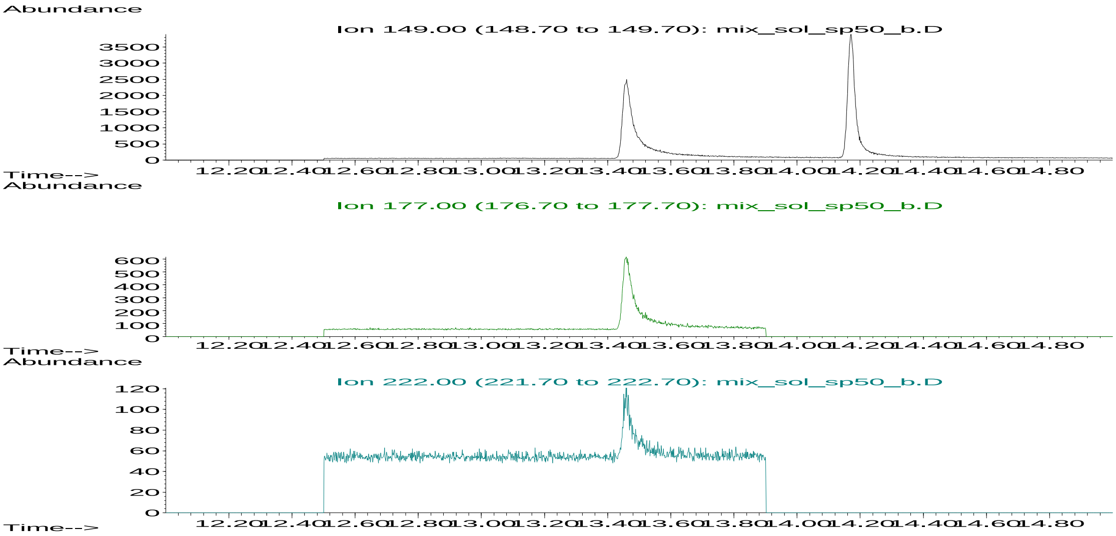
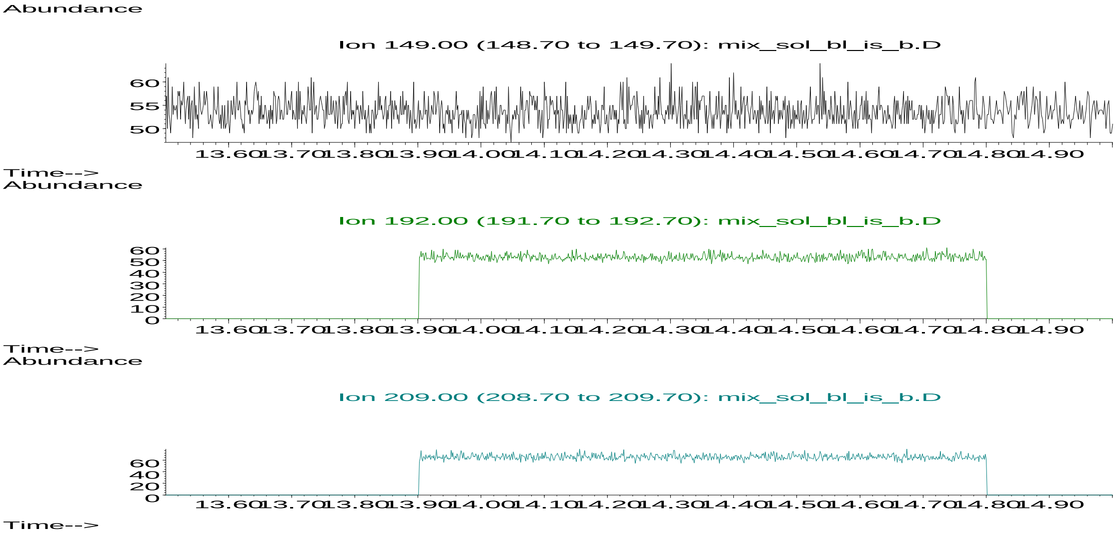


Figure 3. Diisopropyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

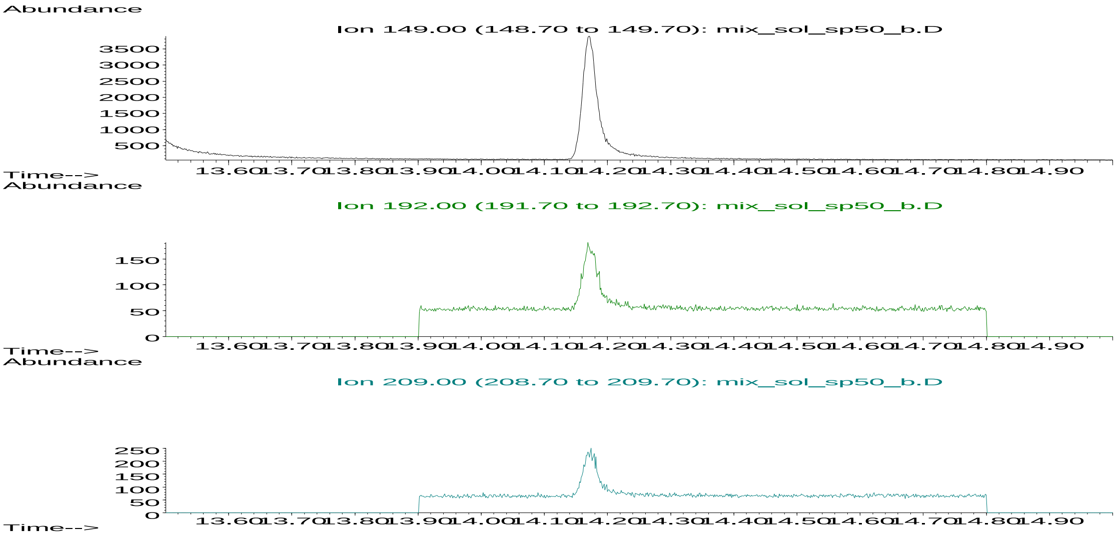


Figure 4. Diallyl phthalate in the procedural blank

(a) Procedural blank

(b) Procedural blank + overspike at 0.05 mg/kg

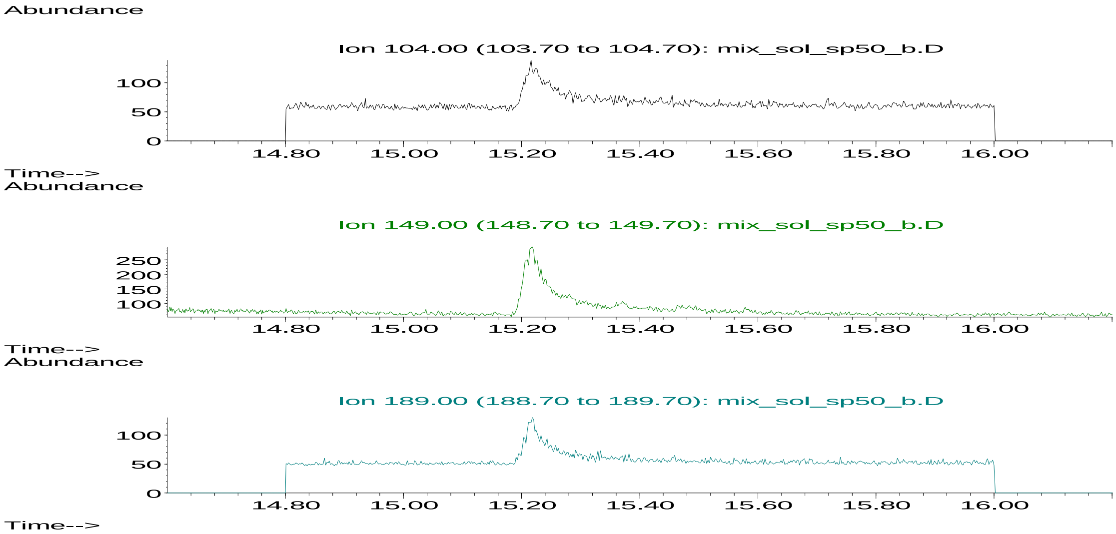
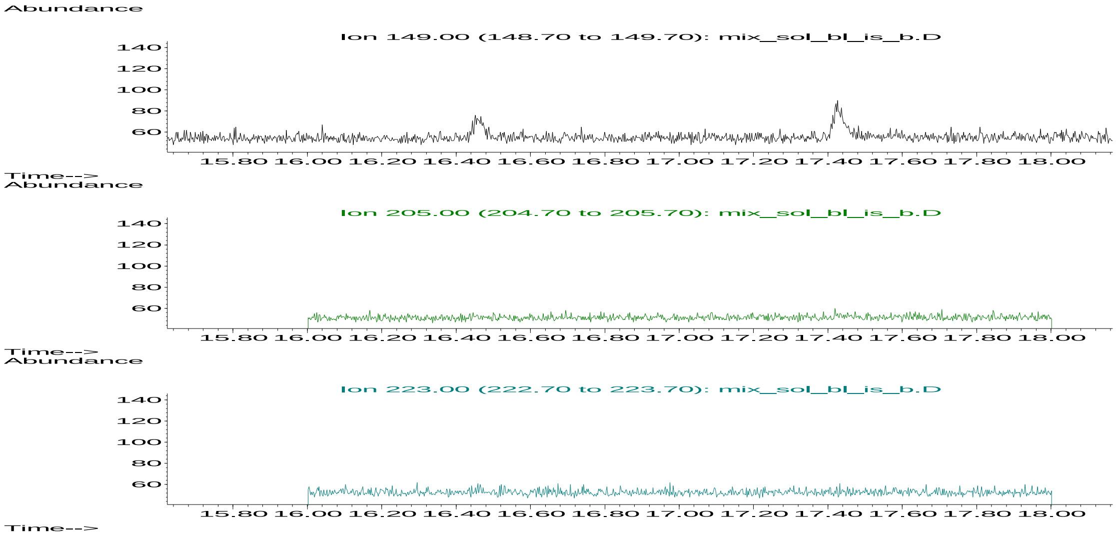


Figure 5. Diisobutyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

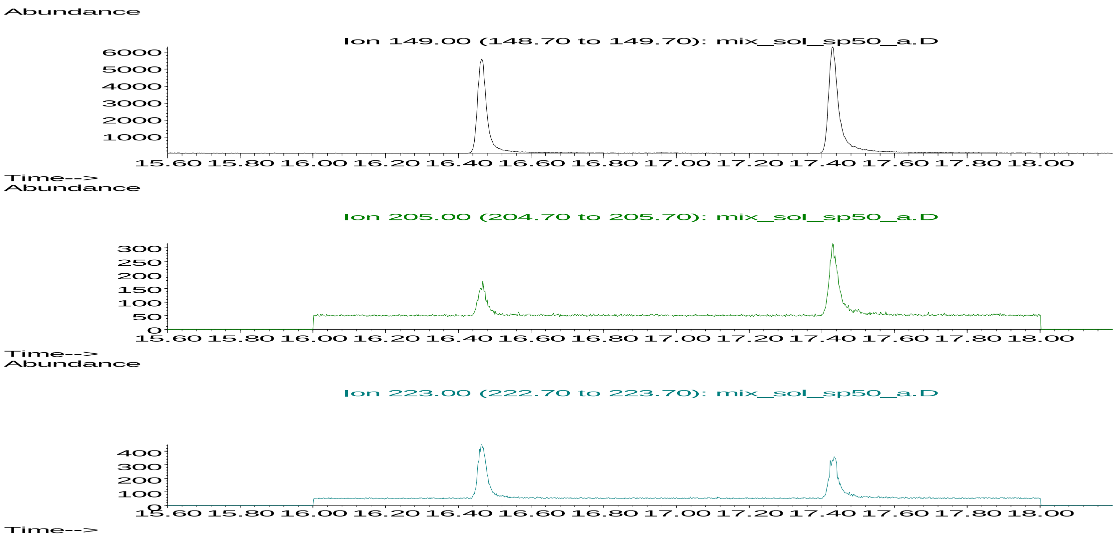
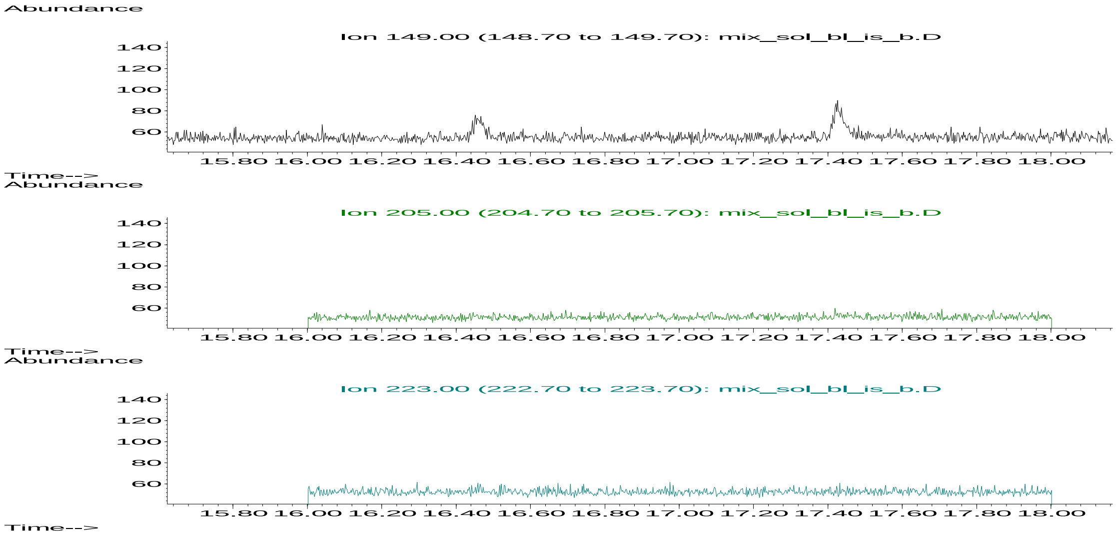


Figure 6. Dibutyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

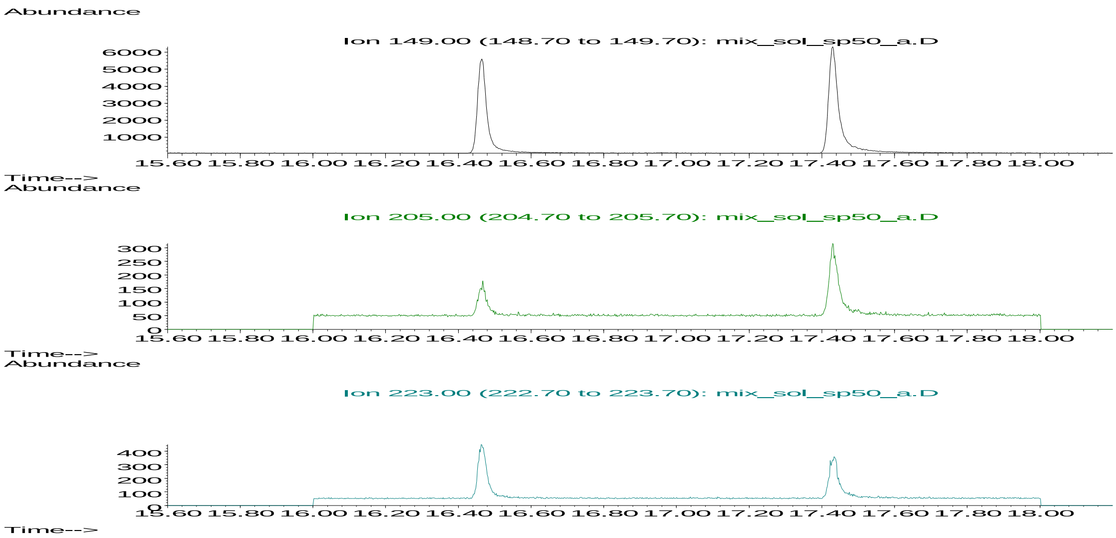
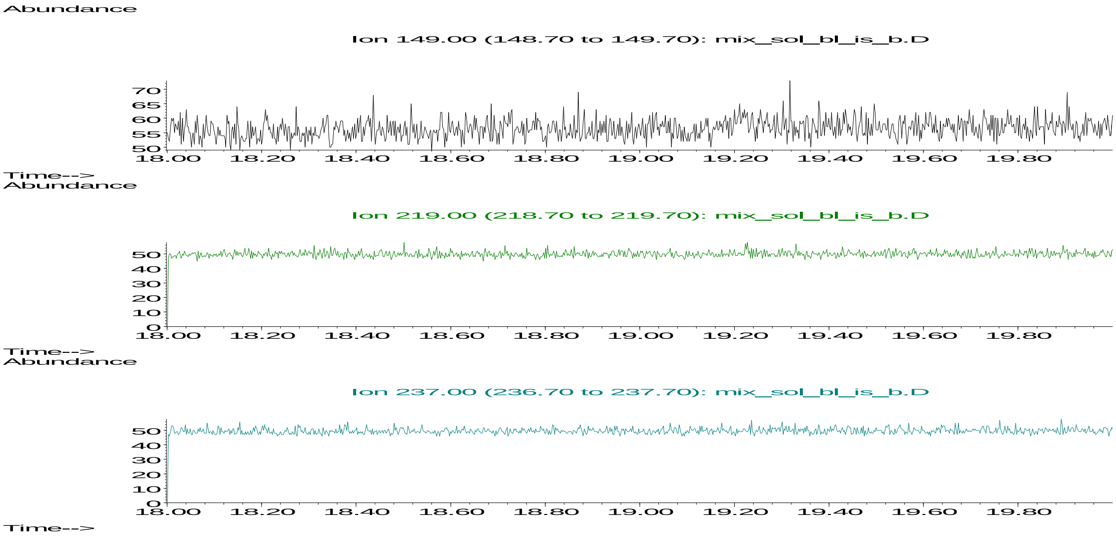


Figure 7. Dipentyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

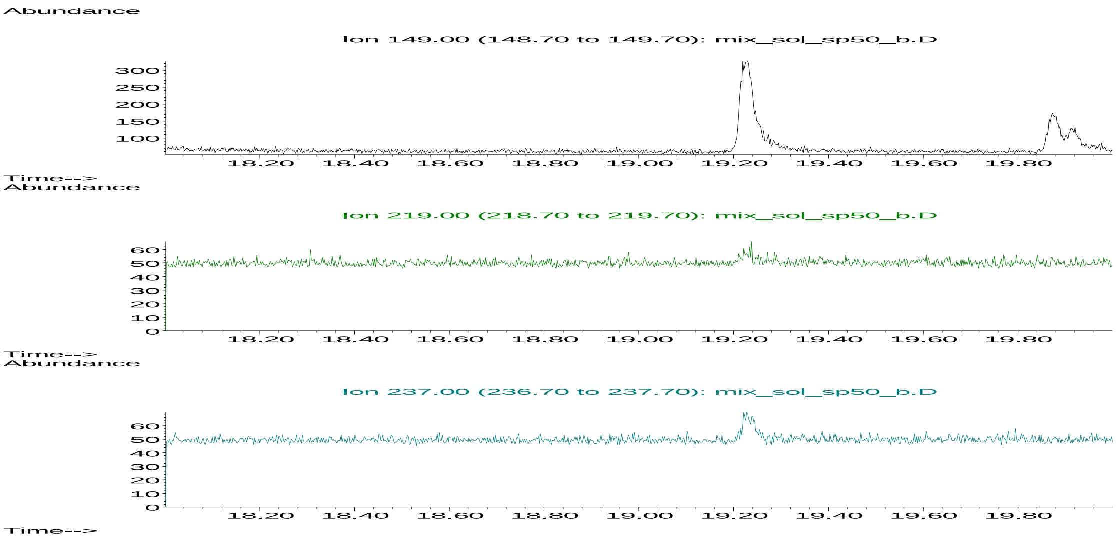
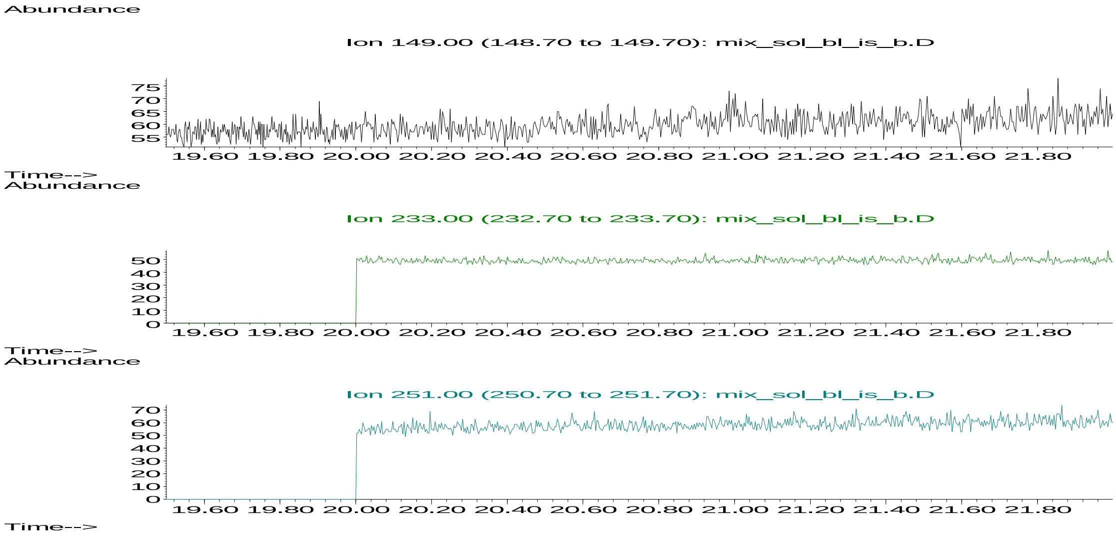


Figure 8. Di-n-hexyl phthalate in the procedural blank

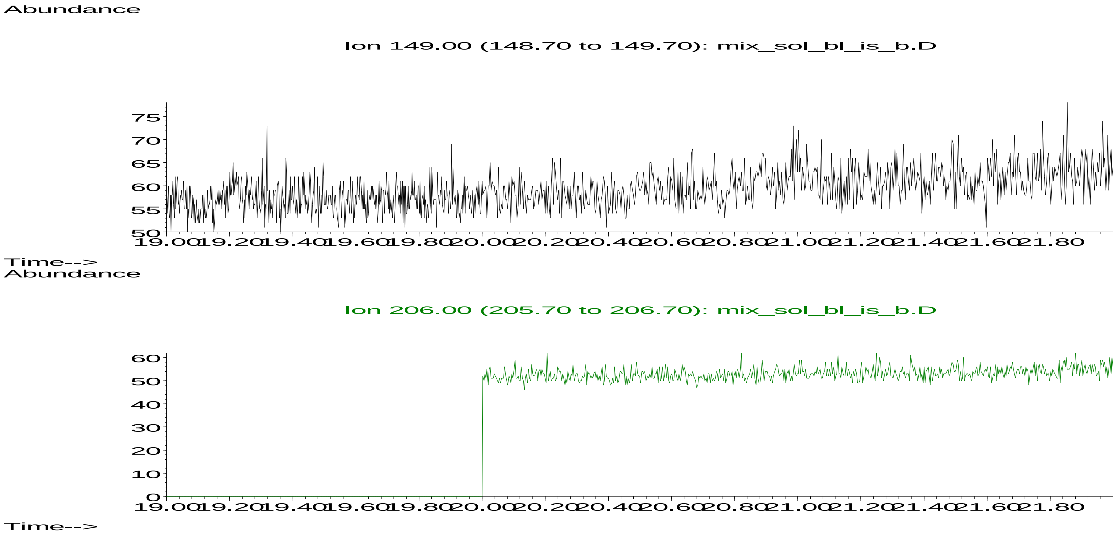
(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

Figure 9. Benzyl butyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

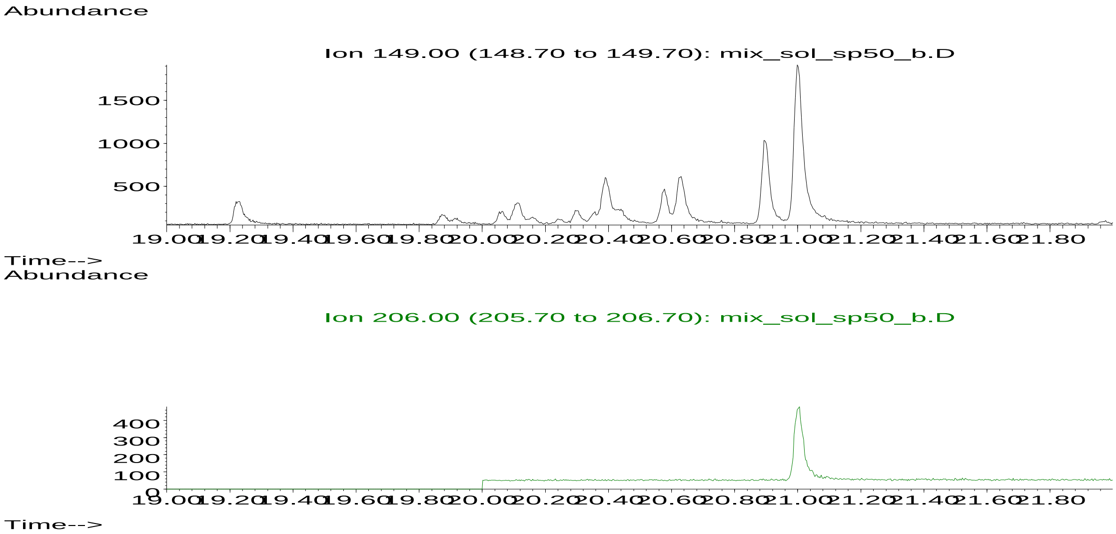
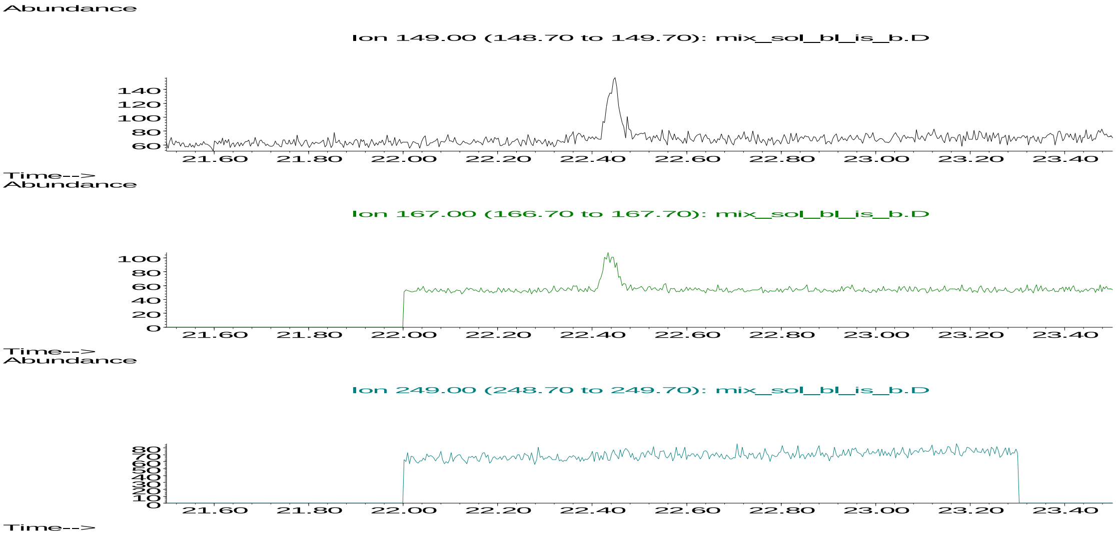


Figure 10. Dicyclohexyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

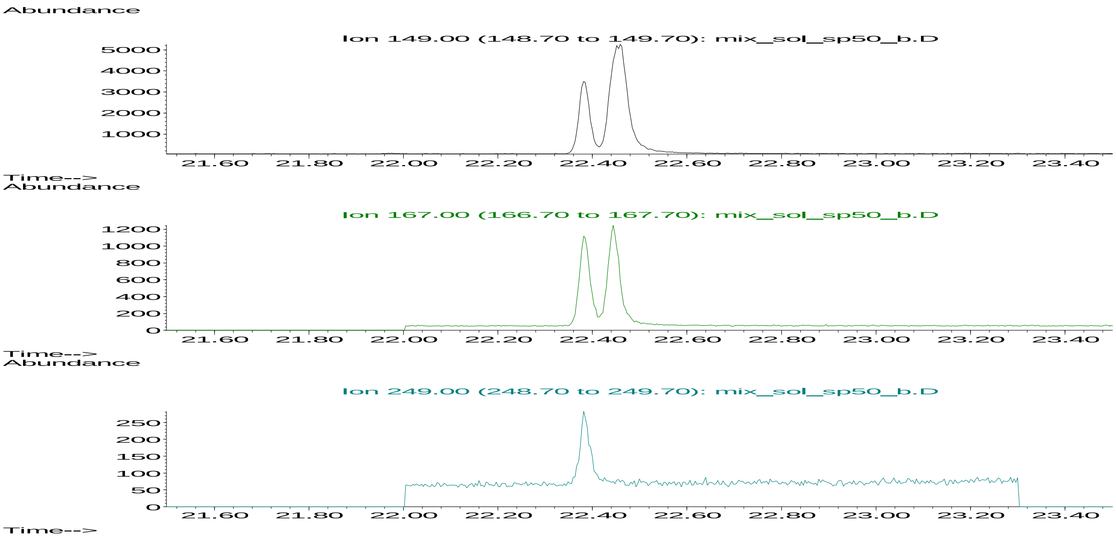
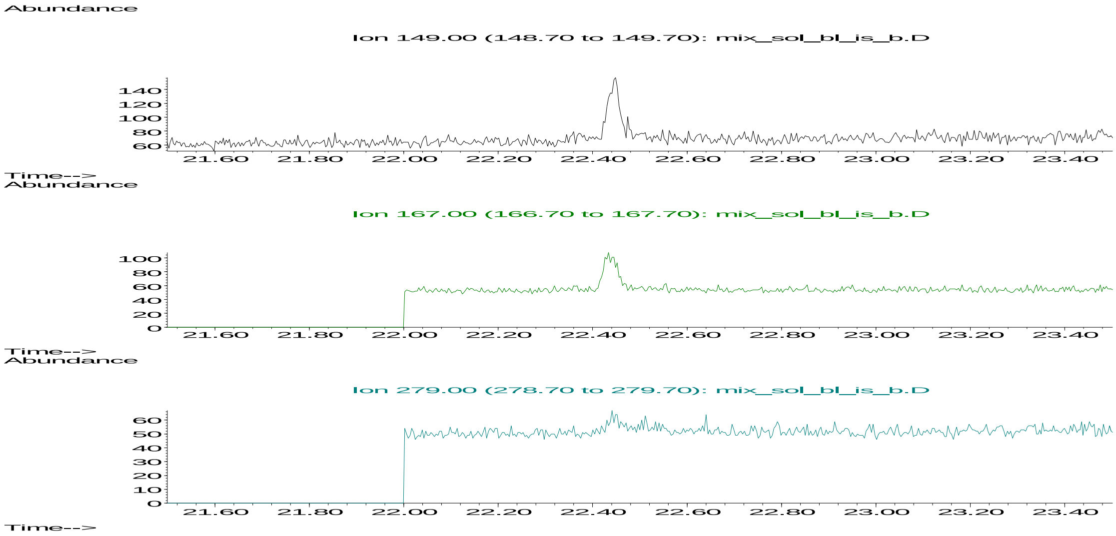


Figure 11. Di-(2-ethylhexyl) phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

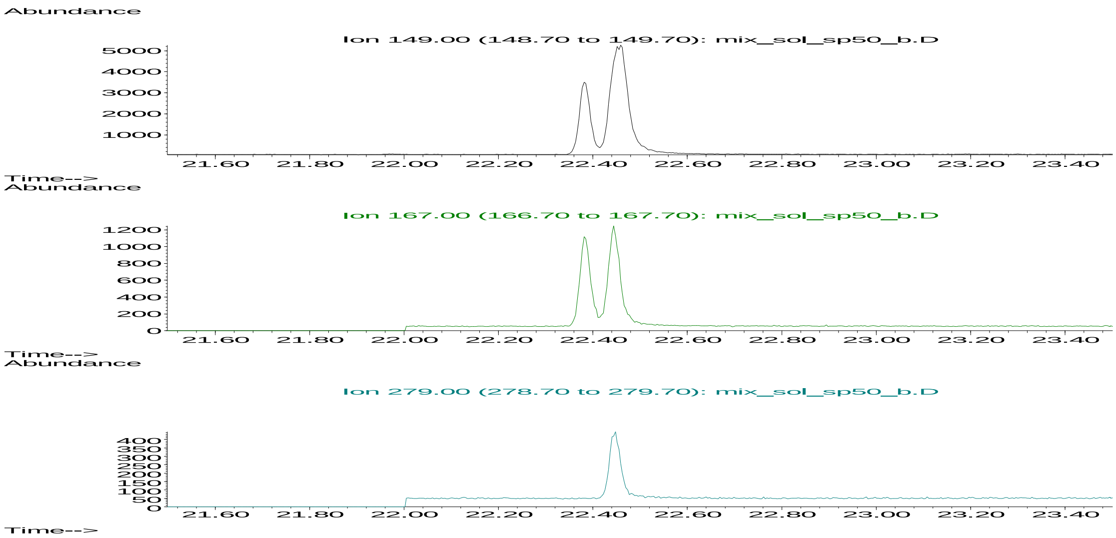
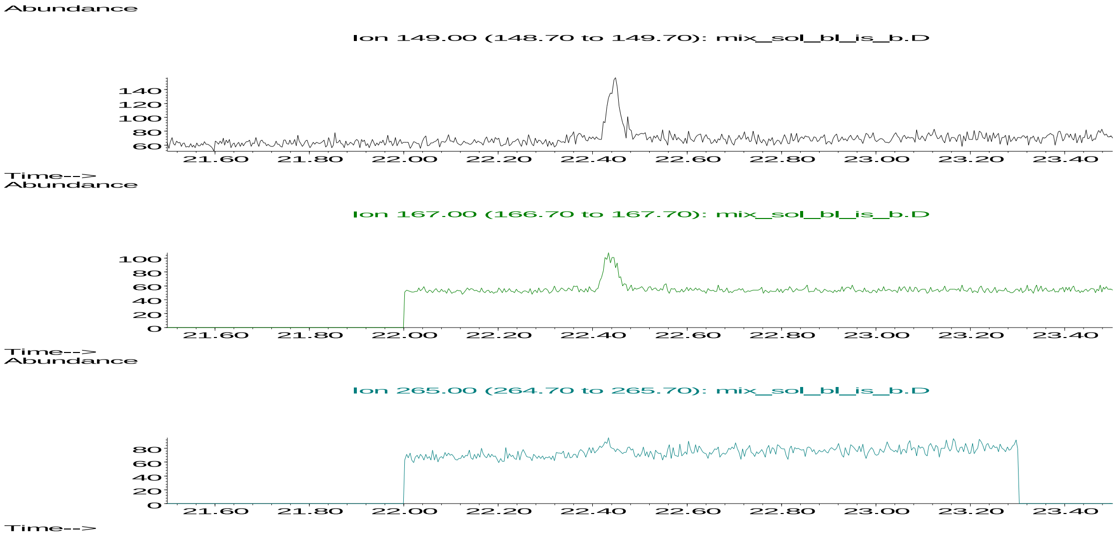


Figure 12. Di-n-heptyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

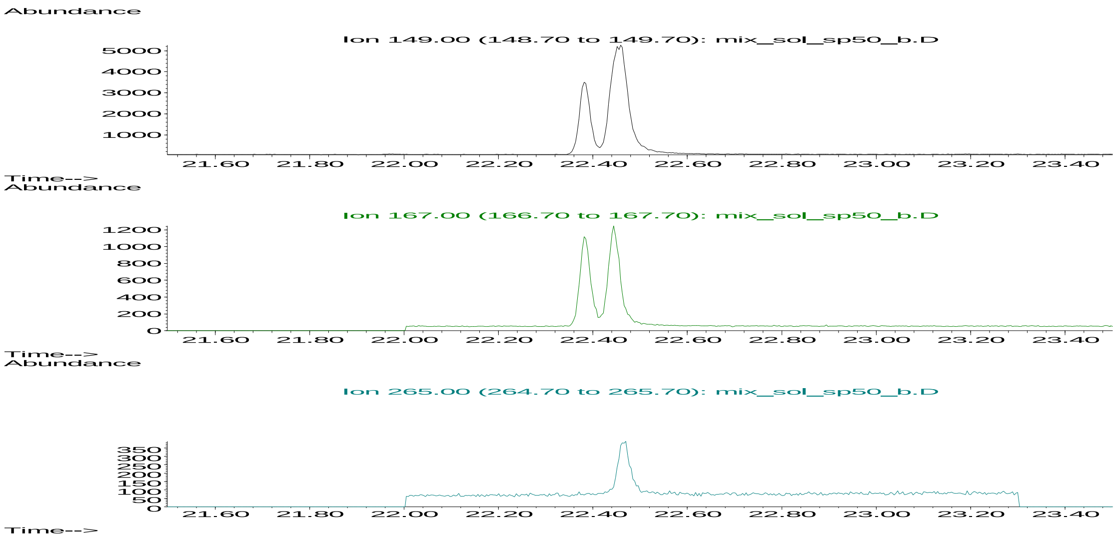
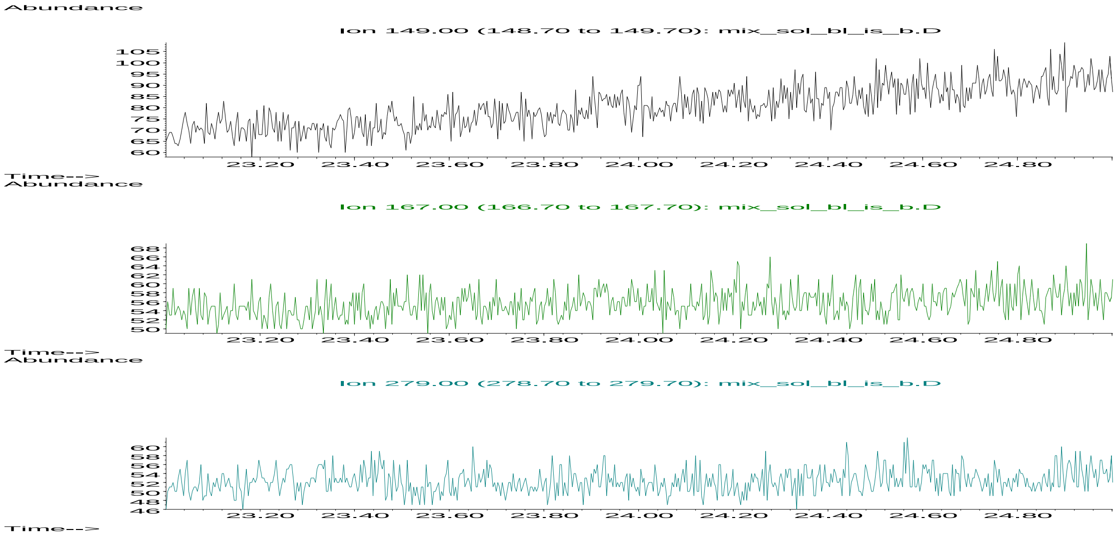


Figure 13. Di-n-octyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

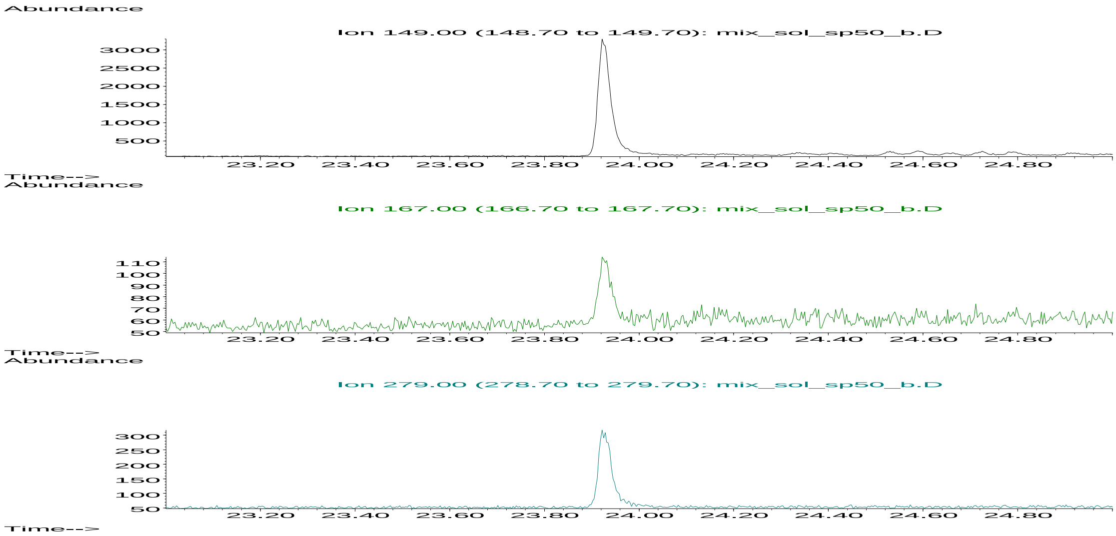
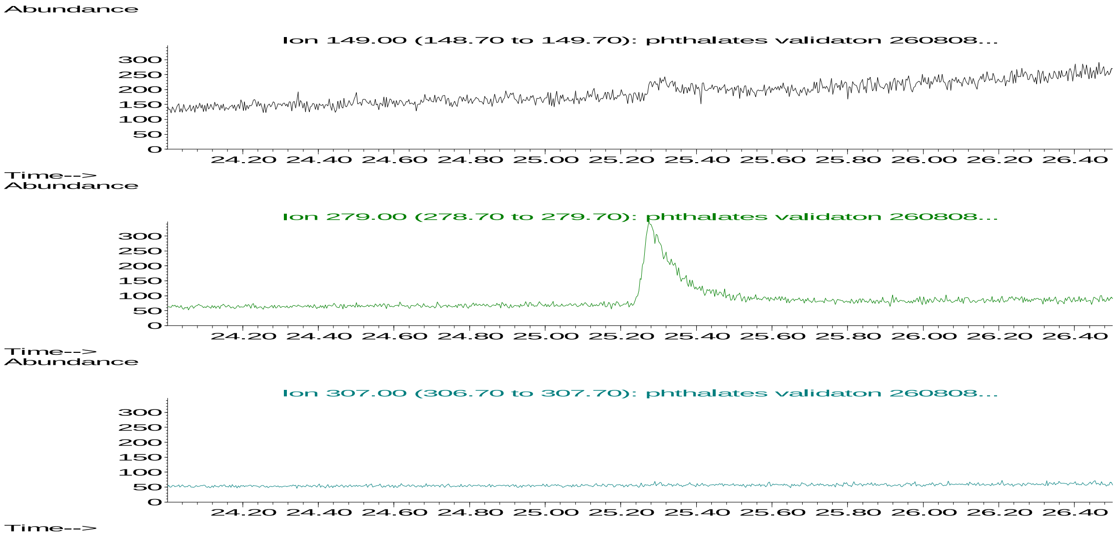


Figure 14. n-Octyl-n-decyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

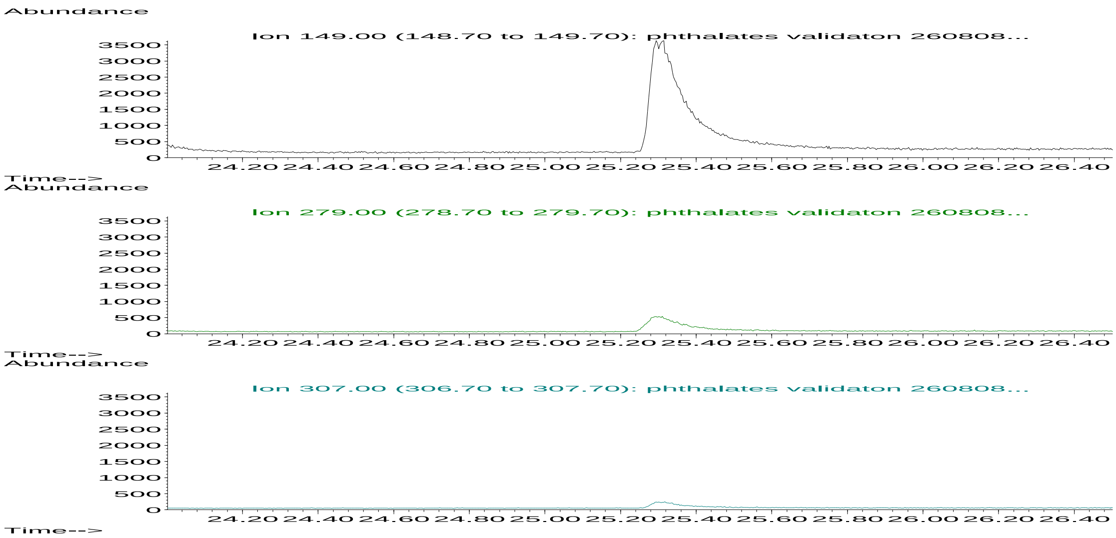
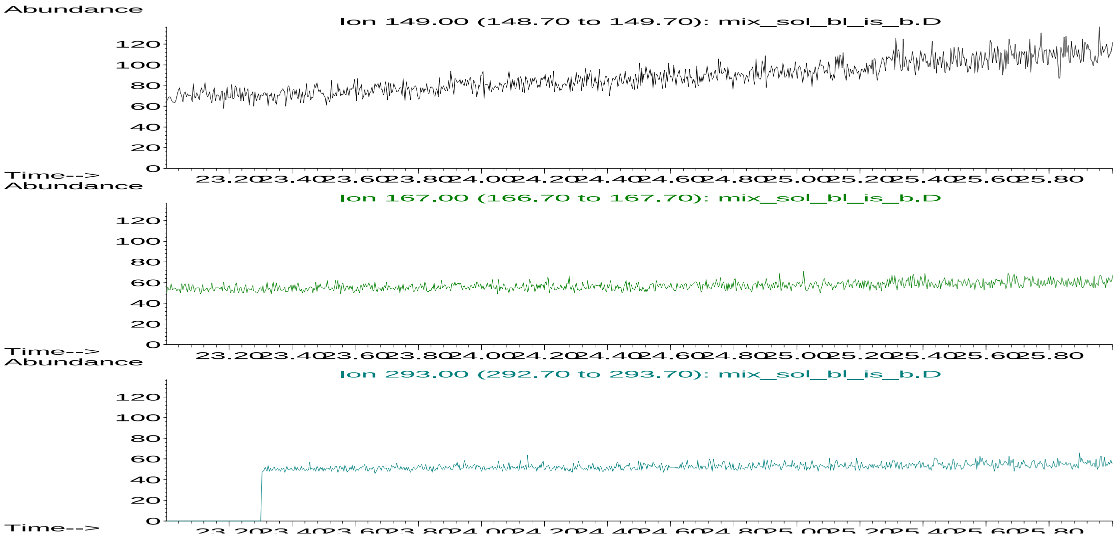
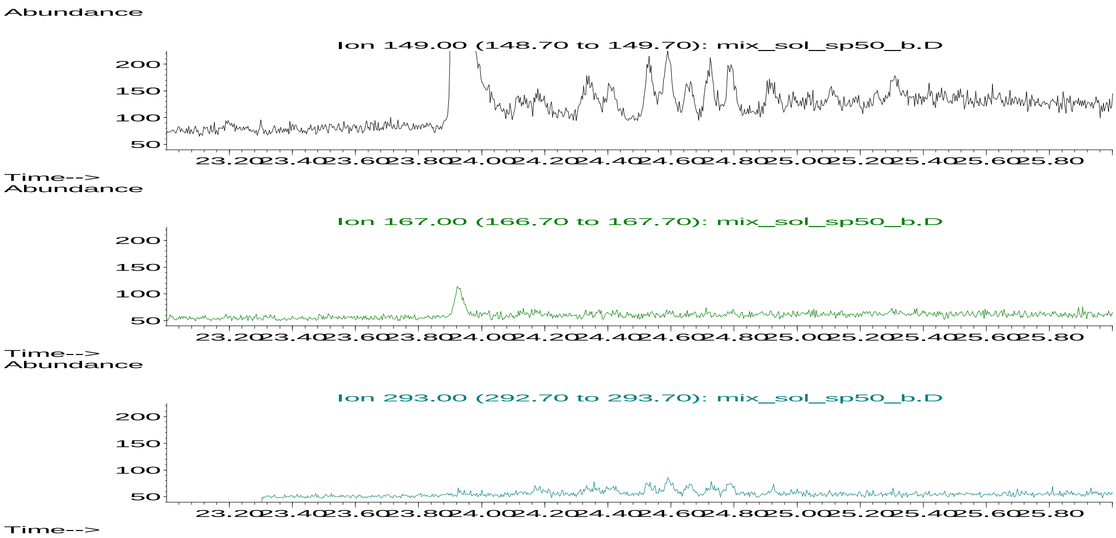


Figure 15. Diisononyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg



(c) Procedural blank + overspike at 0.3 mg/kg

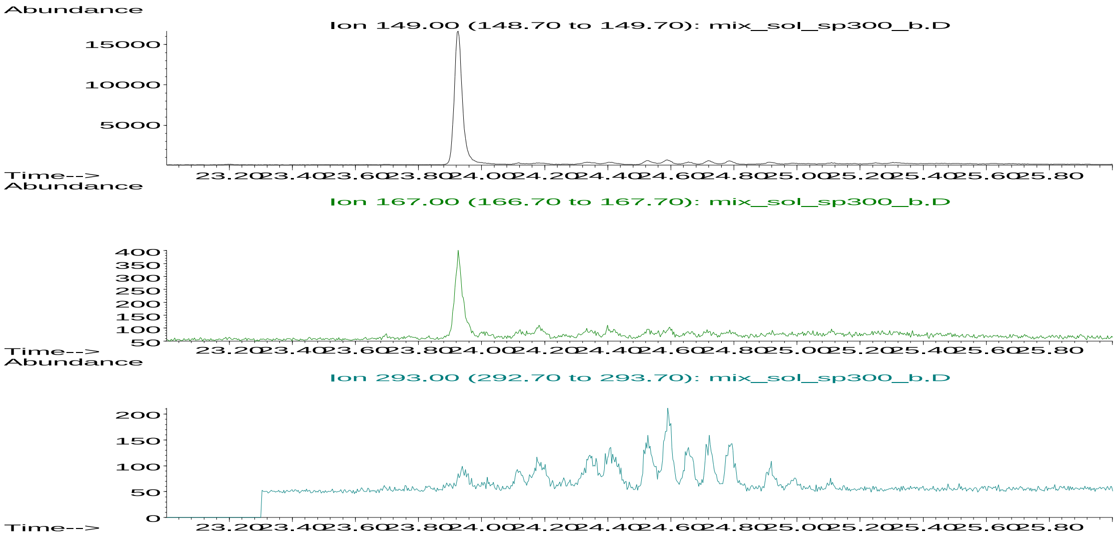
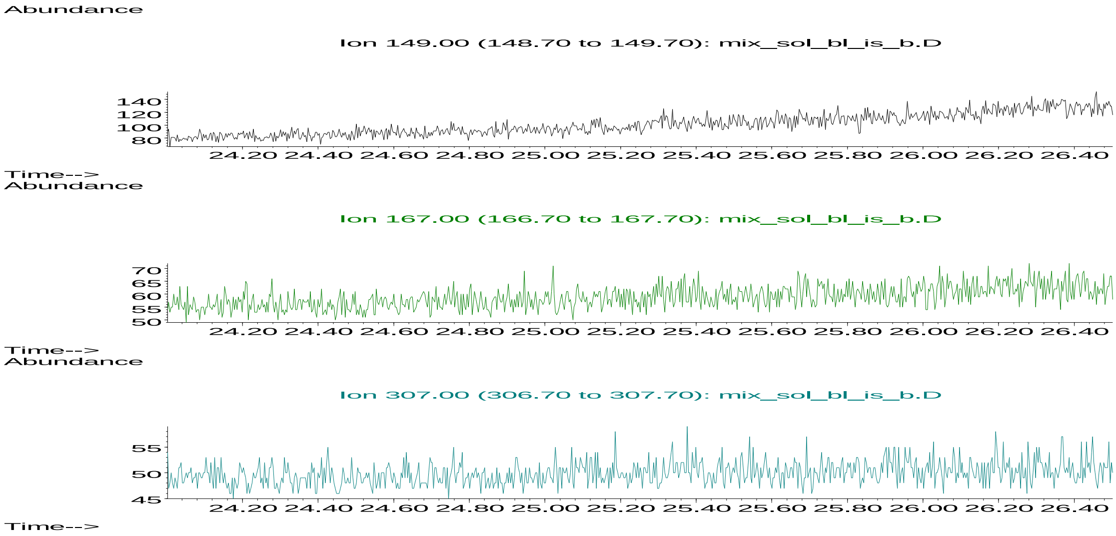


Figure 16. Diisodecyl phthalate in the procedural blank

(a) Procedural blank

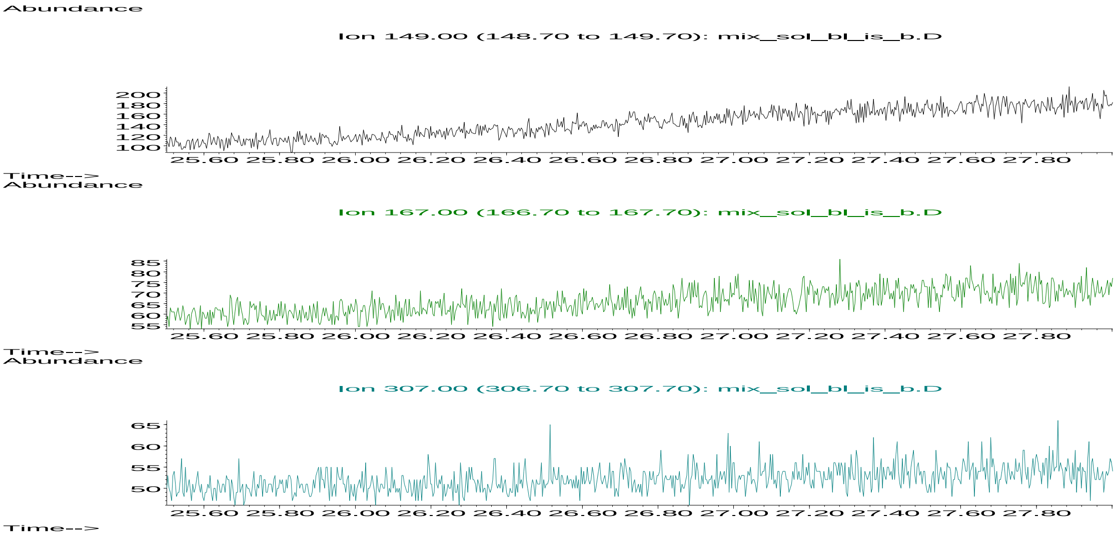


(b) Procedural blank + overspike at 0.05 mg/kg

(c) Procedural blank + overspike at 0.3 mg/kg

Figure 17. Di-n-decyl phthalate in the procedural blank

(a) Procedural blank



(b) Procedural blank + overspike at 0.05 mg/kg

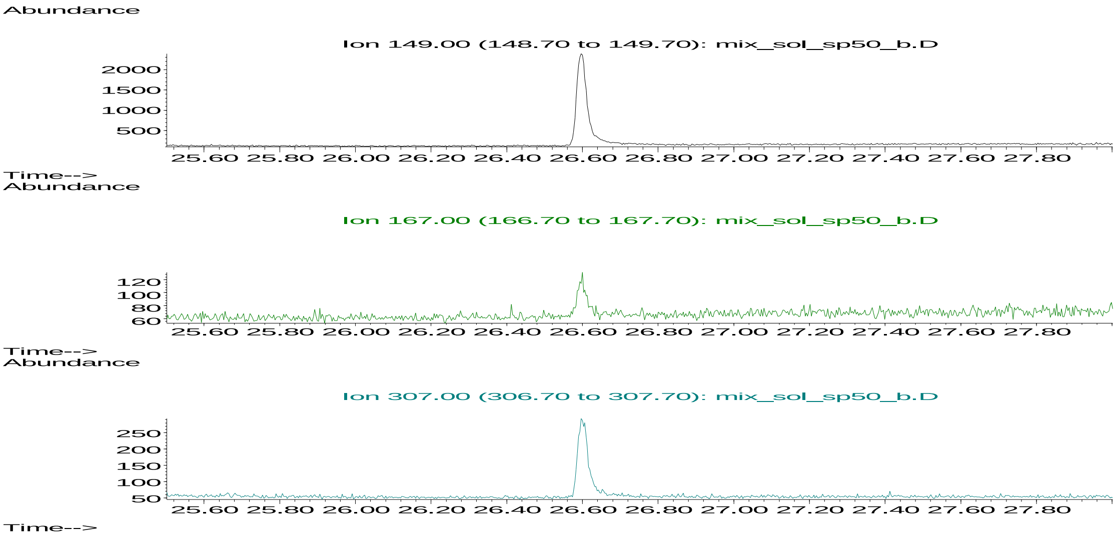
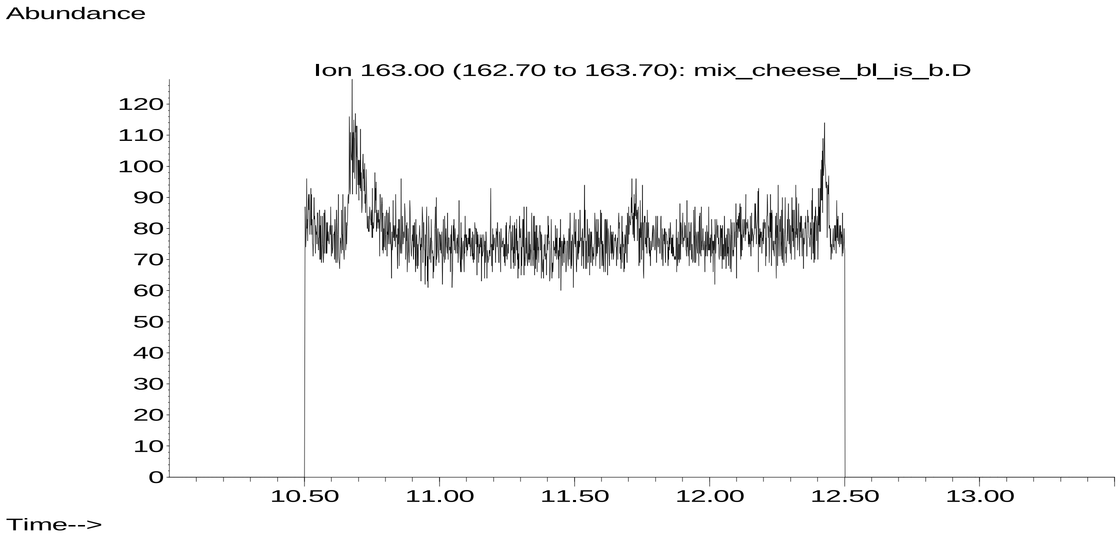
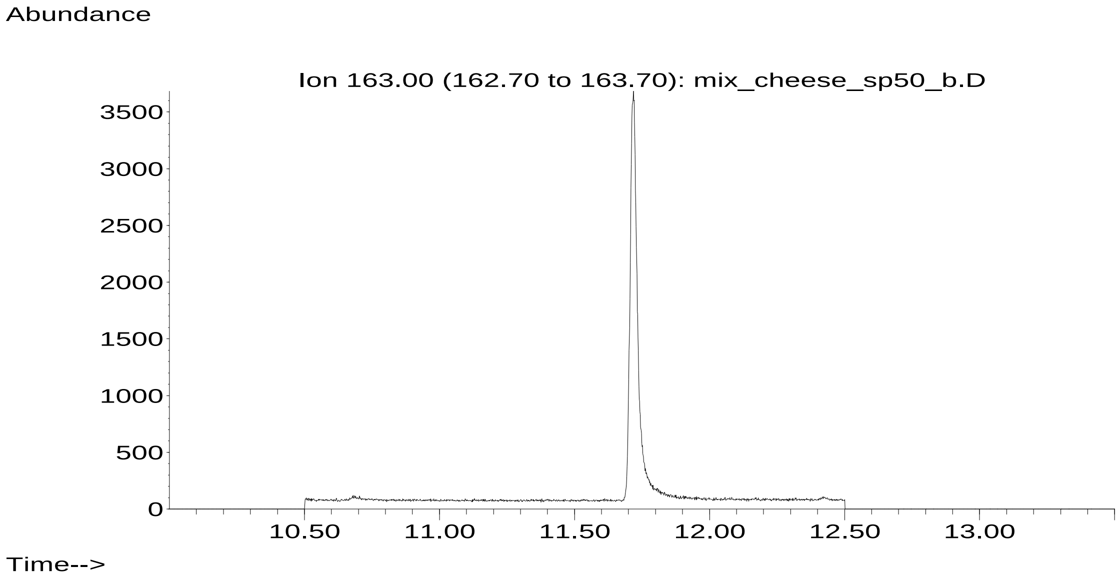


Figure 18. Dimethyl phthalate spiked into foods at the target LOQ

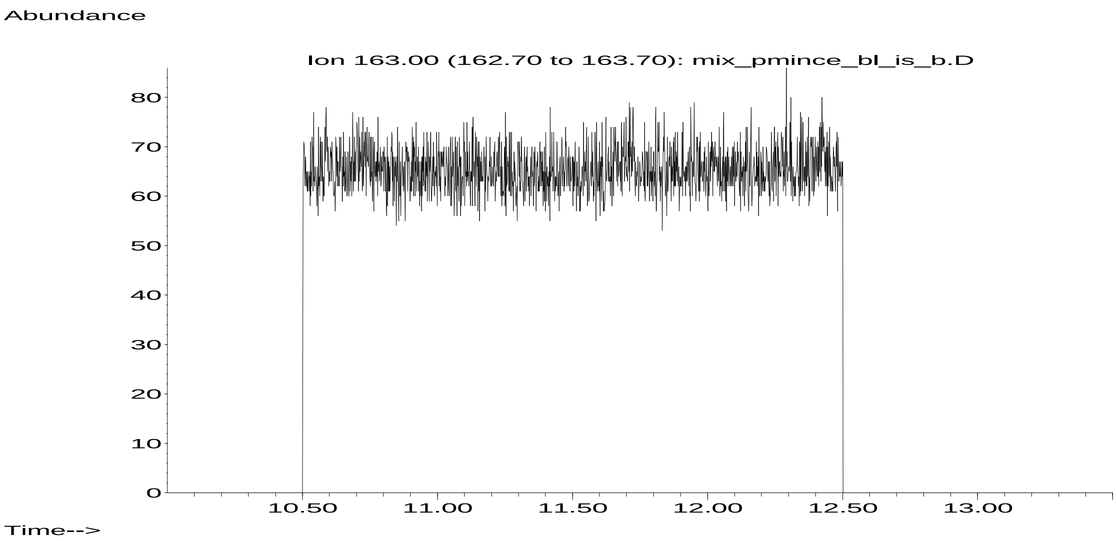
(a) Cheese only



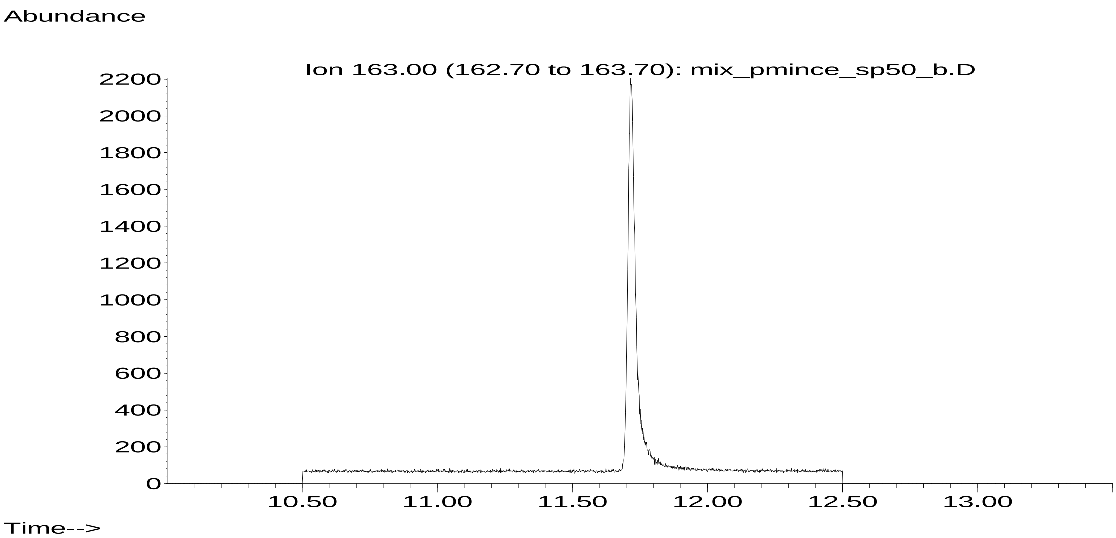
(b) Cheese overspiked with dimethyl phthalate at 0.05 mg/kg



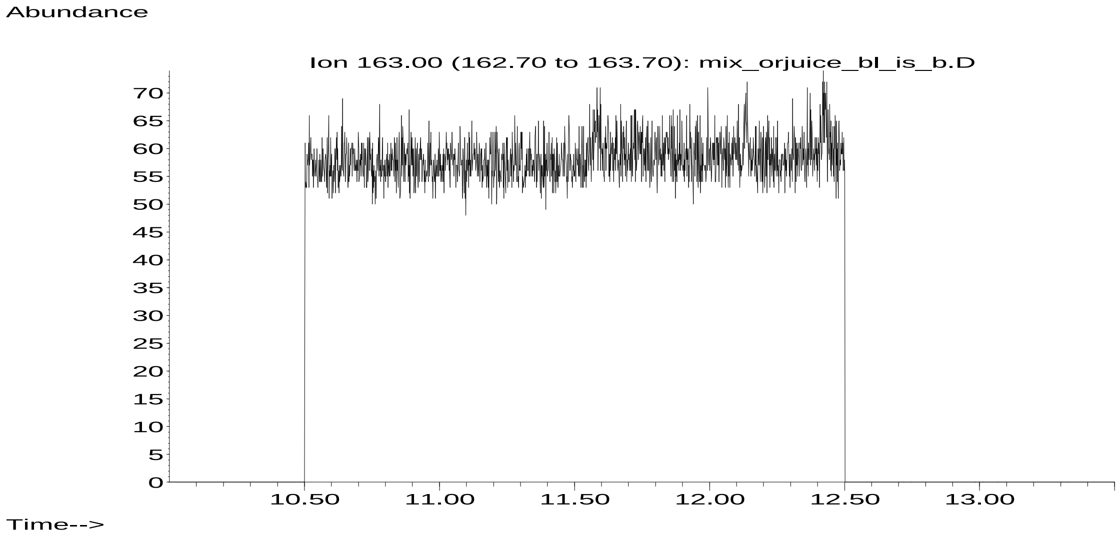
(c) Pork mince only



(d) Pork mince overspiked with dimethyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with dimethyl phthalate at 0.05 mg/kg

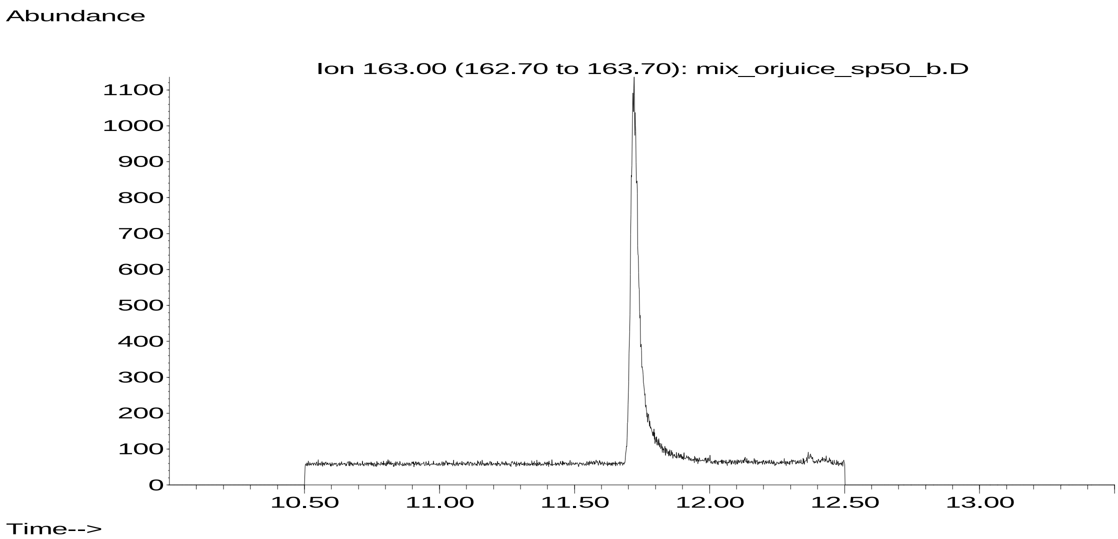
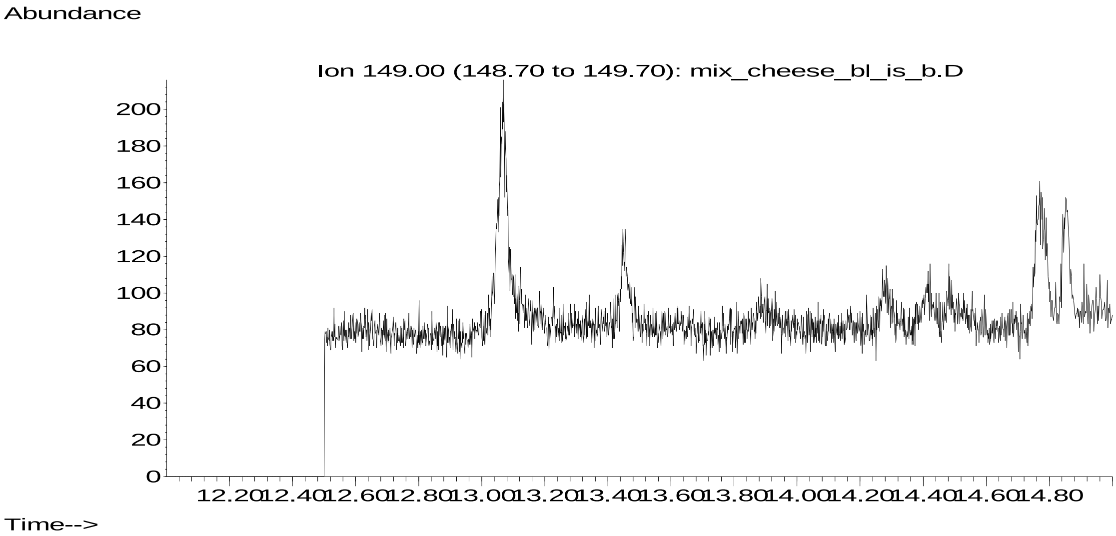
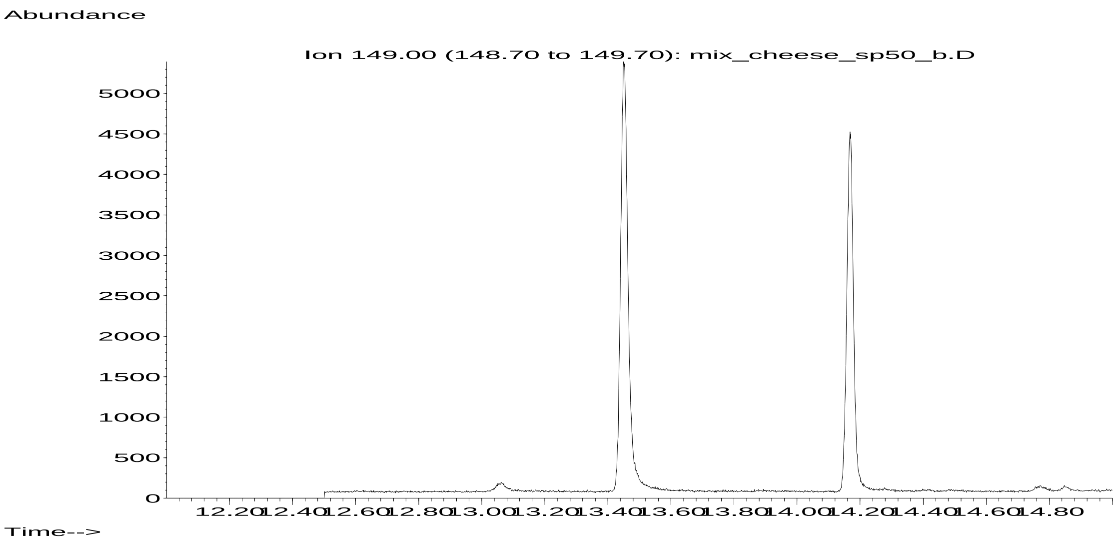


Figure 19. Diethyl phthalate spiked into foods at the target LOQ

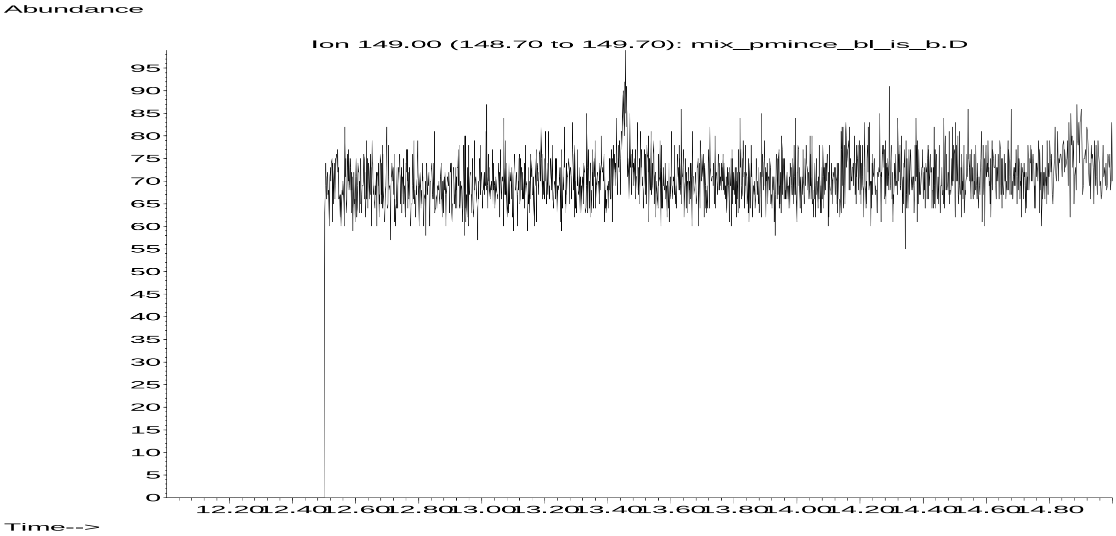
(a) Cheese only



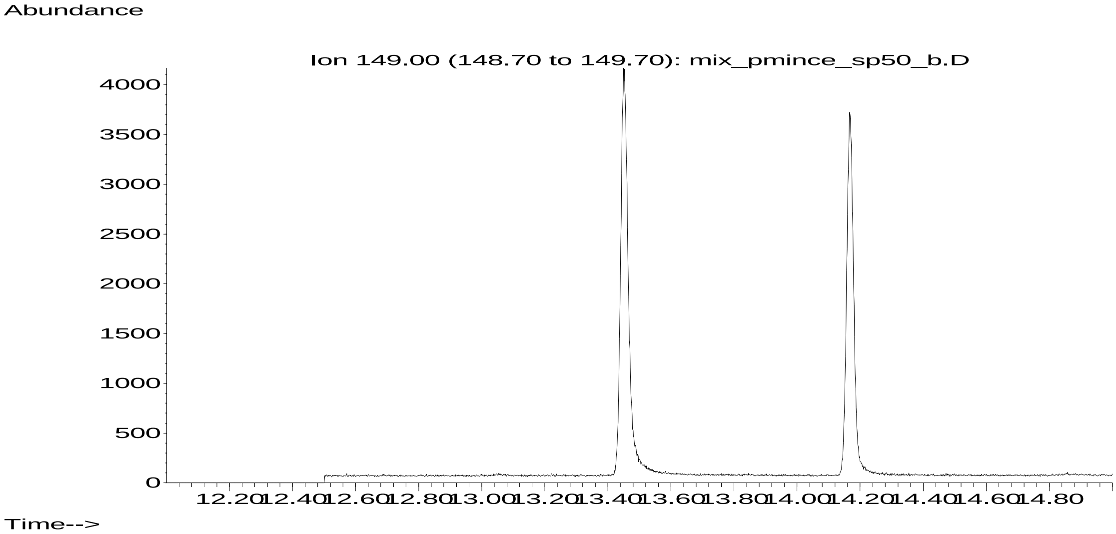
(b) Cheese overspiked with diethyl phthalate at 0.05 mg/kg



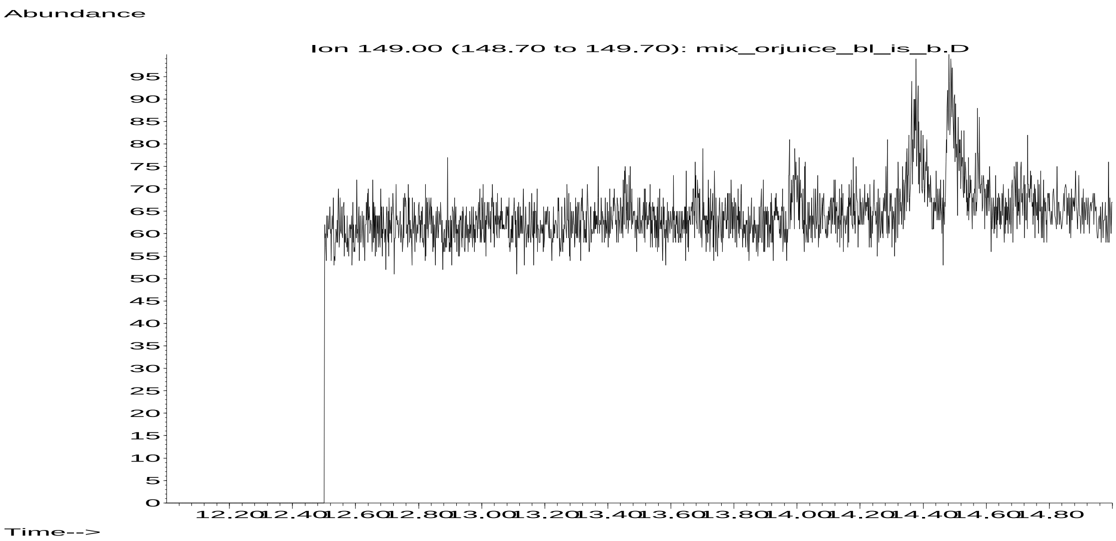
(c) Pork mince only



(d) Pork mince overspiked with diethyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with diethyl phthalate at 0.05 mg/kg

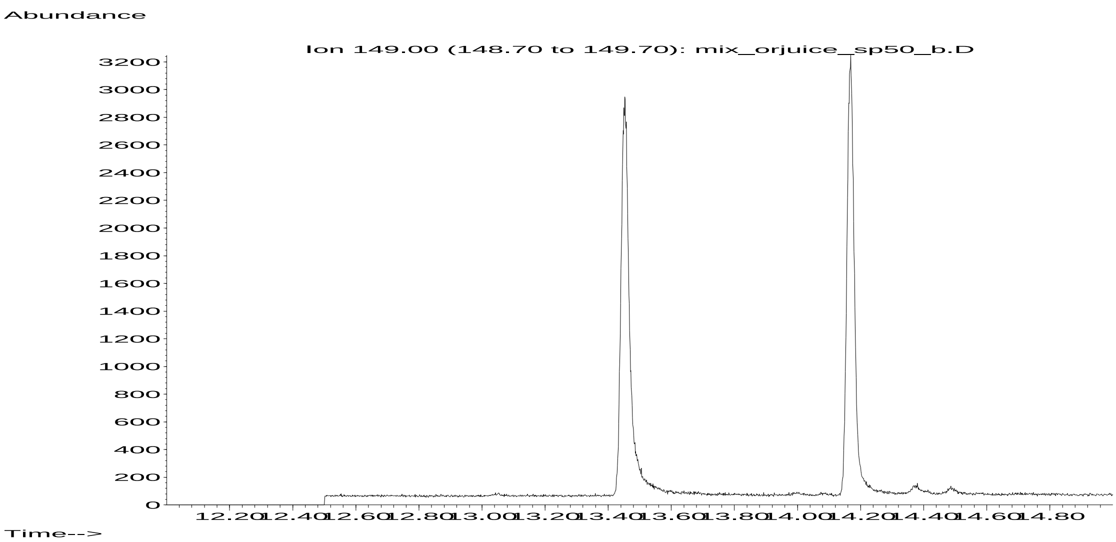
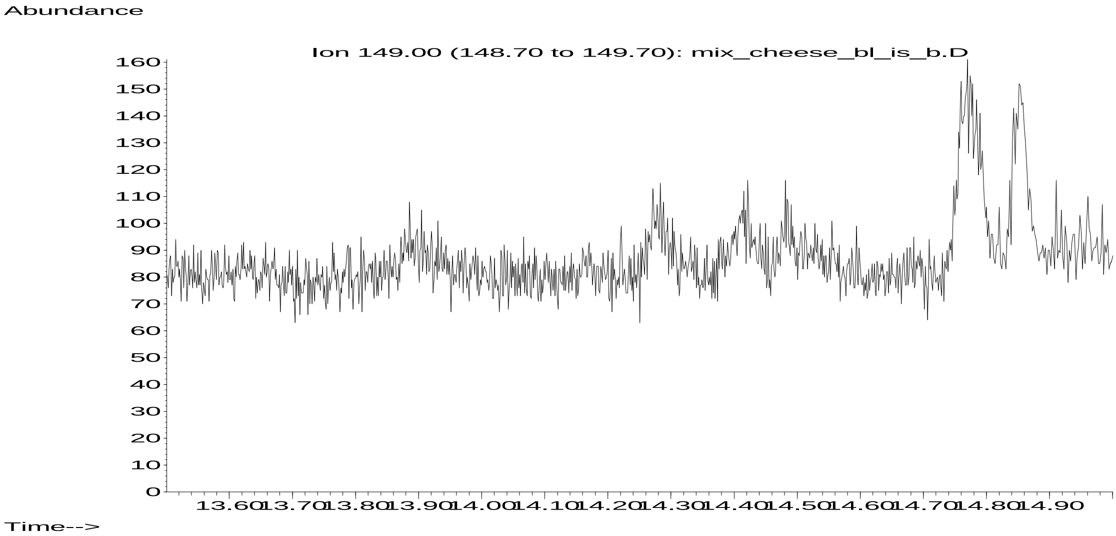
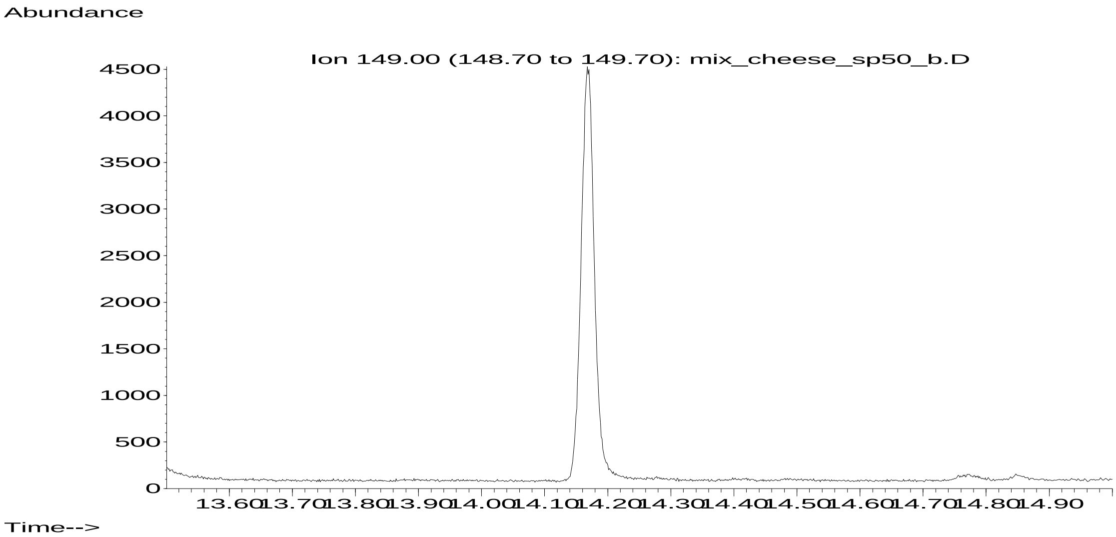


Figure 20. Diisopropyl phthalate spiked into foods at the target LOQ

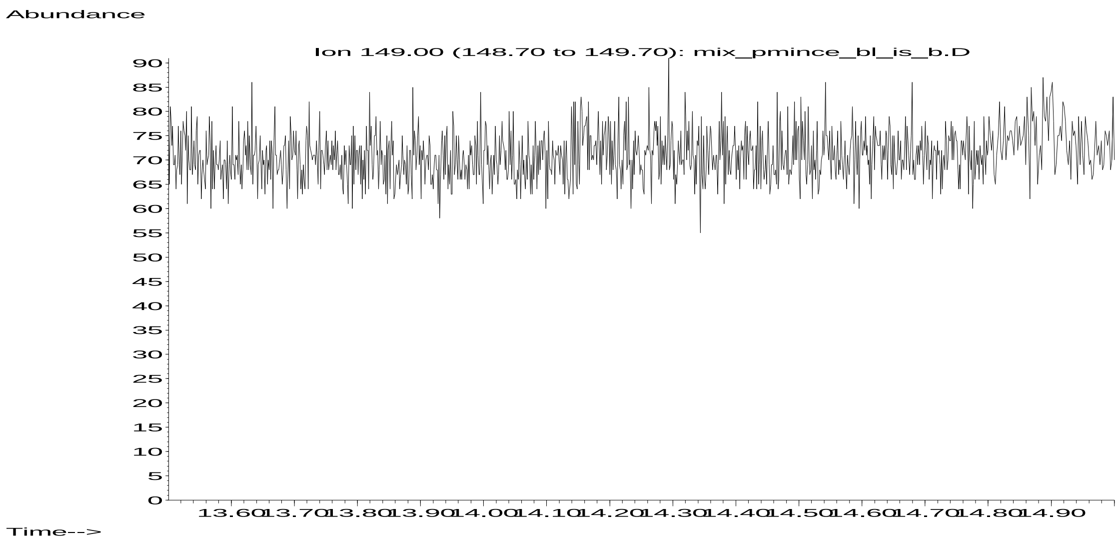
(a) Cheese only



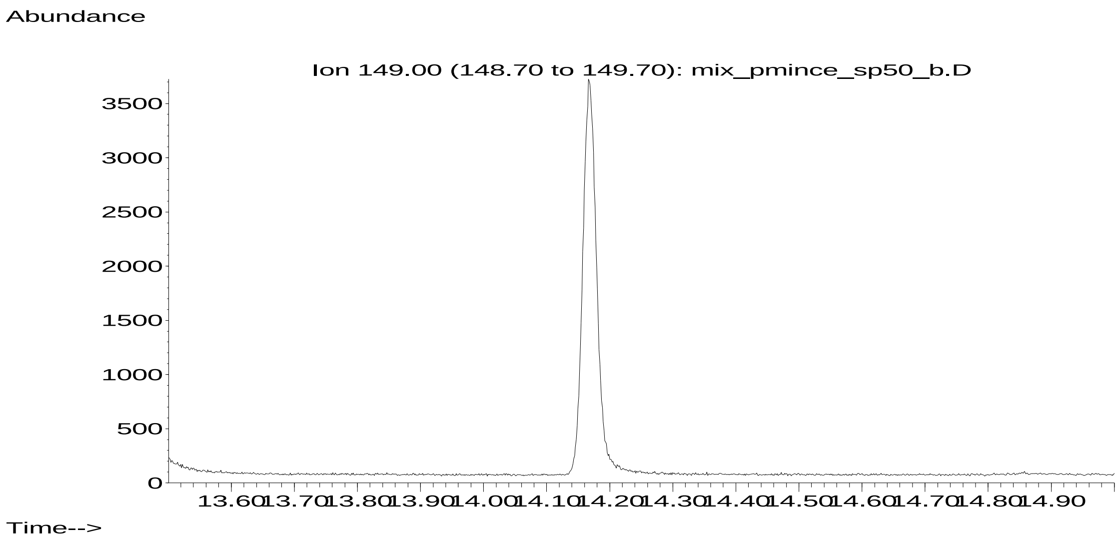
(b) Cheese overspiked with diisopropyl phthalate at 0.05 mg/kg



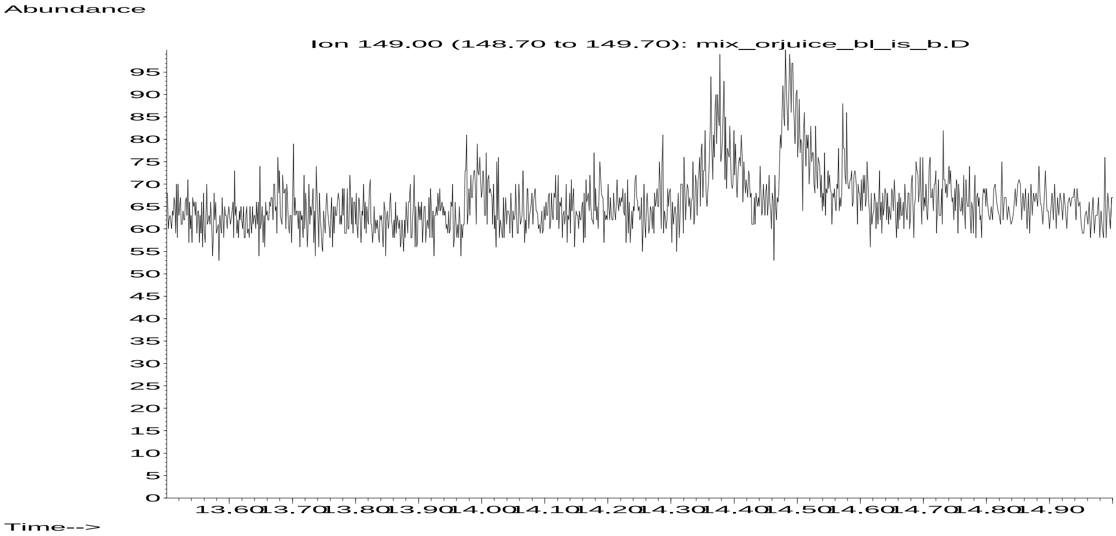
(c) Pork mince only



(d) Pork mince overspiked with diisopropyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with diisopropyl phthalate at 0.05 mg/kg

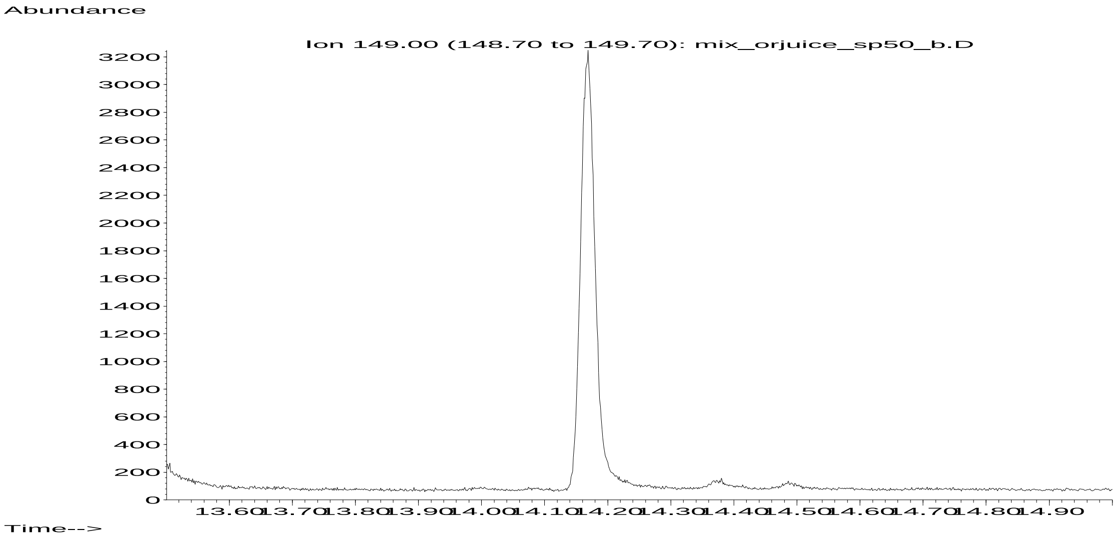
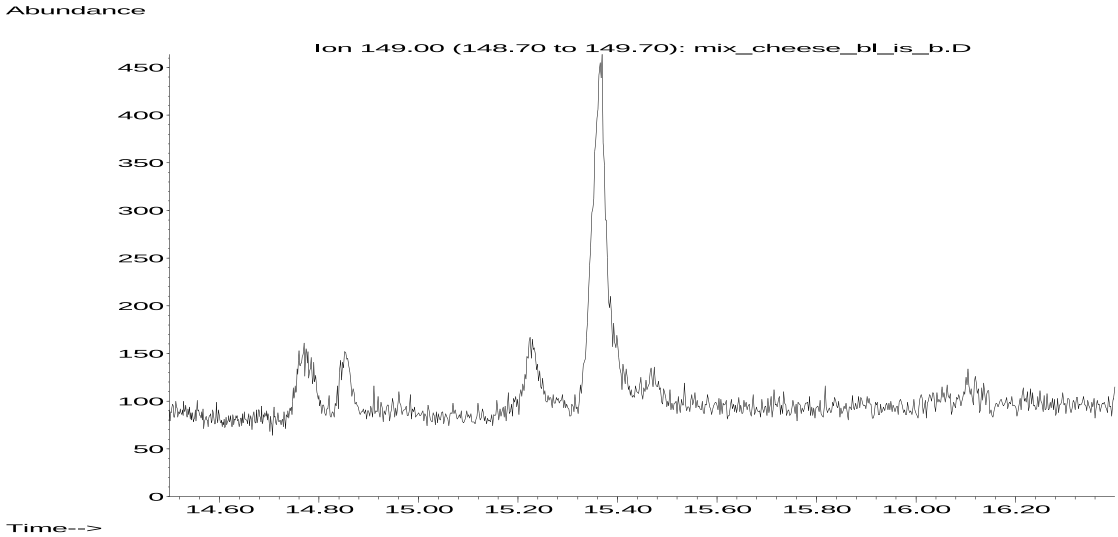
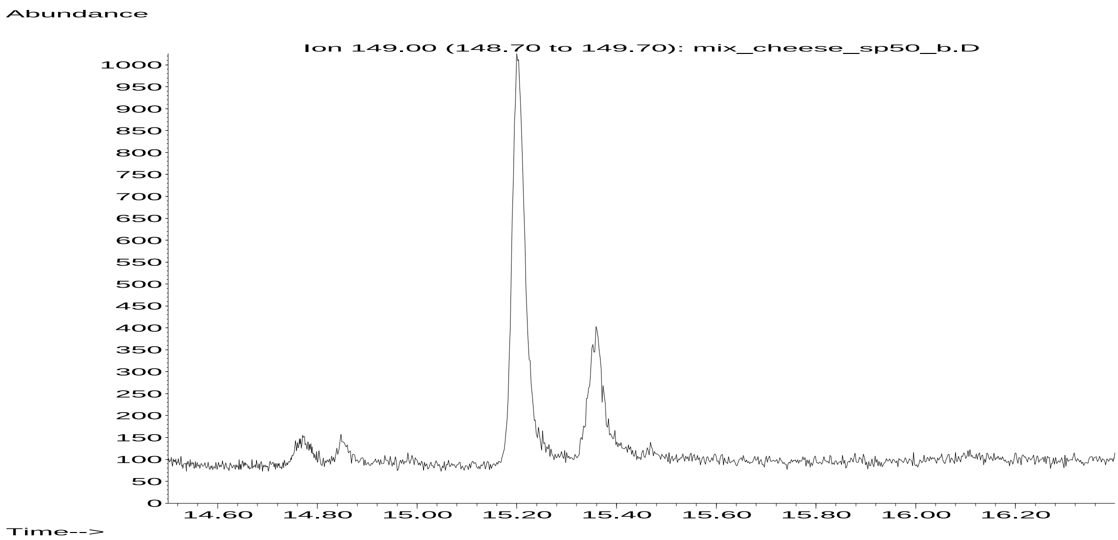


Figure 21. Diallyl phthalate spiked into foods at the target LOQ

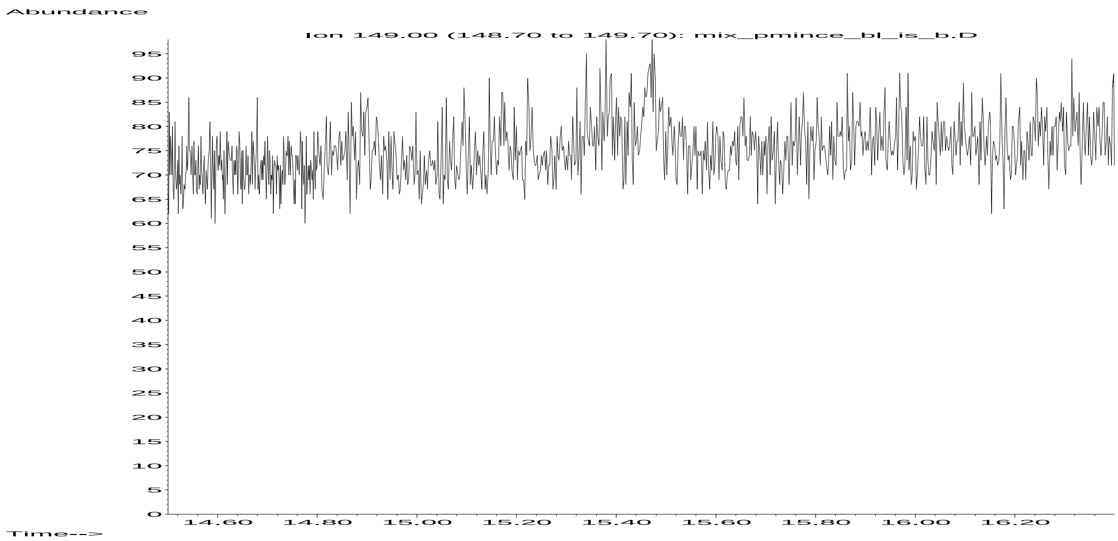
(a) Cheese only



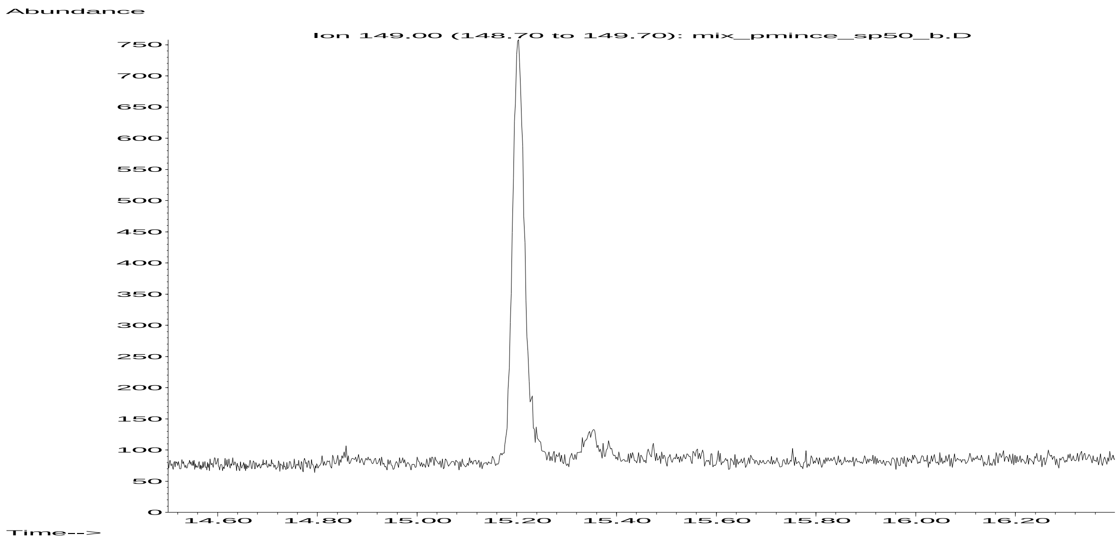
(b) Cheese overspiked with diallyl phthalate at 0.05 mg/kg



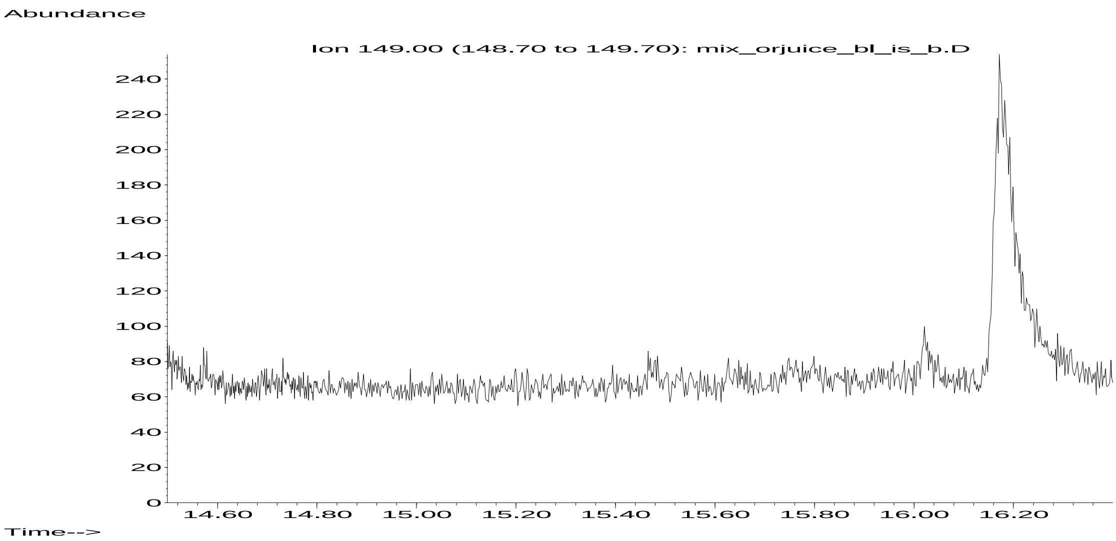
(c) Pork mince only



(d) Pork mince overspiked with diallyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with diallyl phthalate at 0.05 mg/kg

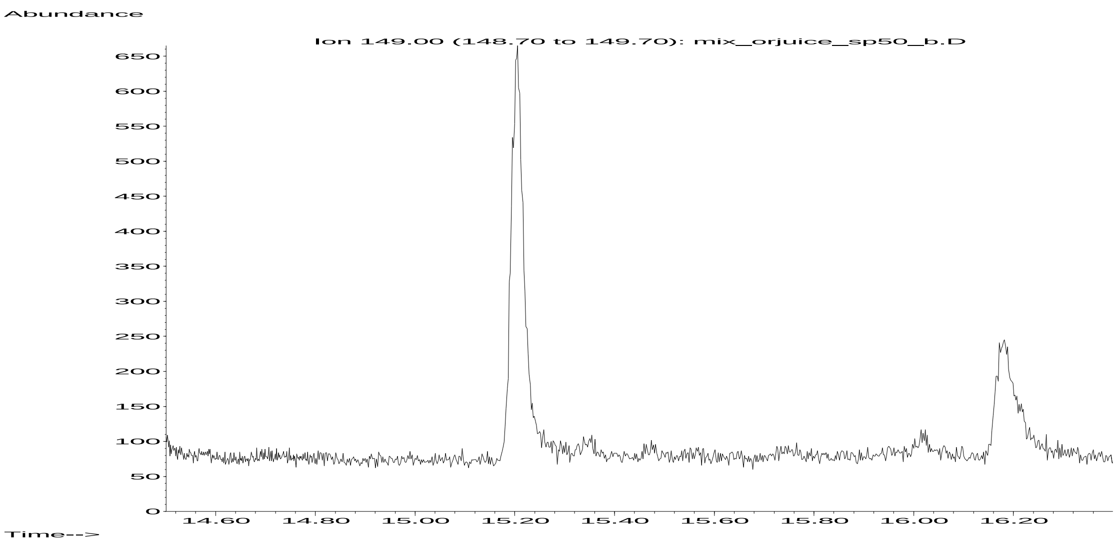
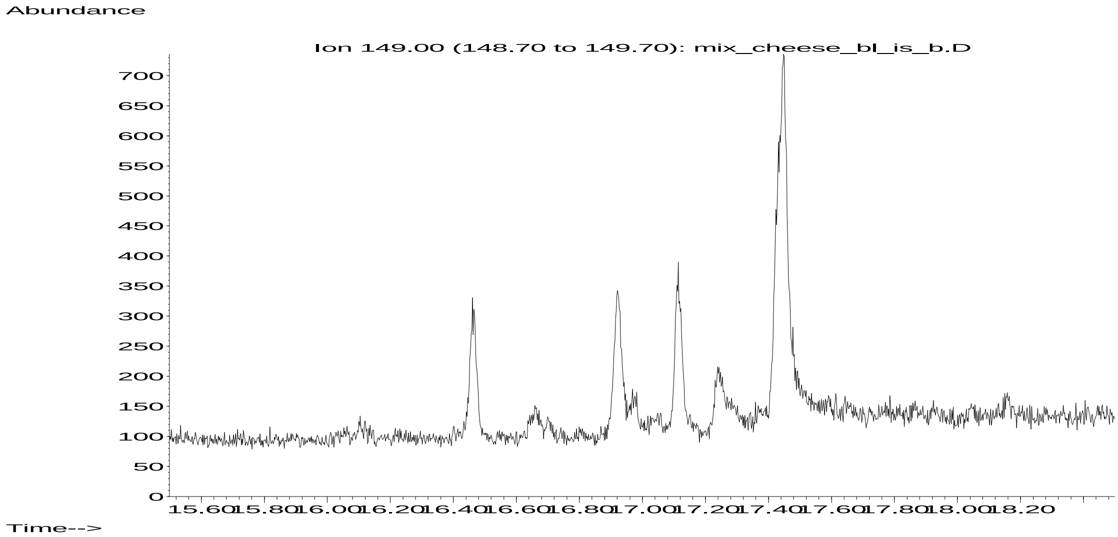
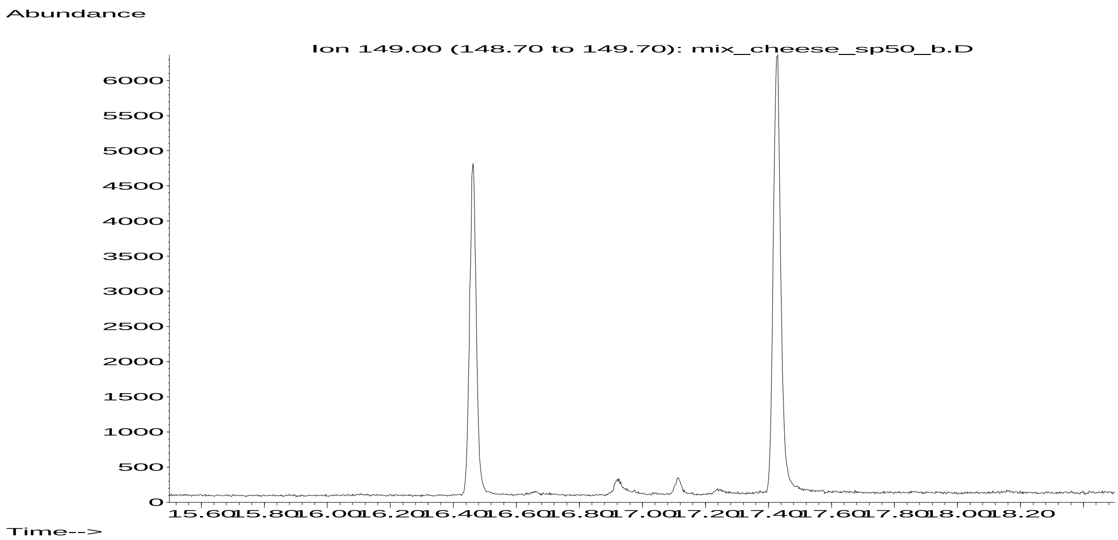


Figure 22. Dibutyl and diisobutyl phthalate spiked into foods at the target LOQ

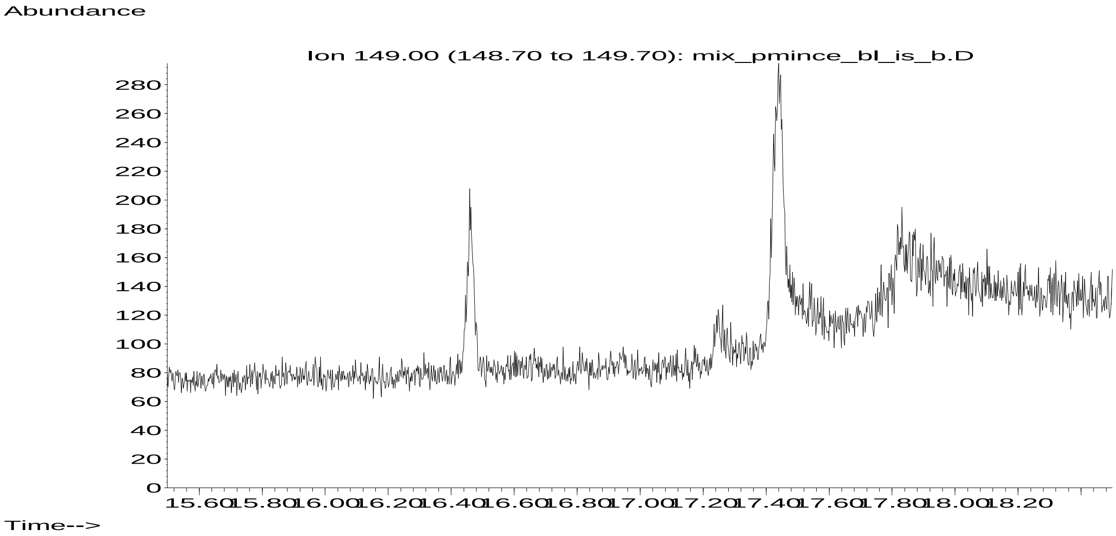
(a) Cheese only



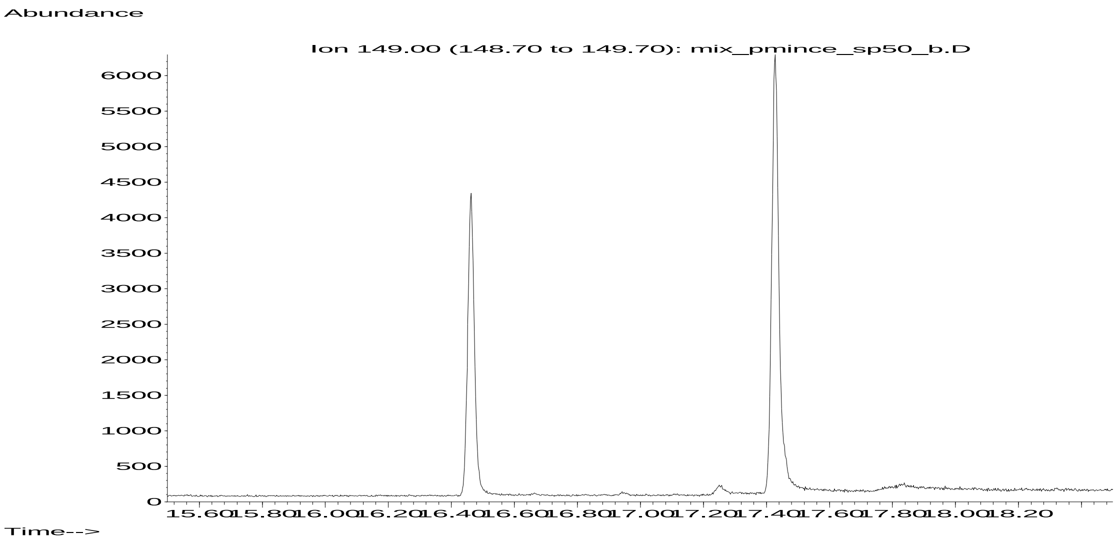
(b) Cheese overspiked with dibutyl and diisobutyl phthalate at 0.05 mg/kg



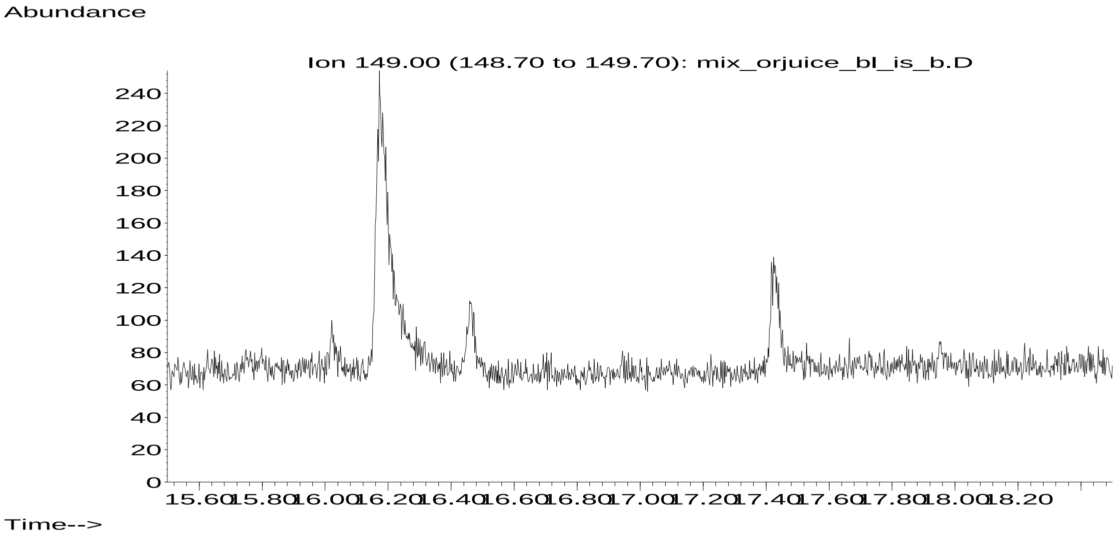
(c) Pork mince only



(d) Pork mince overspiked with dibutyl and diisobutyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with dibutyl and diisobutyl phthalate at 0.05 mg/kg

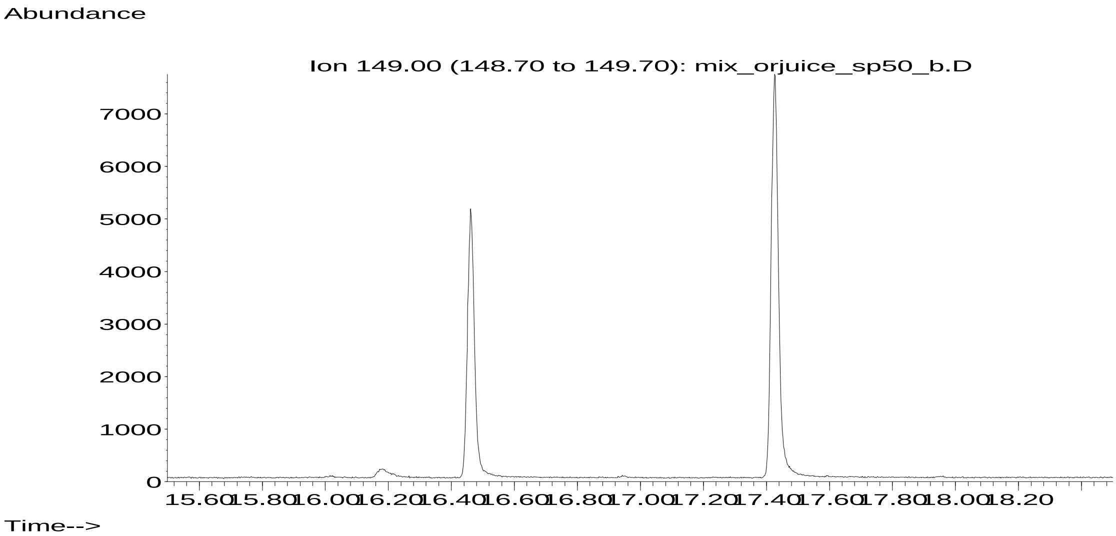
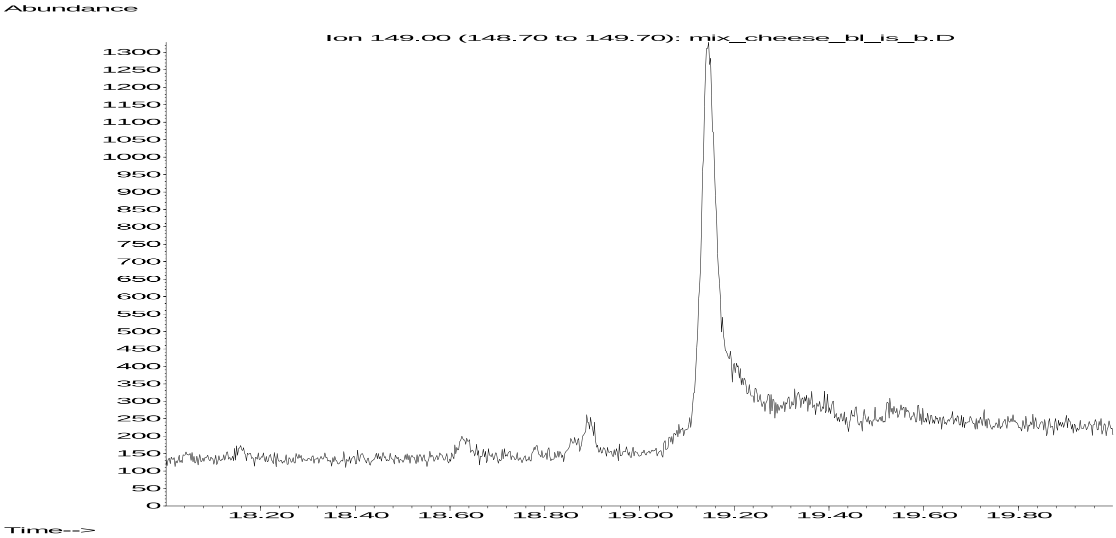
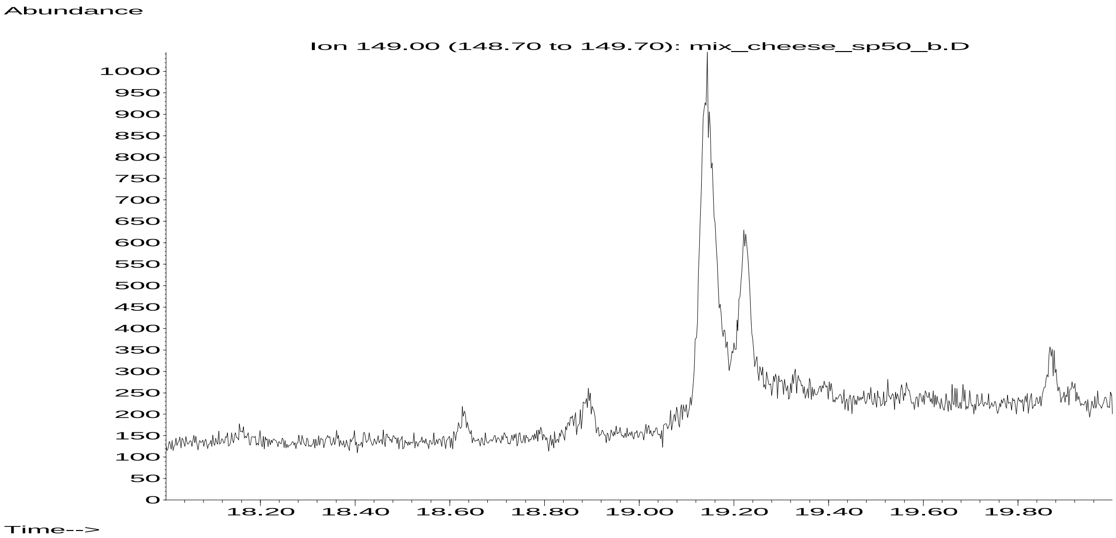


Figure 23. Dipentyl phthalate spiked into foods at the target LOQ

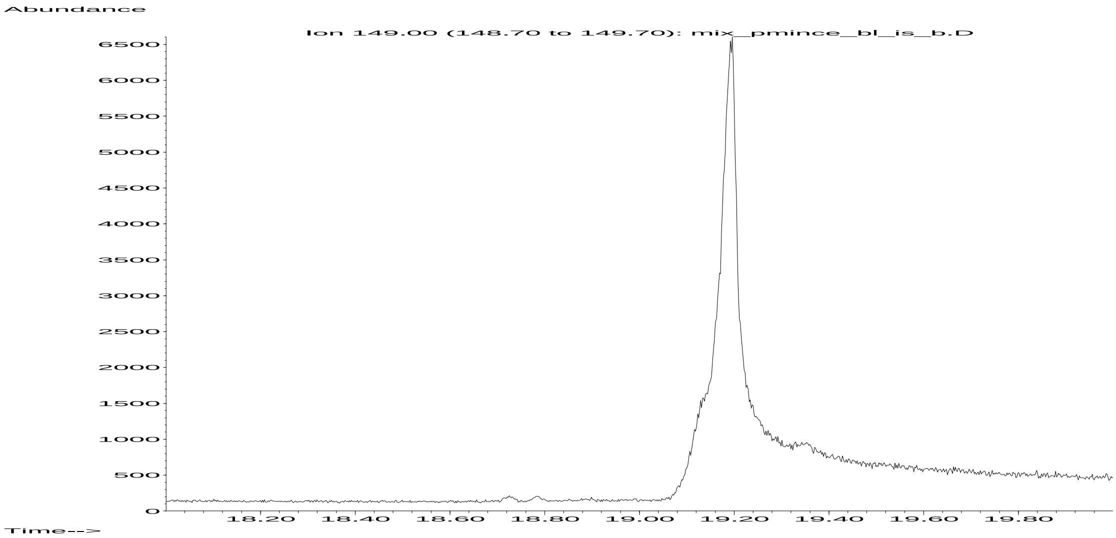
(a) Cheese only



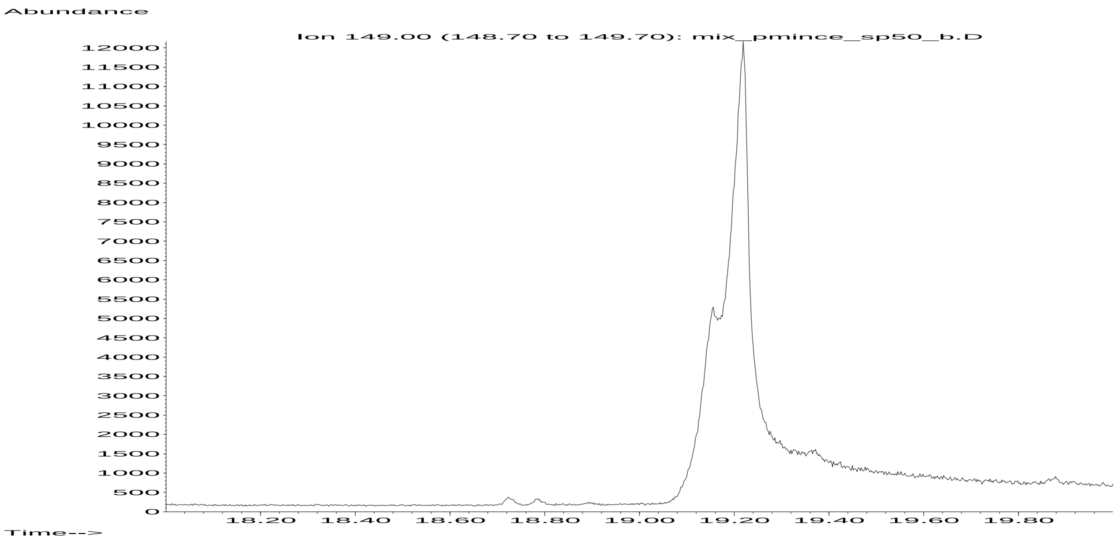
(b) Cheese overspiked with dipentyl phthalate at 0.05 mg/kg



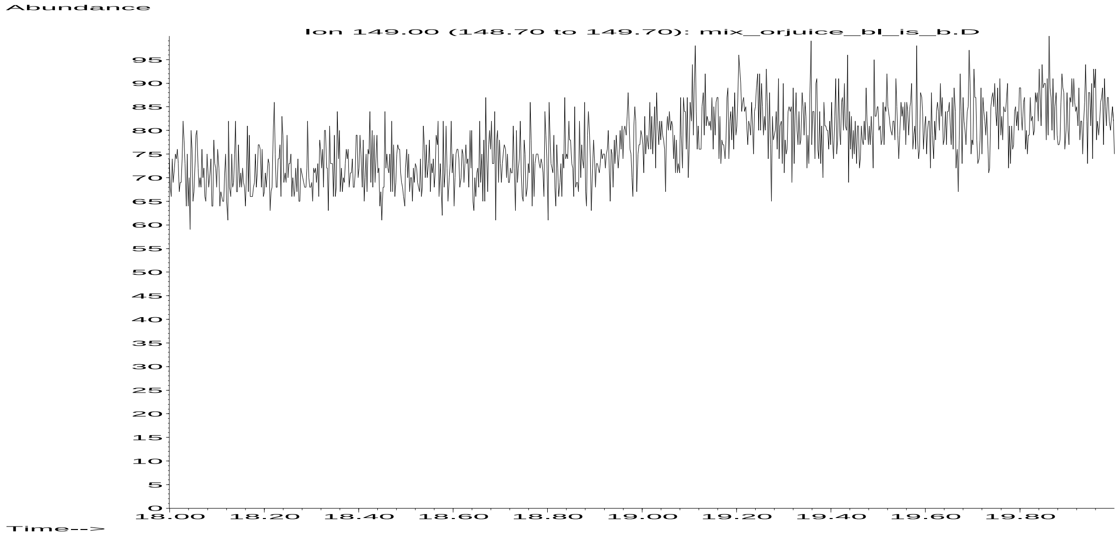
(c) Pork mince only



(d) Pork mince overspiked with dipentyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with dipentyl phthalate at 0.05 mg/kg

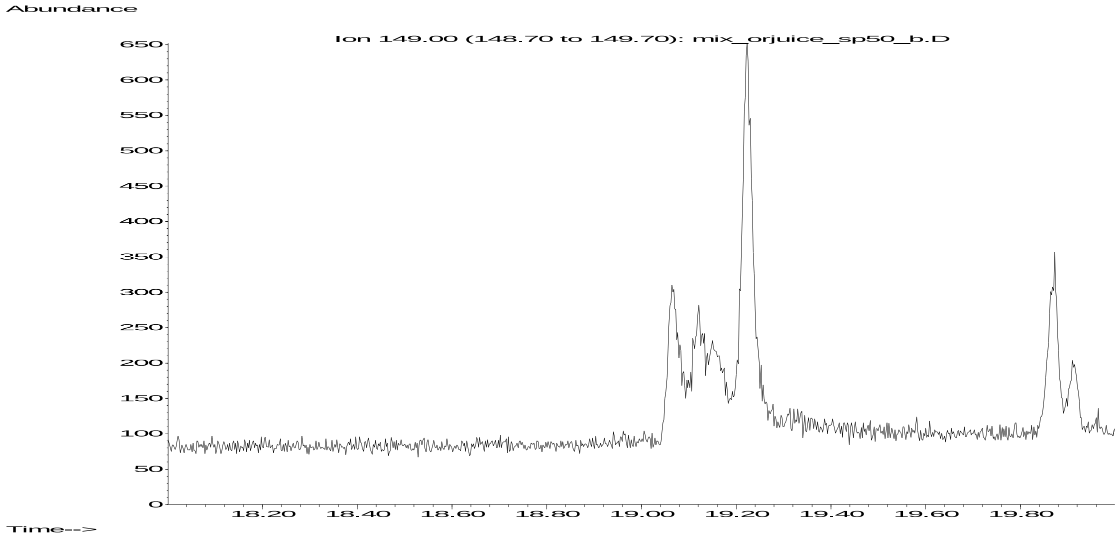
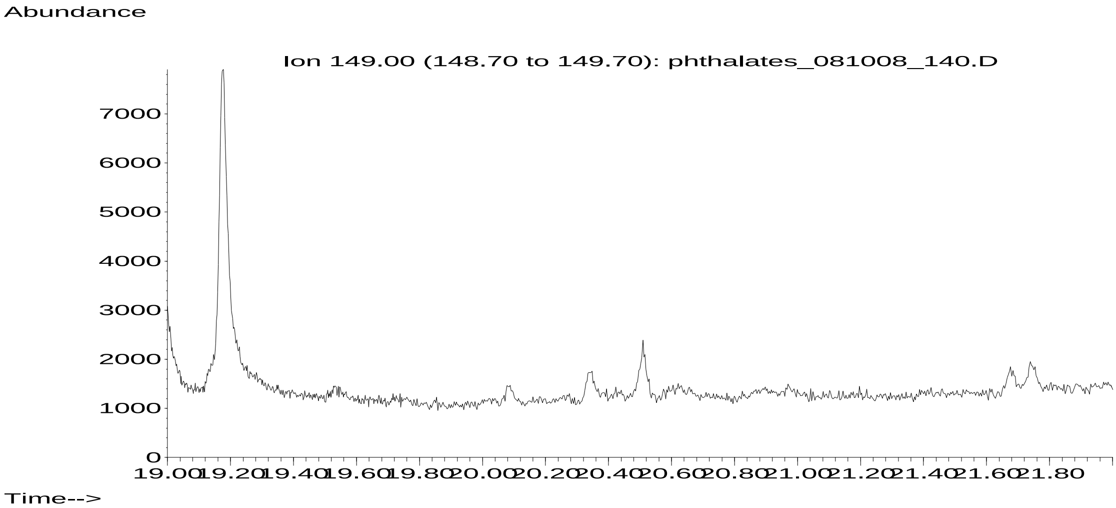
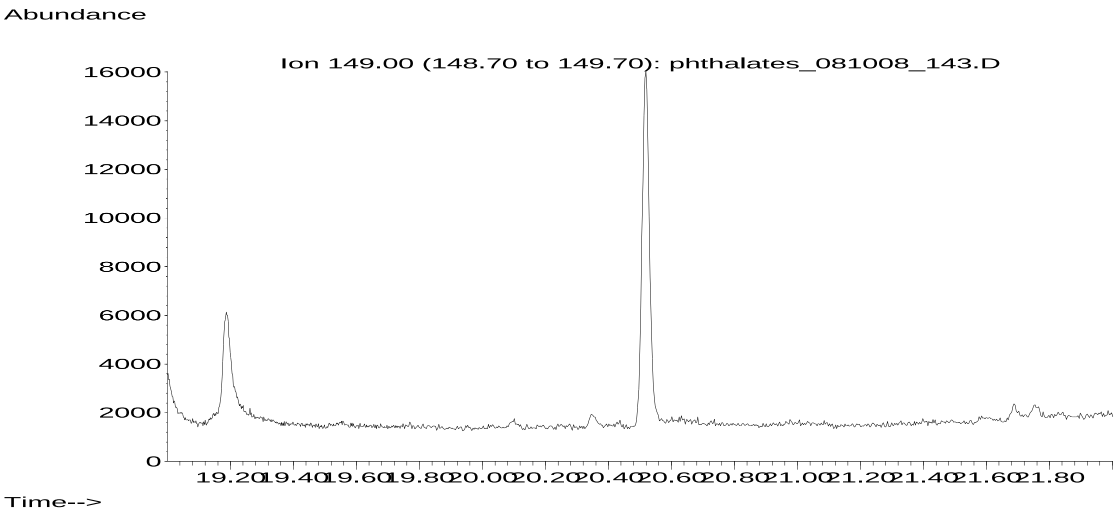


Figure 24. Di-n-hexyl phthalate spiked into foods at the target LOQ

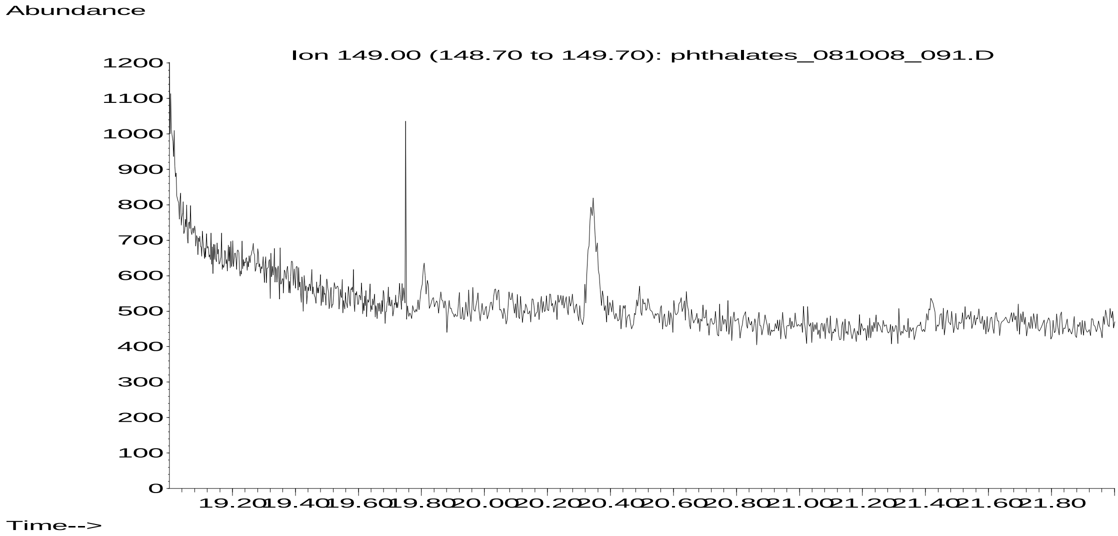
(a) Cheese only



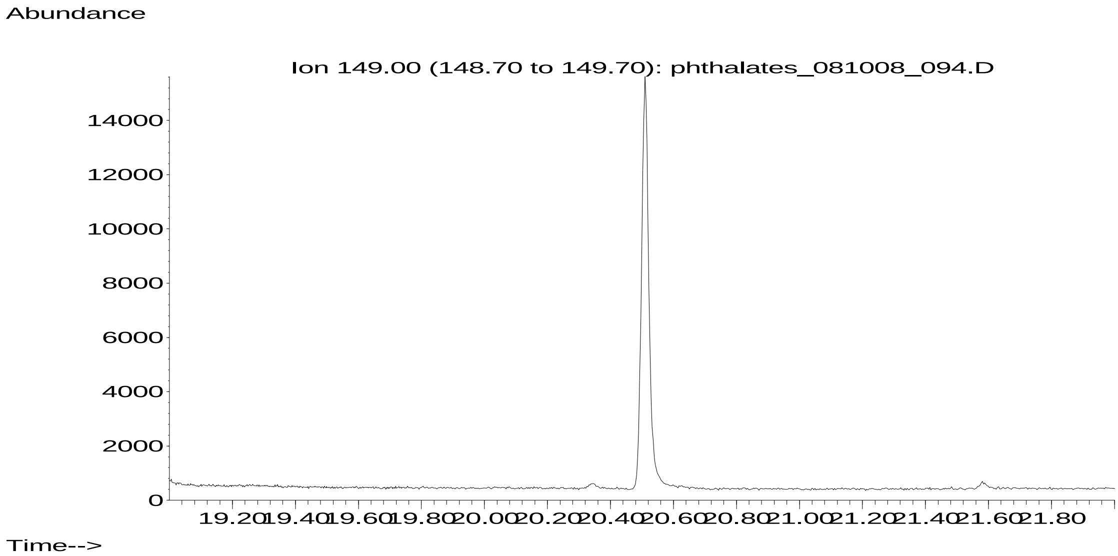
(b) Cheese overspiked with di-n-hexyl phthalate at 0.05 mg/kg



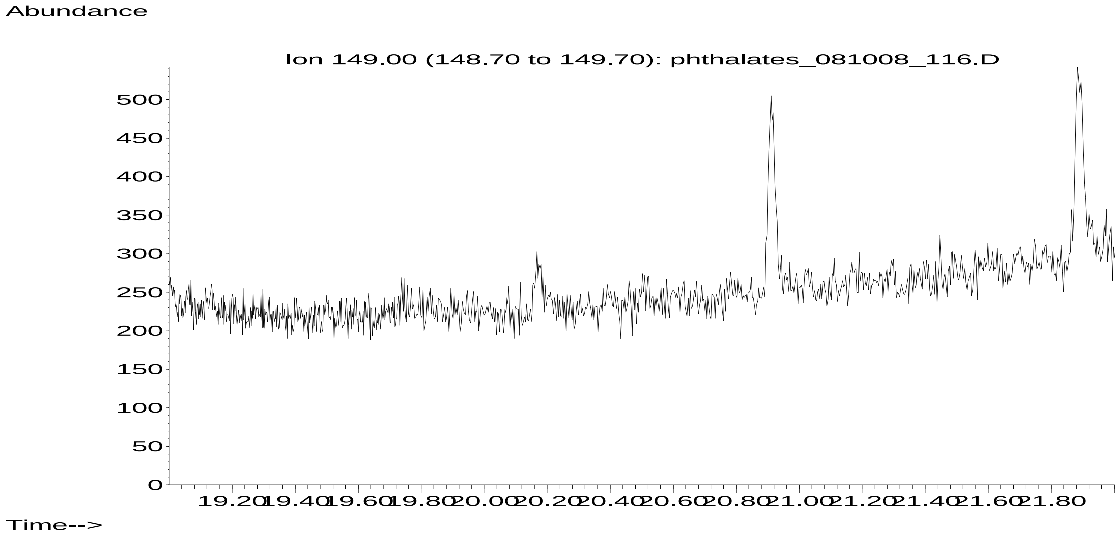
(c) Pork mince only



(d) Pork mince overspiked with di-n-hexyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with di-n-hexyl phthalate at 0.05 mg/kg

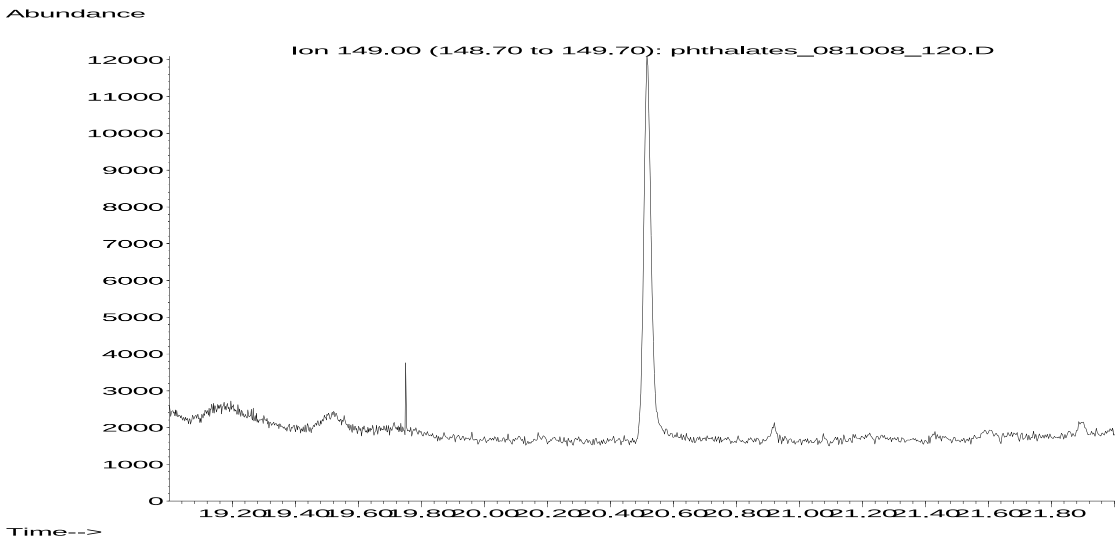
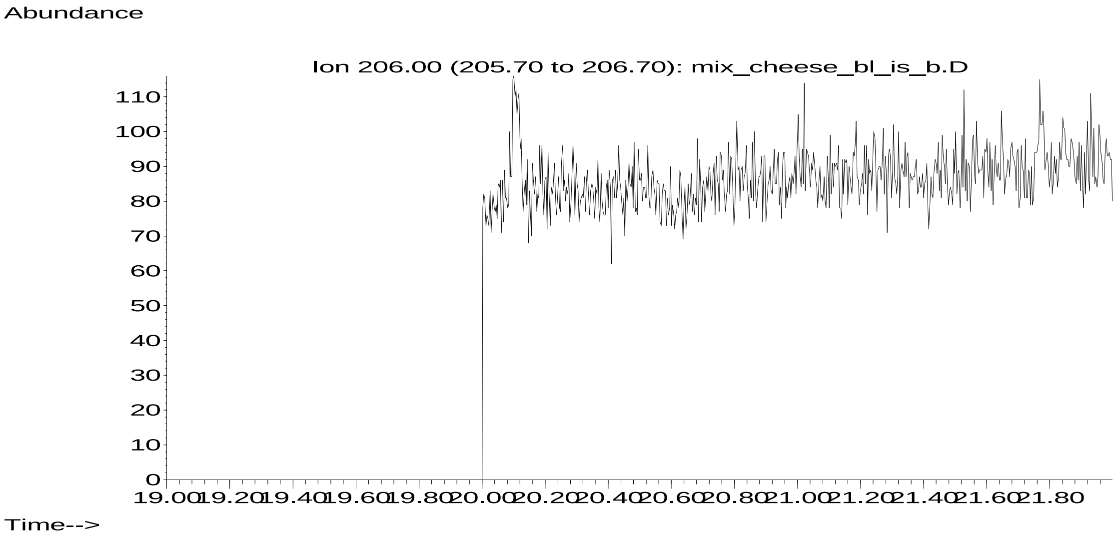
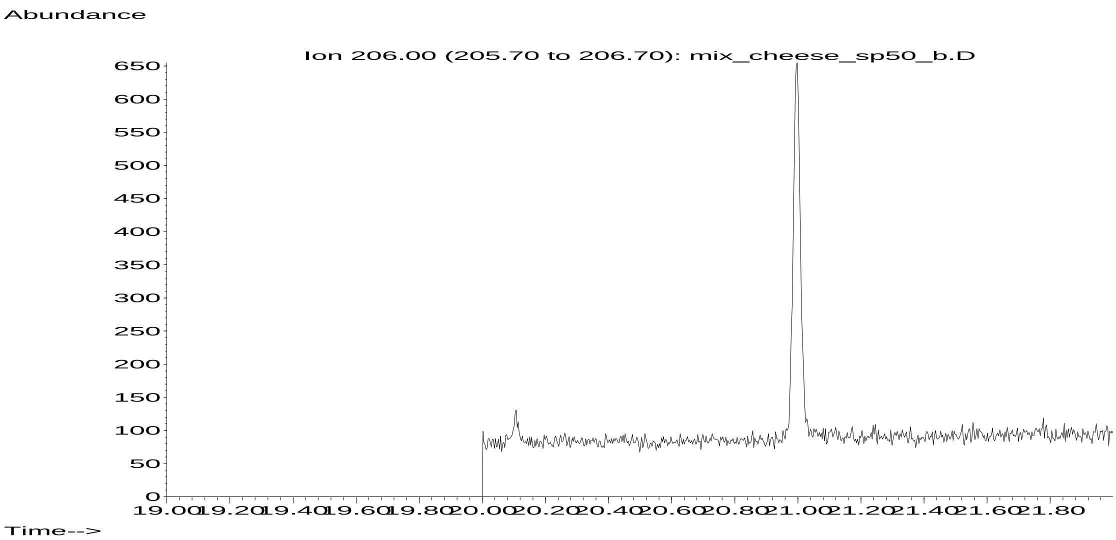


Figure 25. Benzyl butyl phthalate spiked into foods at the target LOQ

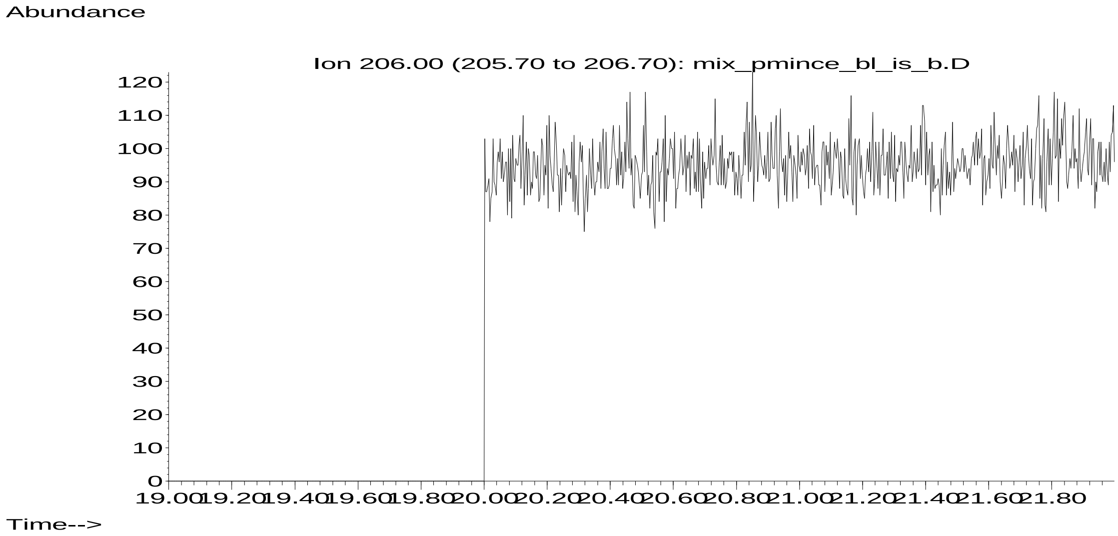
(a) Cheese only



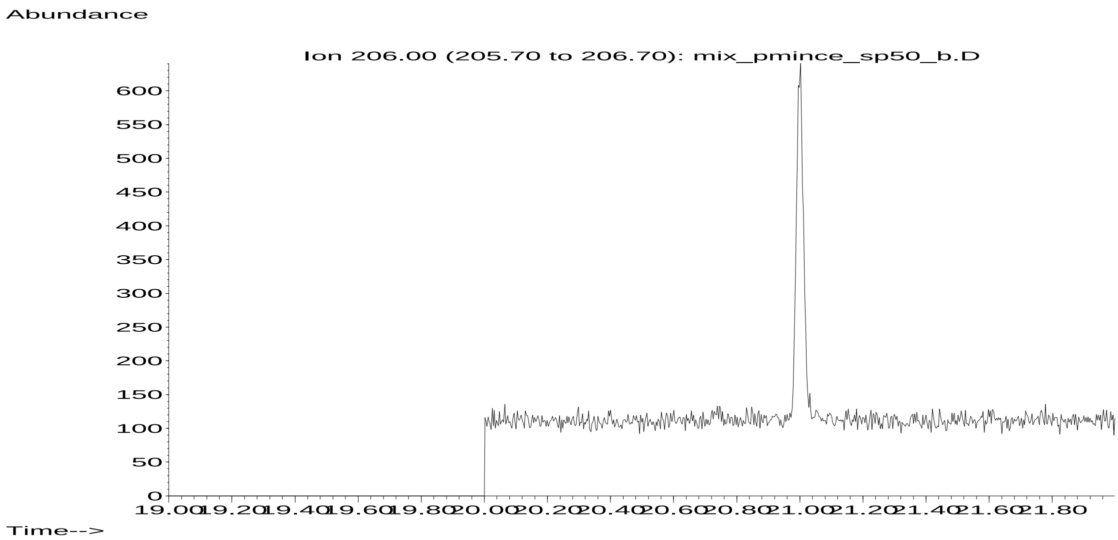
(b) Cheese overspiked with benzyl butyl phthalate at 0.05 mg/kg



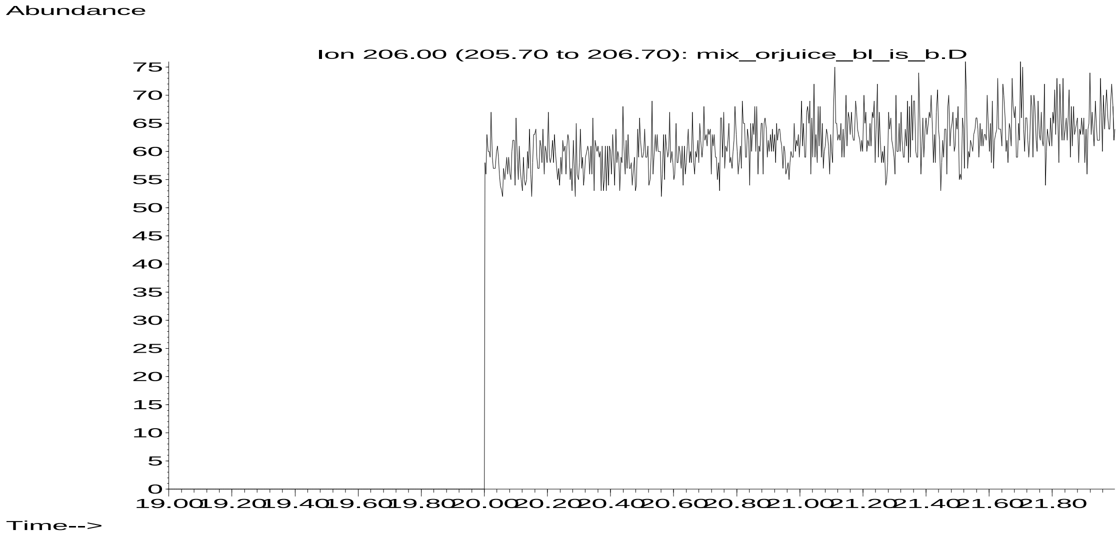
(c) Pork mince only



(d) Pork mince overspiked with benzyl butyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with benzyl butyl phthalate at 0.05 mg/kg

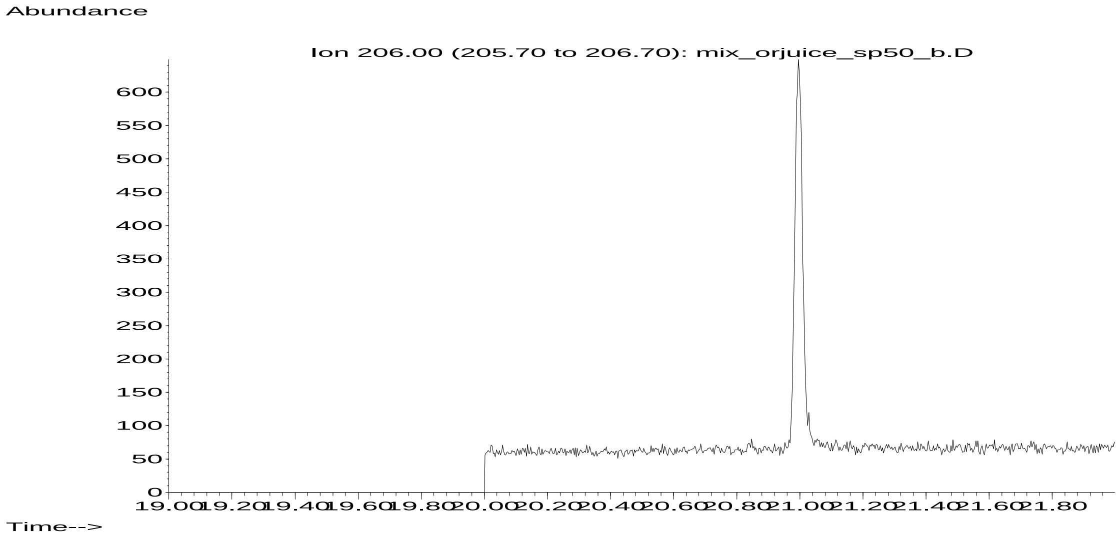
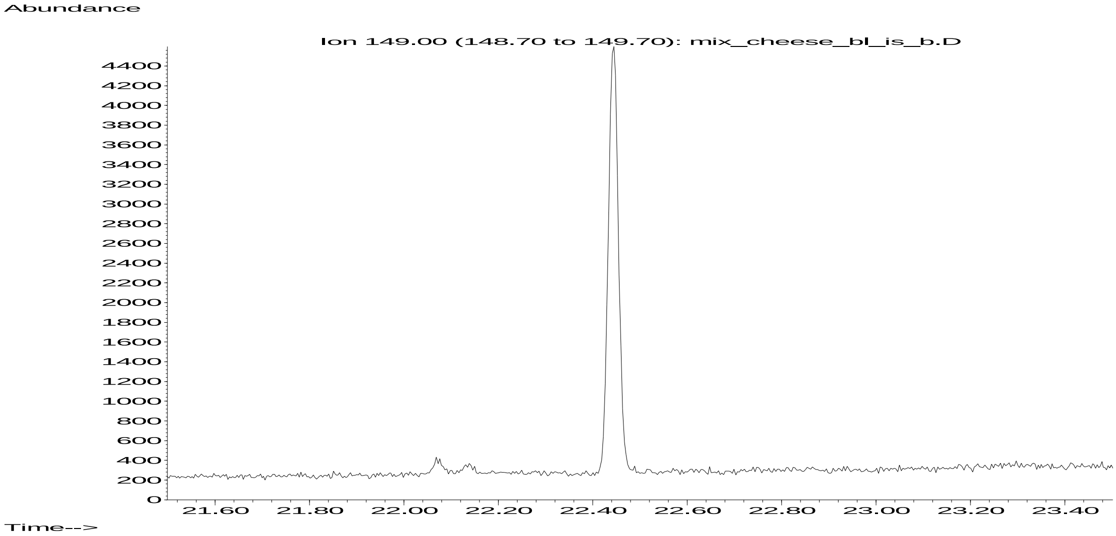
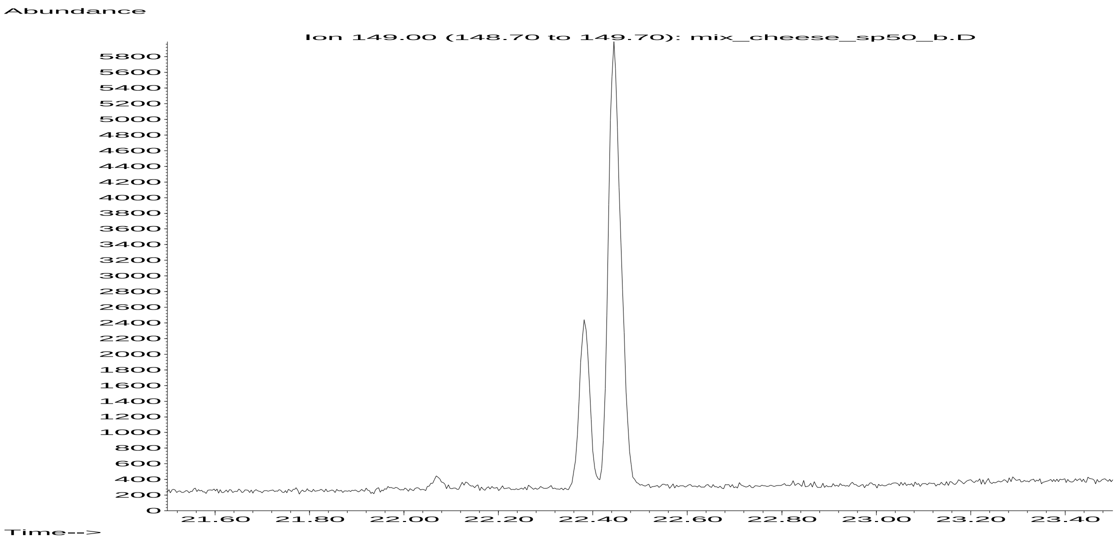


Figure 26. Dicyclohexyl phthalate spiked into foods at the target LOQ

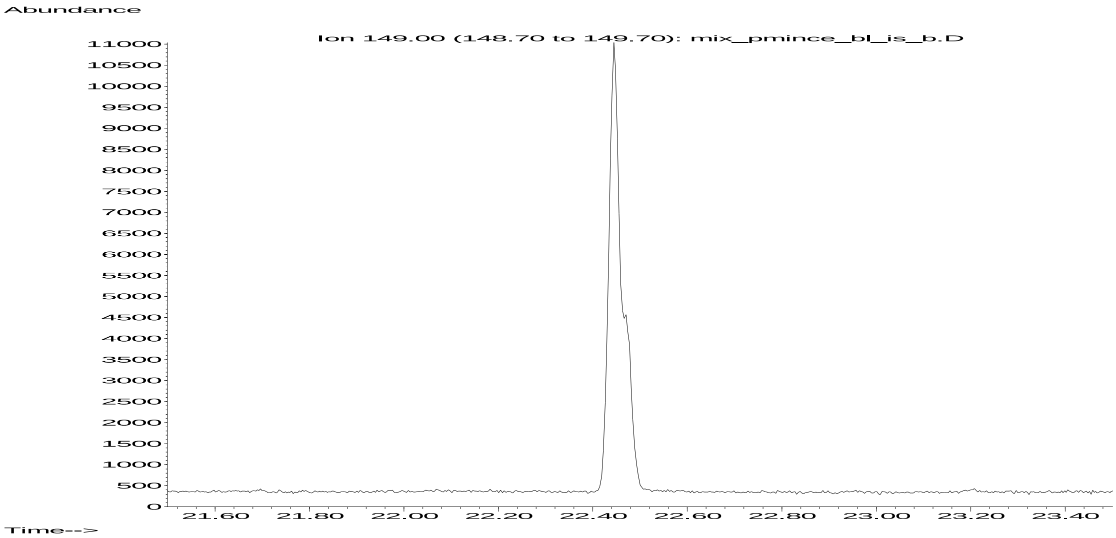
(a) Cheese only



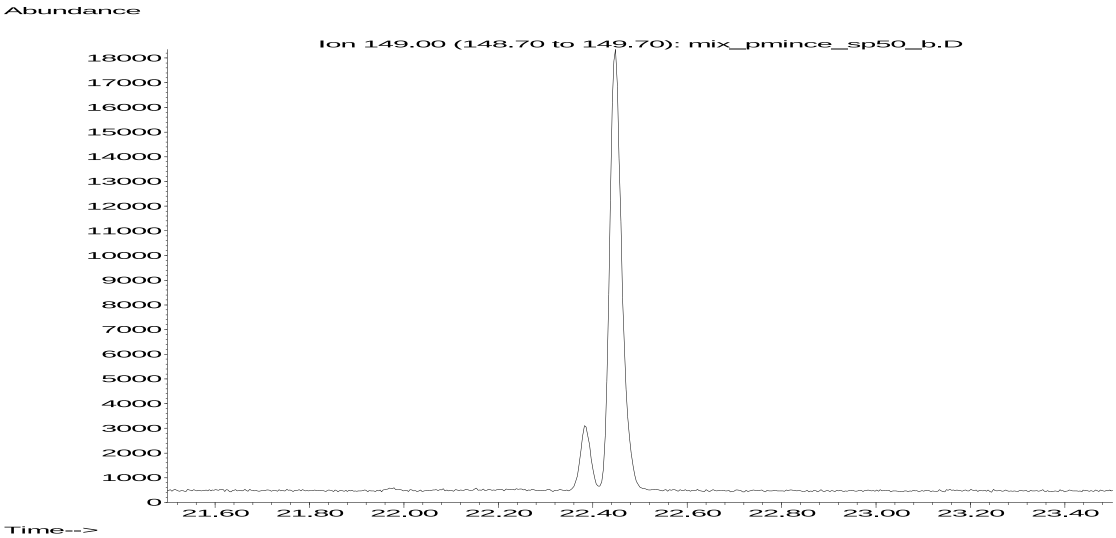
(b) Cheese overspiked with dicyclohexyl phthalate at 0.05 mg/kg



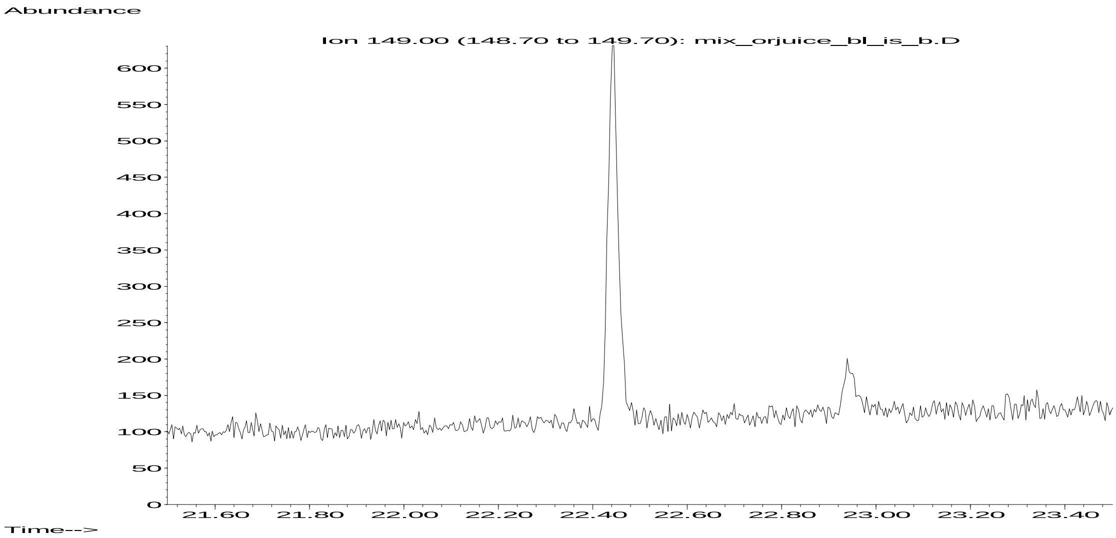
(c) Pork mince only



(d) Pork mince overspiked with dicyclohexyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with dicyclohexyl phthalate at 0.05 mg/kg

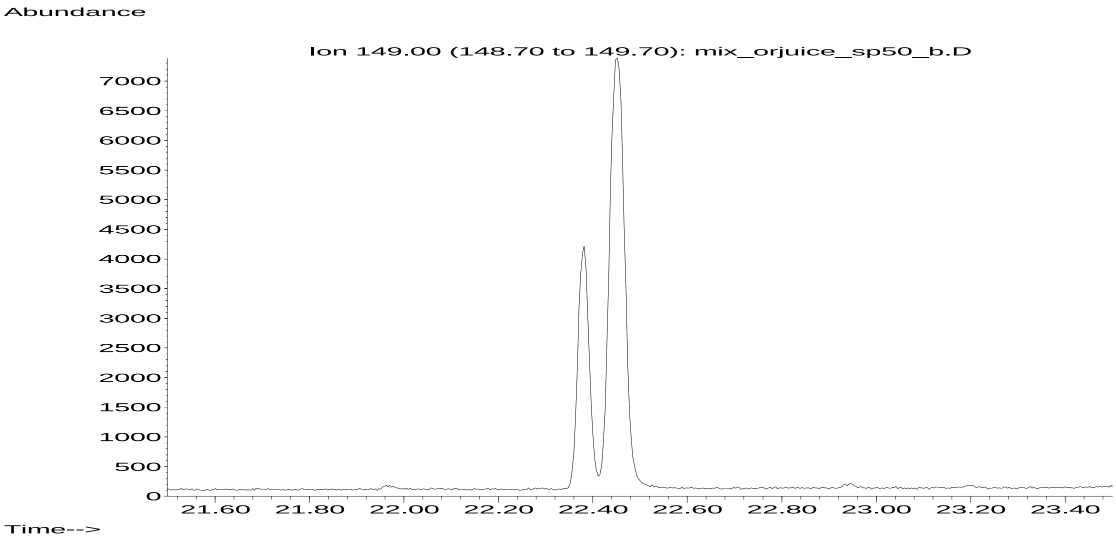
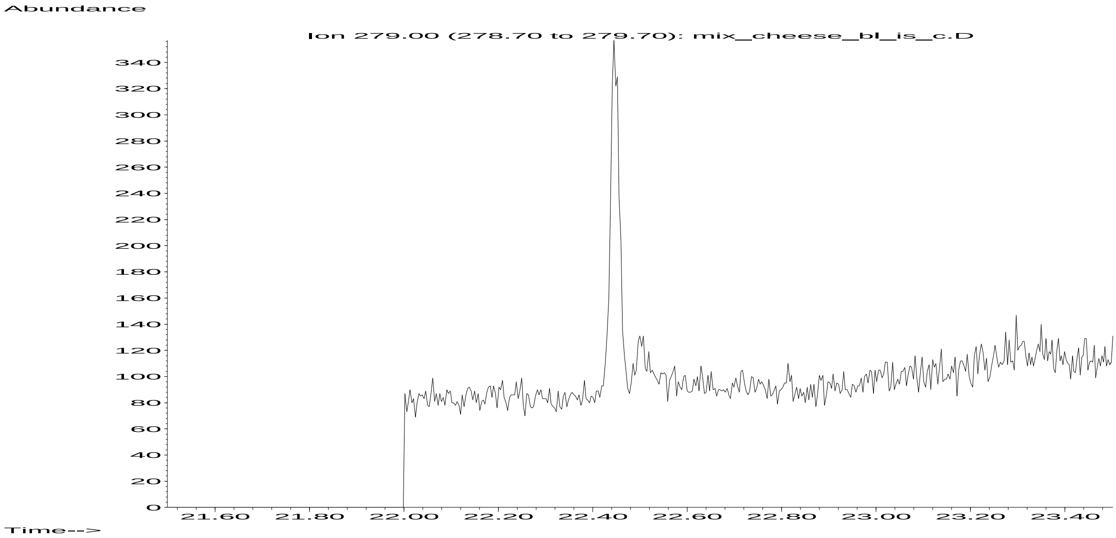
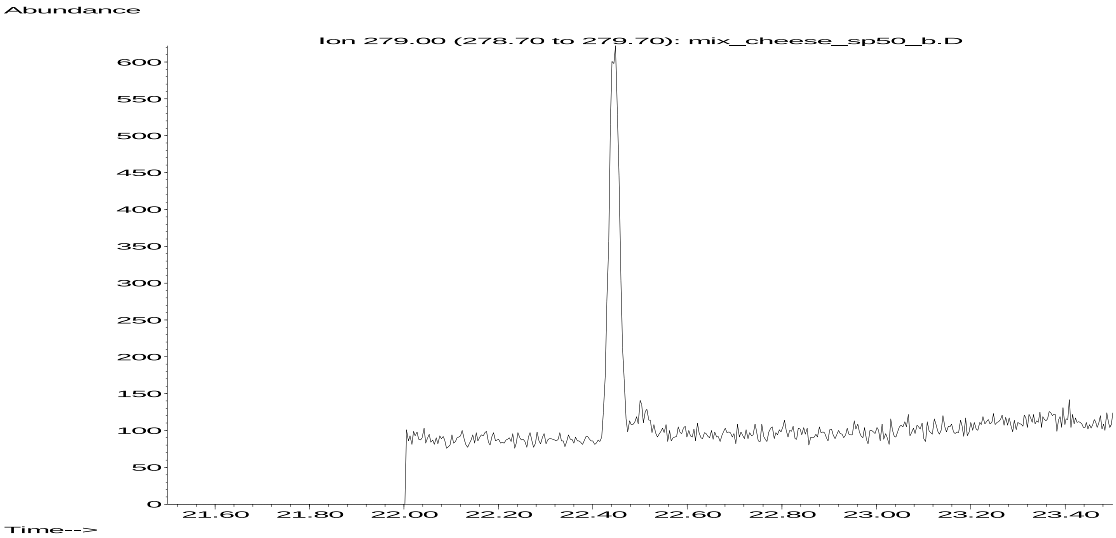


Figure 27. Di-(2-ethylhexyl) phthalate spiked into foods at the target LOQ

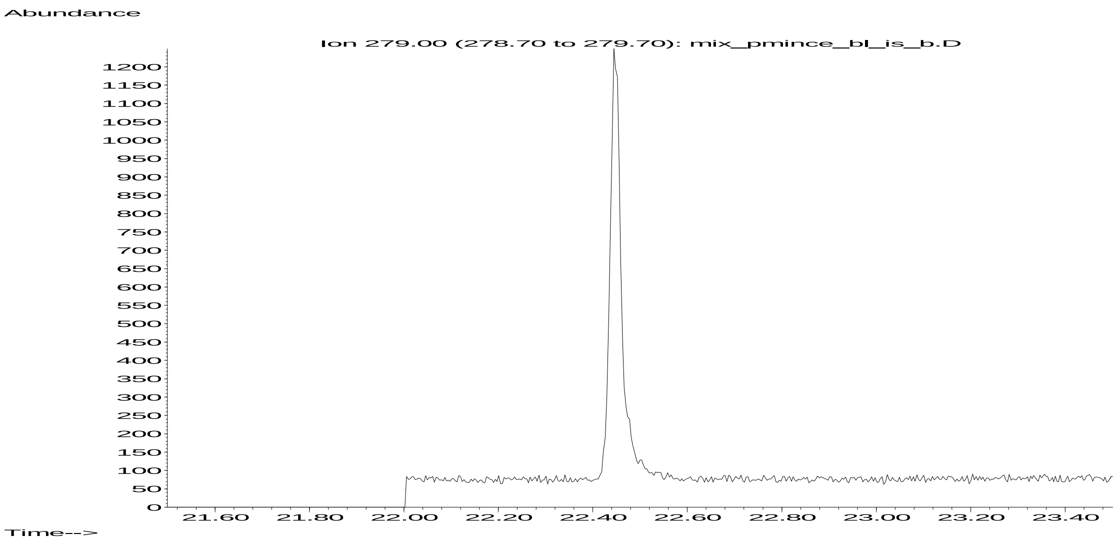
(a) Cheese only



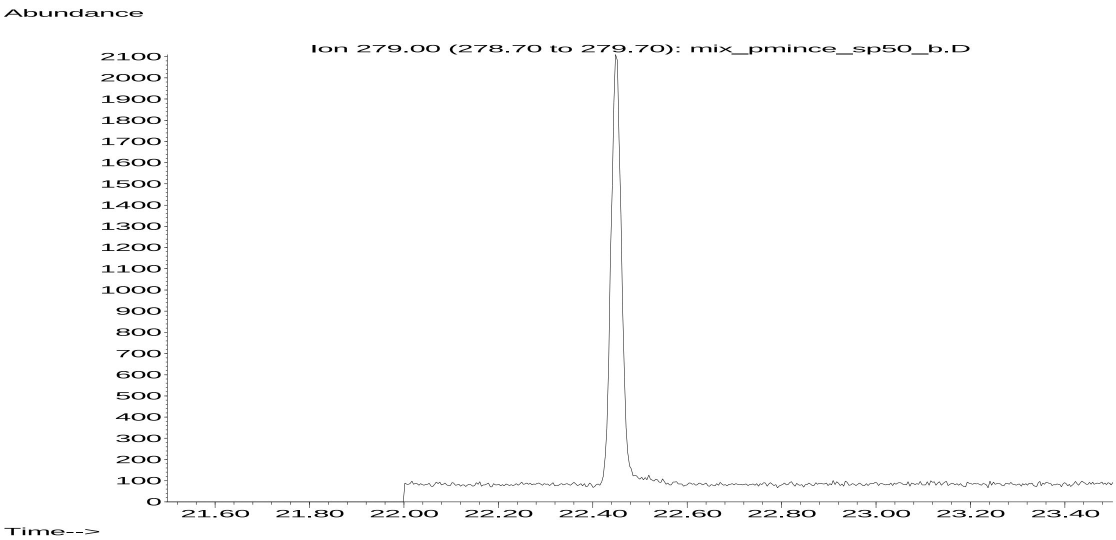
(b) Cheese overspiked with di-(2-ethylhexyl) phthalate at 0.05 mg/kg



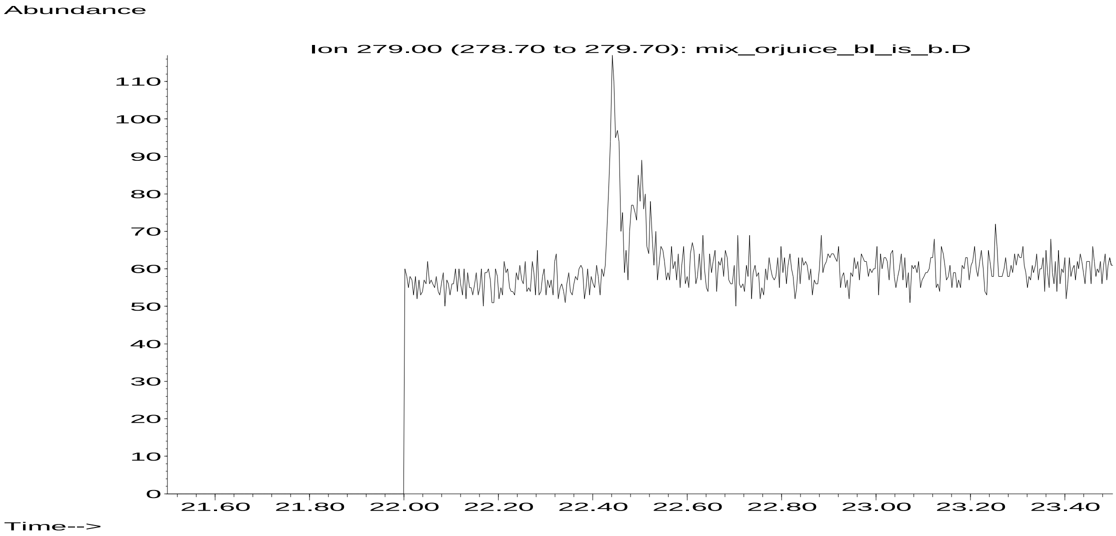
(c) Pork mince only



(d) Pork mince overspiked with di-(2-ethylhexyl) phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with di-(2-ethylhexyl) phthalate at 0.05 mg/kg

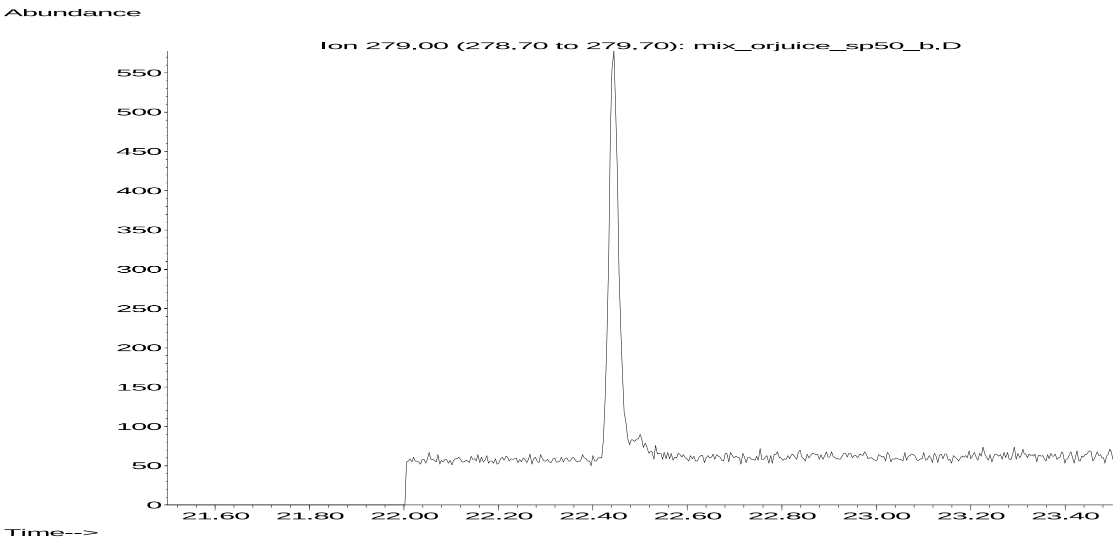
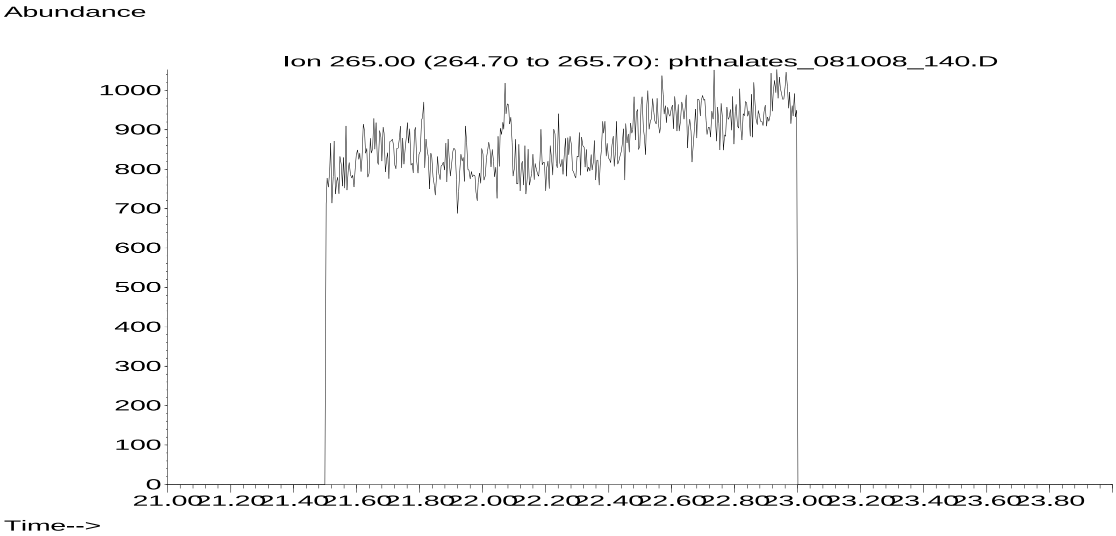
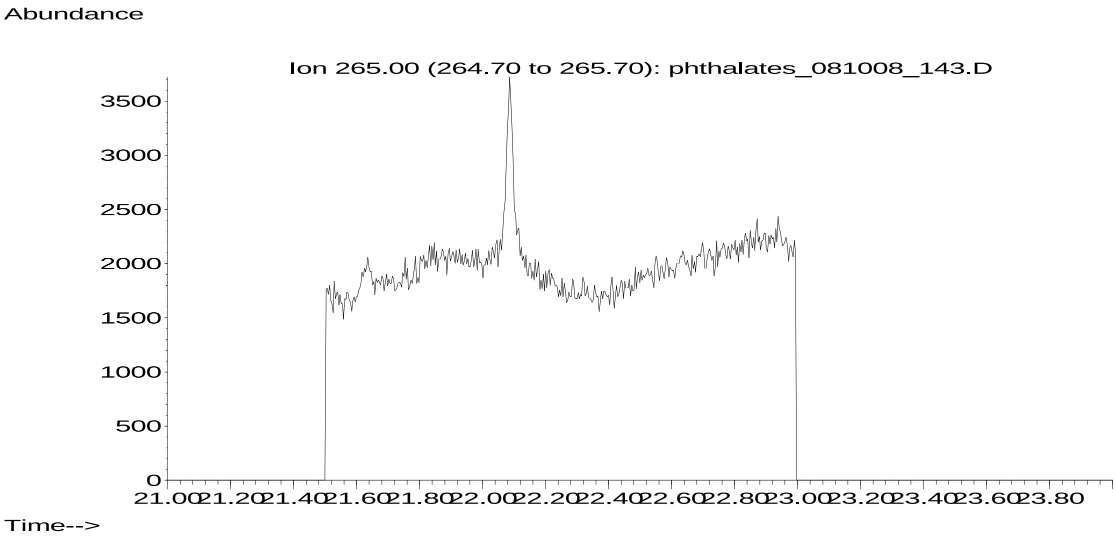


Figure 28. Di-n-heptyl phthalate spiked into foods at the target LOQ

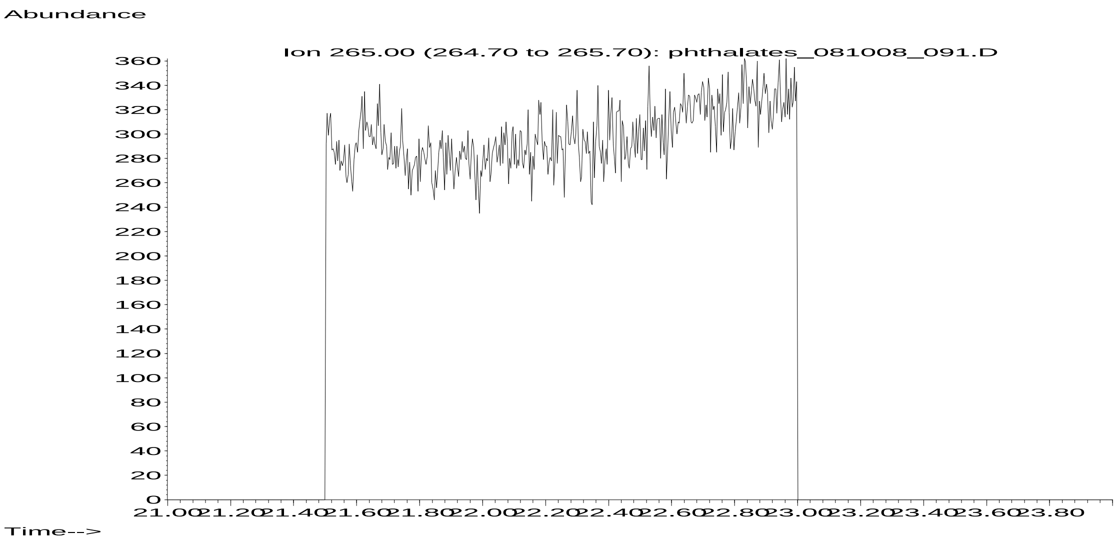
(a) Cheese only



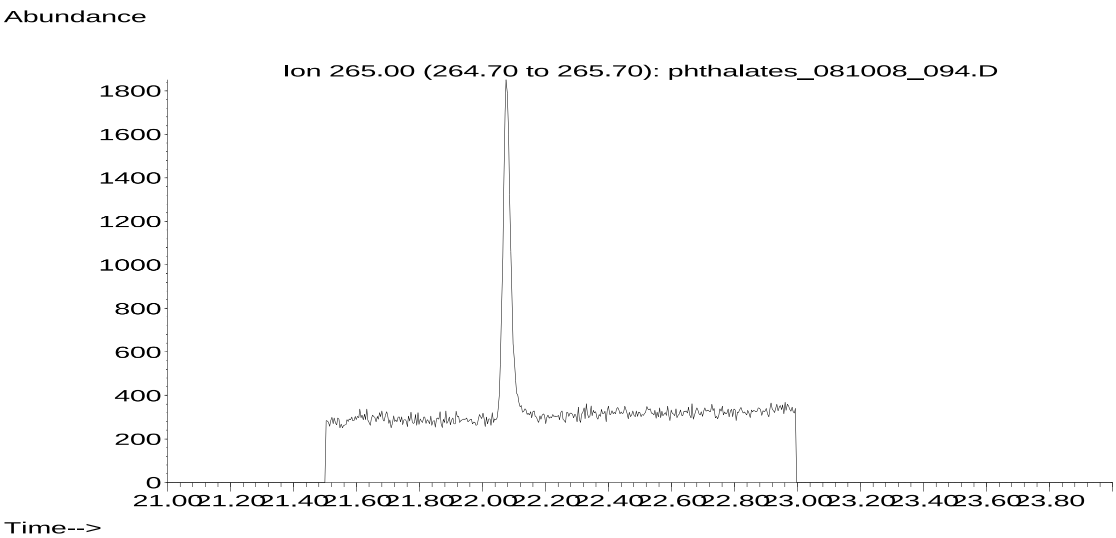
(b) Cheese overspiked with di-n-heptyl phthalate at 0.05 mg/kg



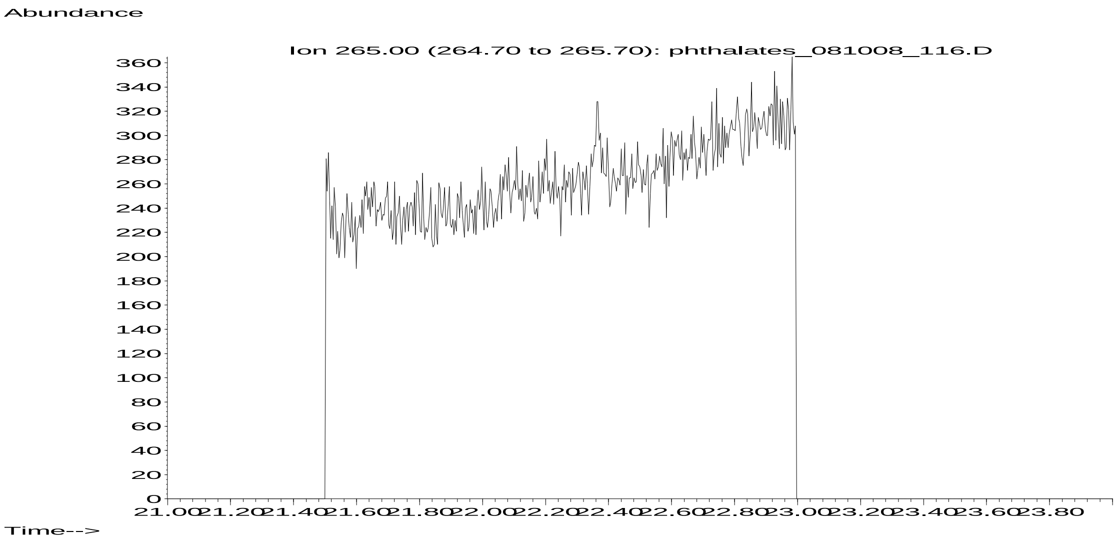
(c) Pork mince only



(d) Pork mince overspiked with di-n-heptyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with di-n-heptyl phthalate at 0.05 mg/kg

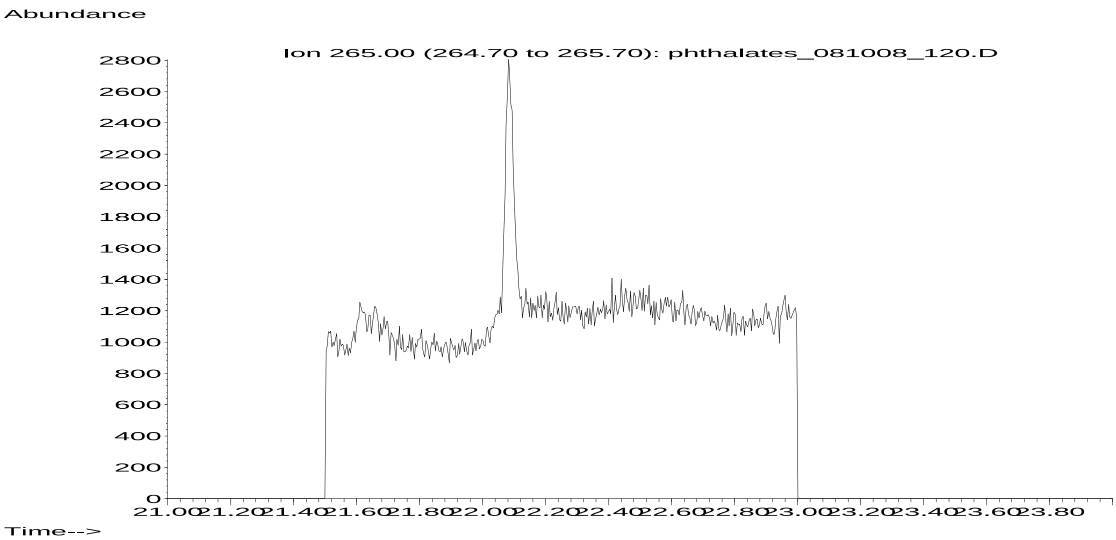
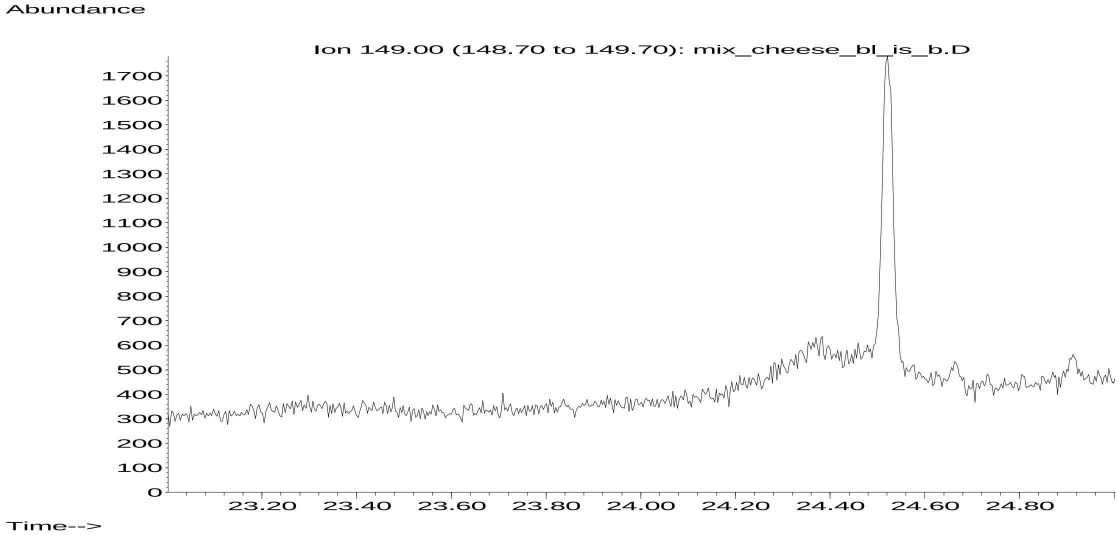
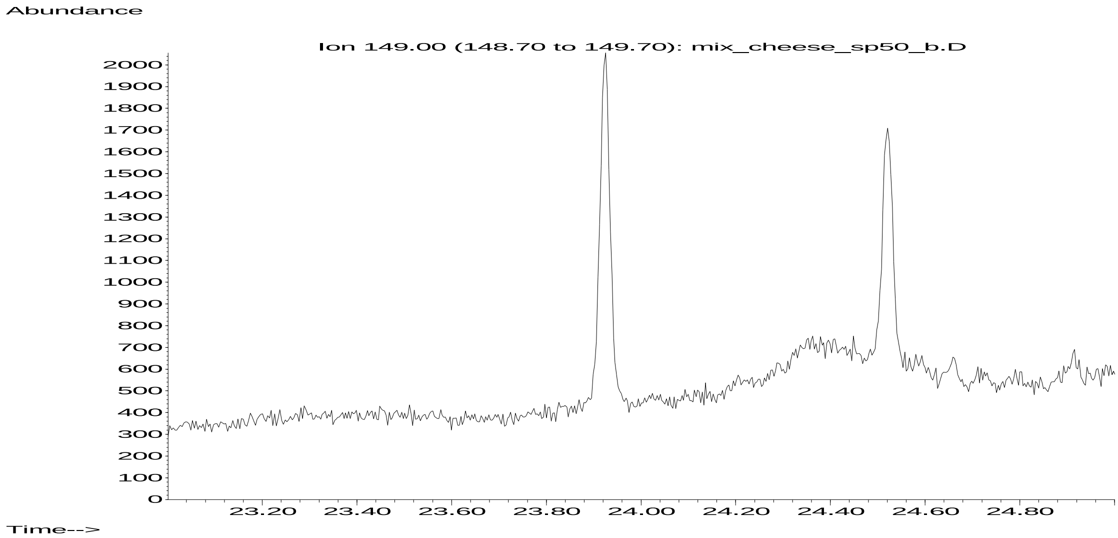


Figure 29. Di-n-octyl phthalate spiked into foods at the target LOQ

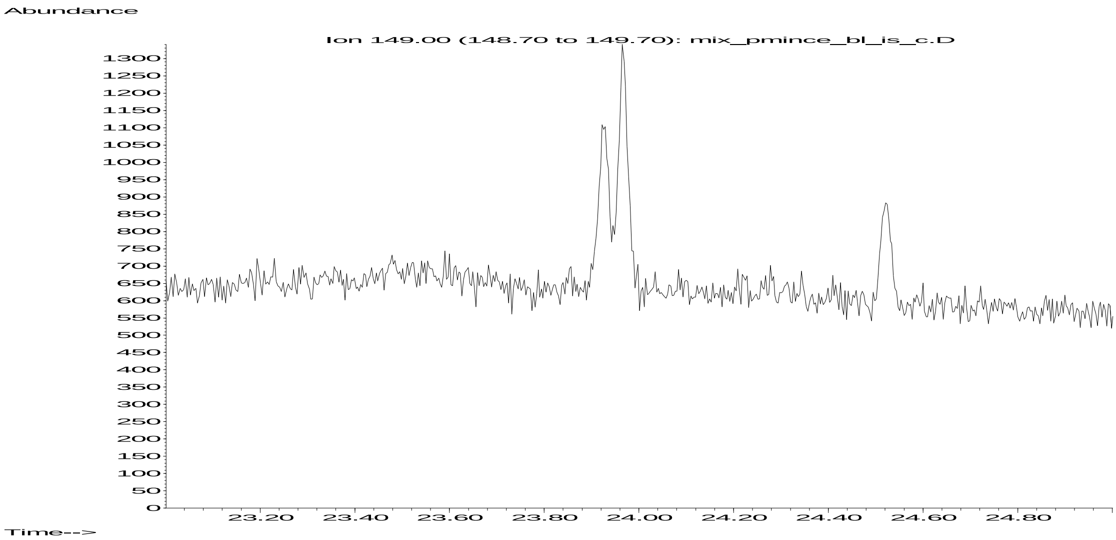
(a) Cheese only



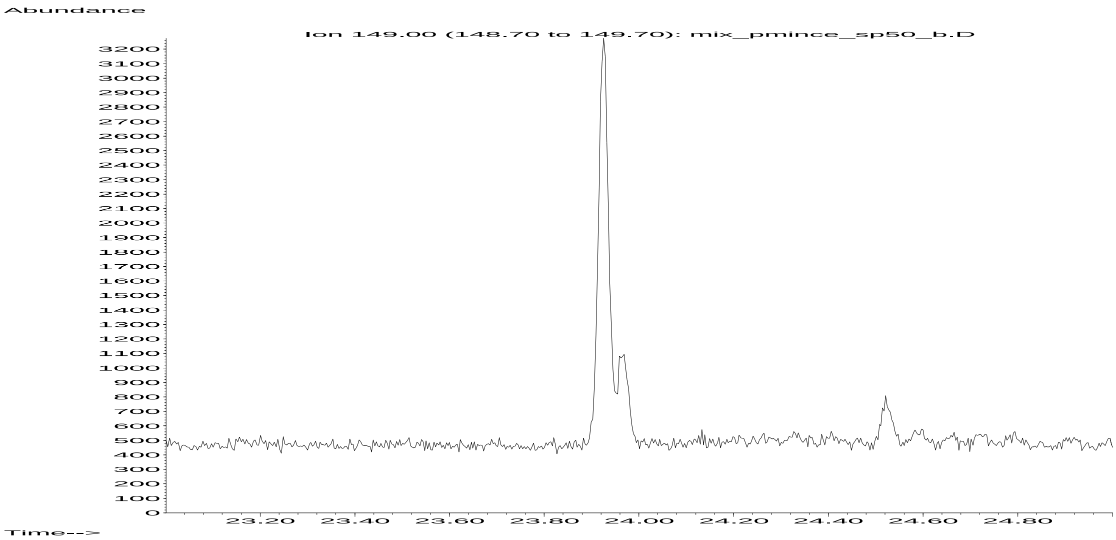
(b) Cheese overspiked with di-n-octyl phthalate at 0.05 mg/kg



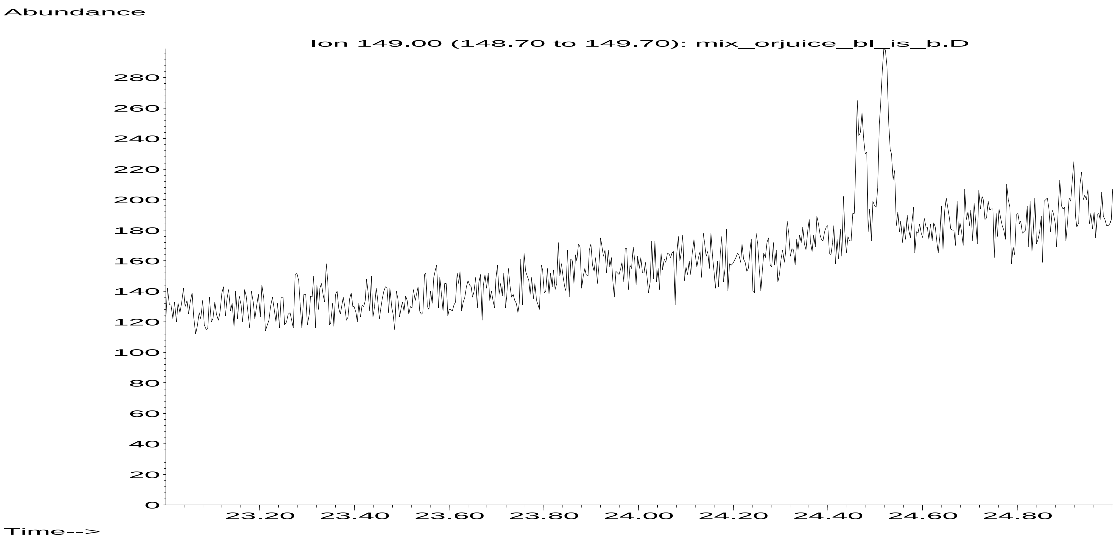
(c) Pork mince only



(d) Pork mince overspiked with di-n-octyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with di-n-octyl phthalate at 0.05 mg/kg

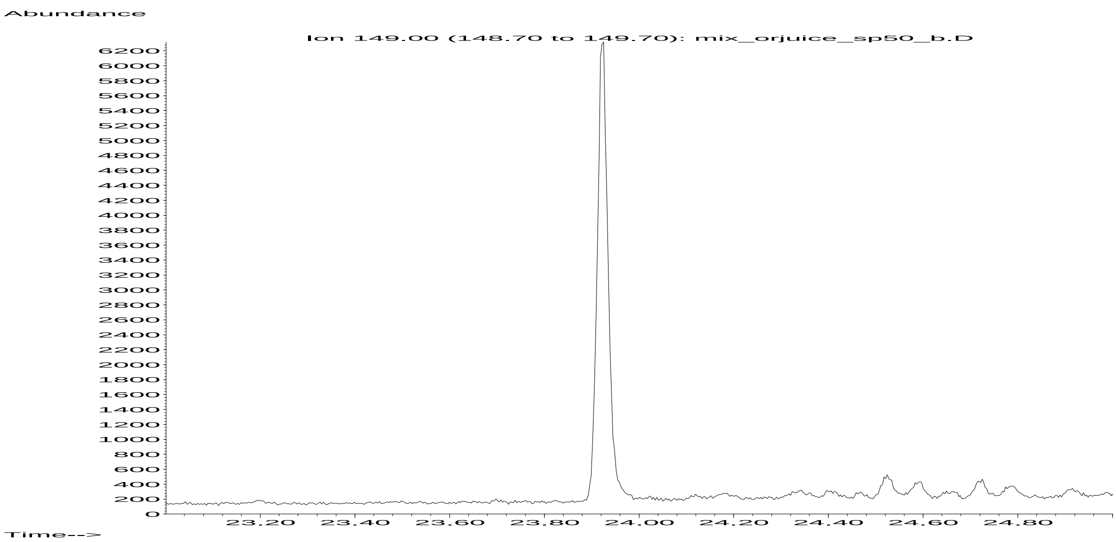
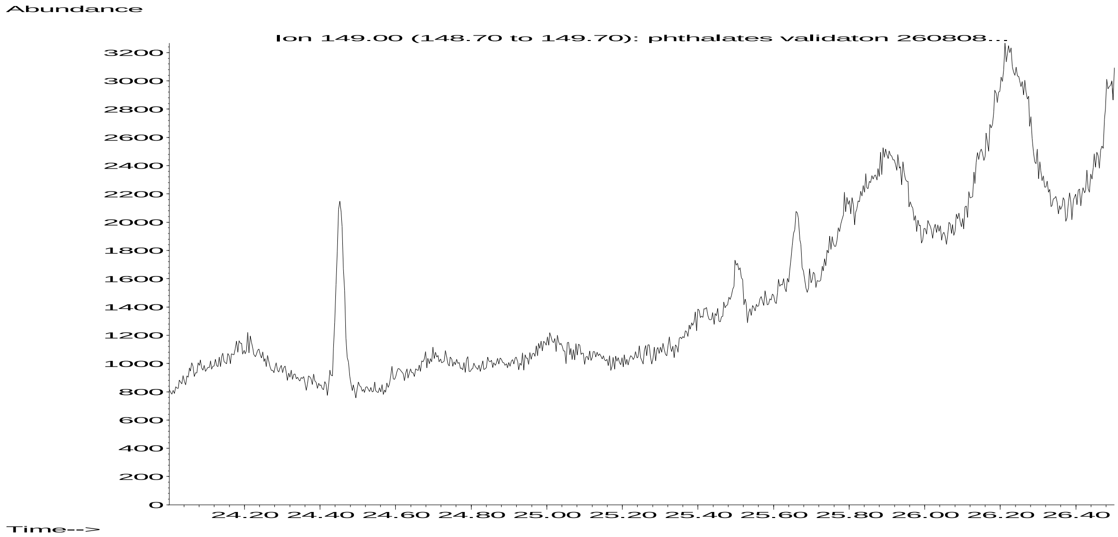
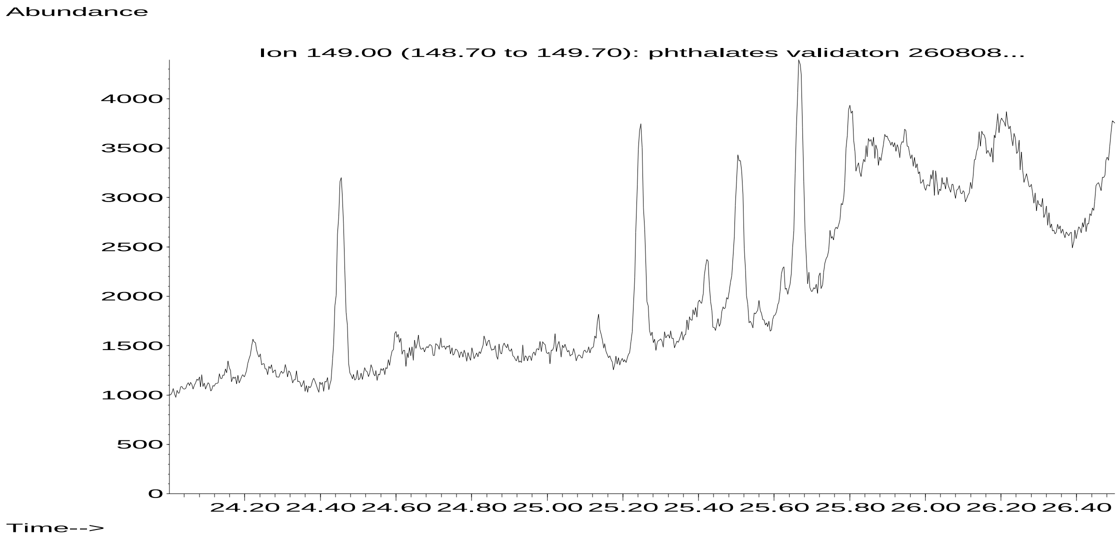


Figure 30. n-Octyl-n-decyl phthalate spiked into foods at the target LOQ

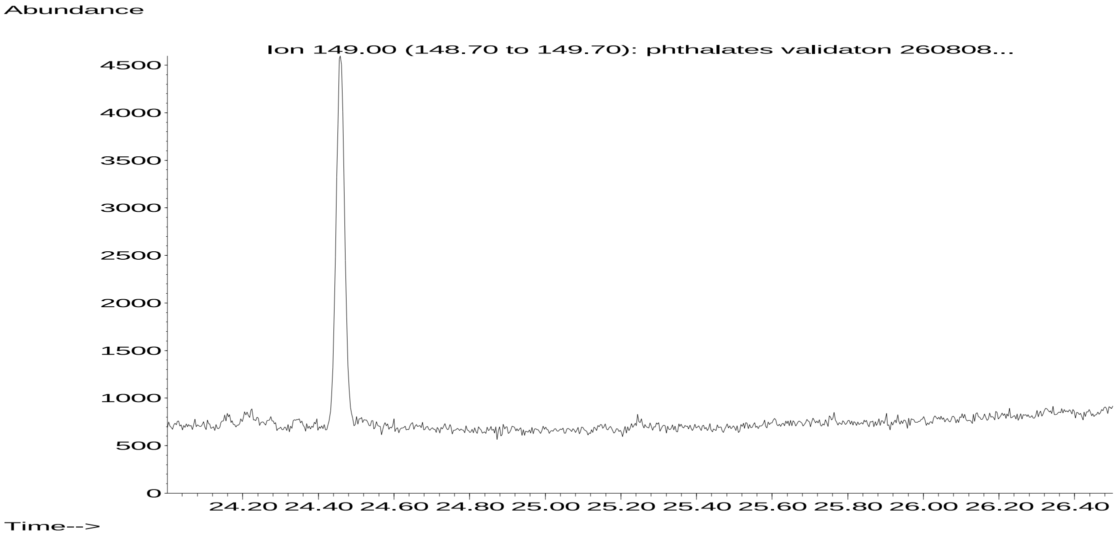
(a) Cheese only



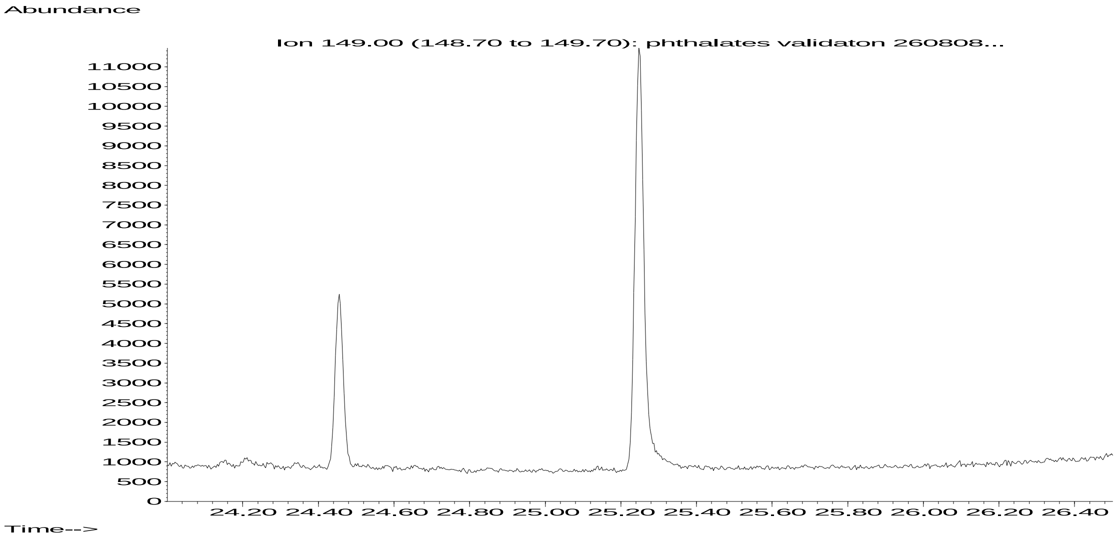
(b) Cheese overspiked with n-octyl-n-decyl phthalate at 0.05 mg/kg



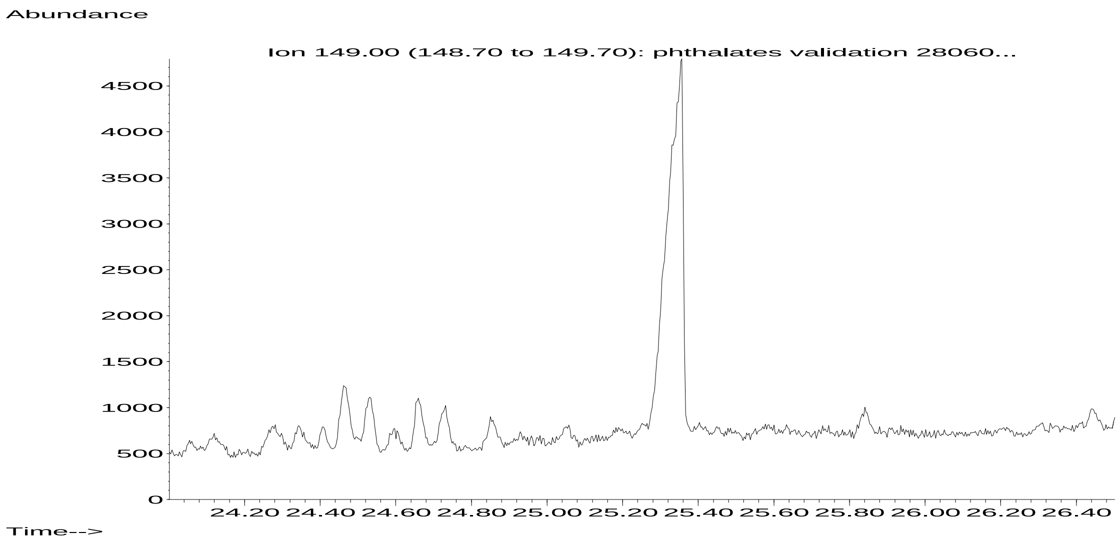
(c) Pork mince only



(d) Pork mince overspiked with n-octyl-n-decyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with n-octyl-n-decyl phthalate at 0.05 mg/kg

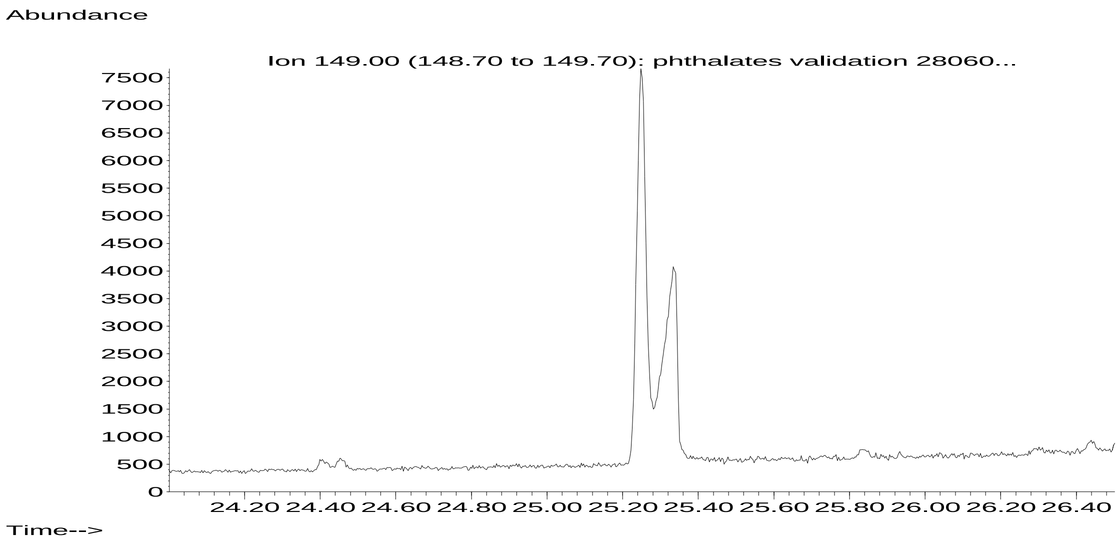
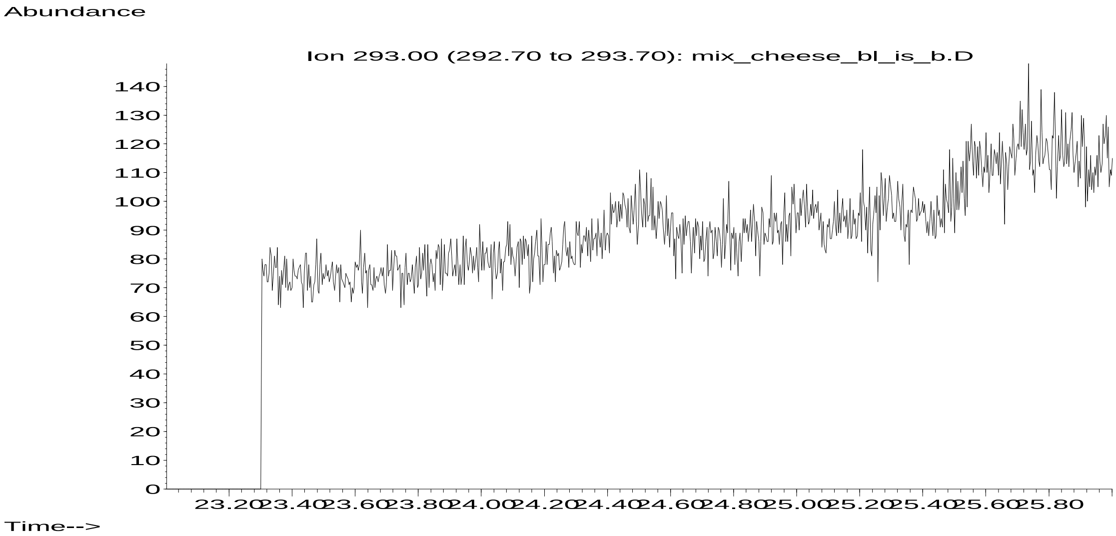
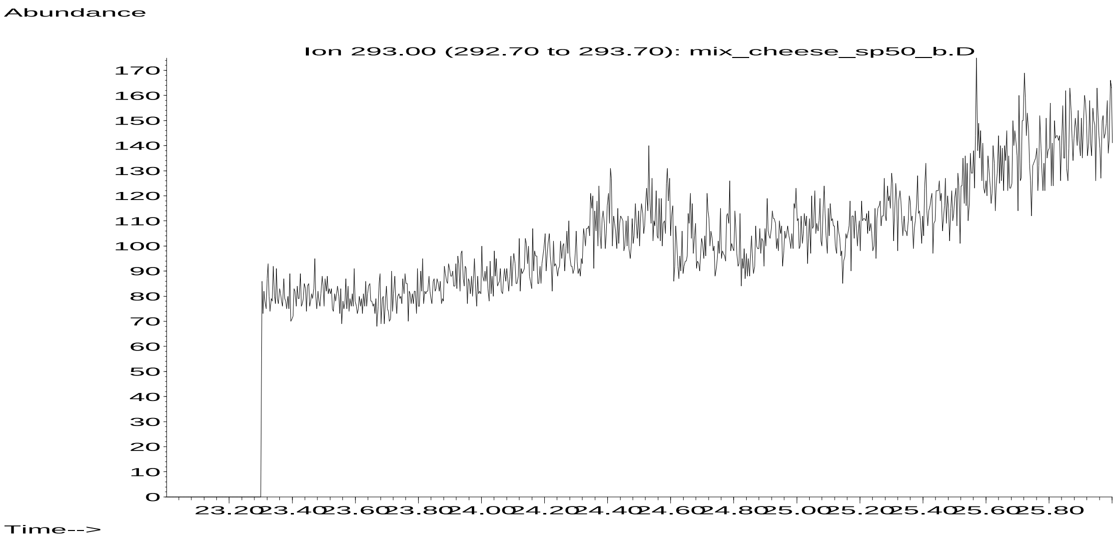


Figure 31. Diisononyl phthalate spiked into foods at the target LOQ

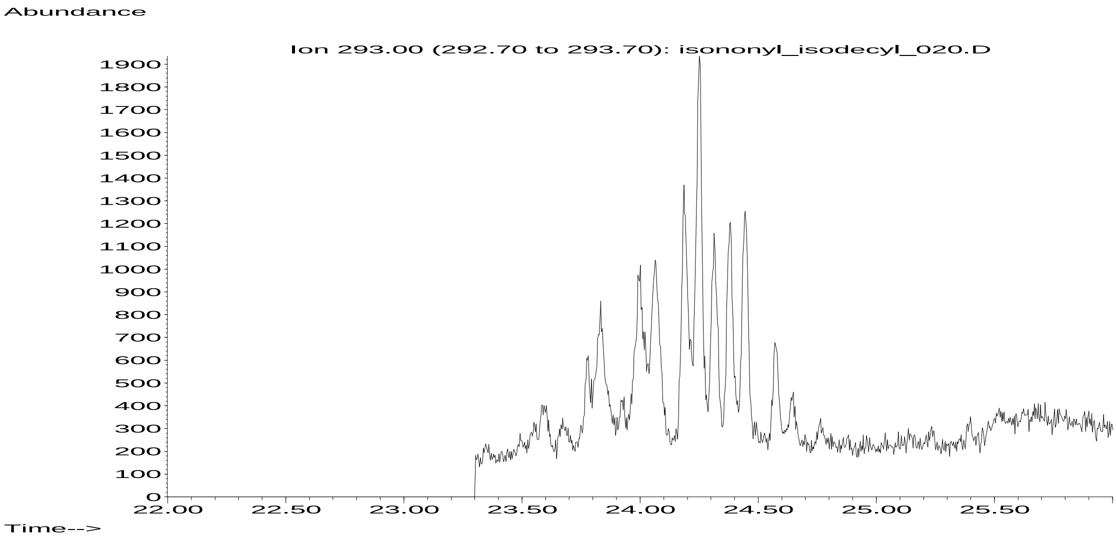
(a) Cheese only



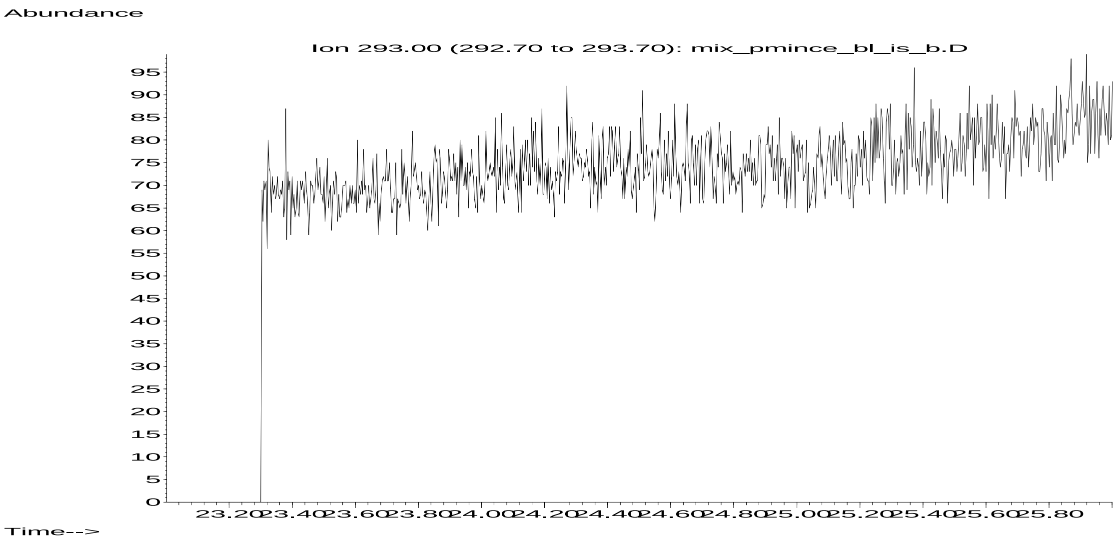
(b) Cheese overspiked with diisononyl phthalate at 0.05 mg/kg



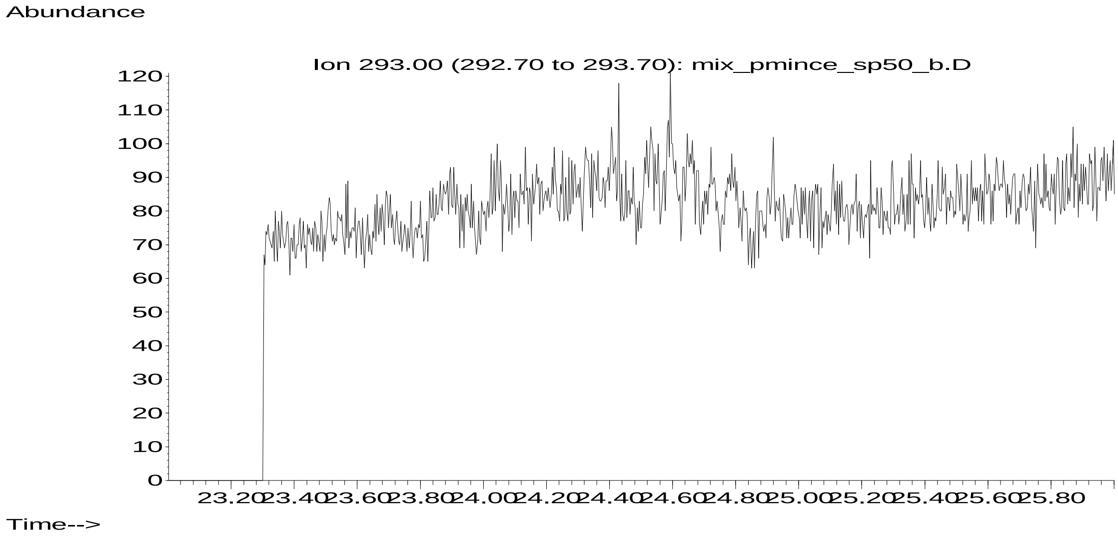
(c) Cheese overspiked with diisononyl phthalate at 1 mg/kg



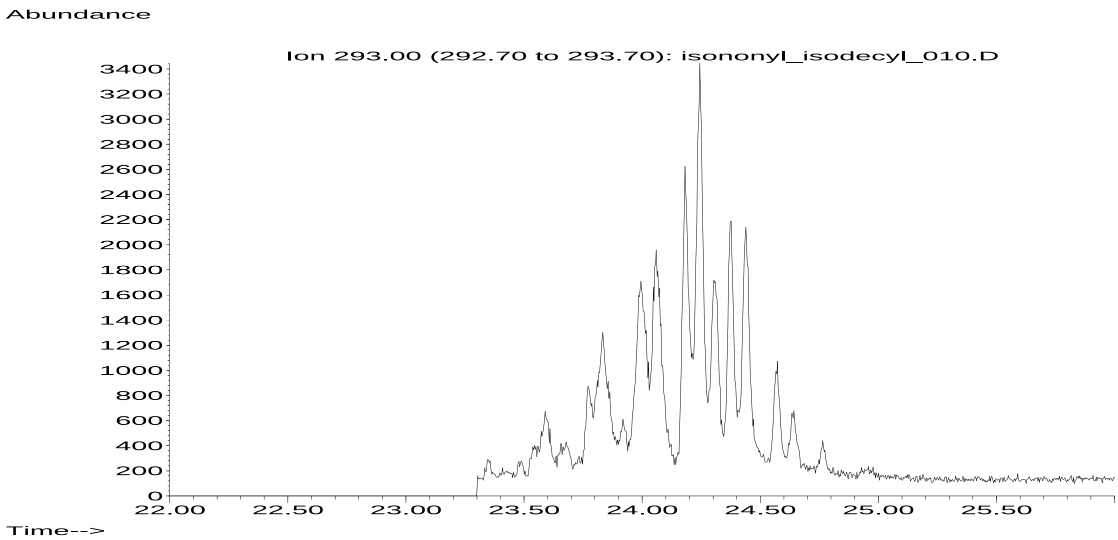
(d) Pork mince only



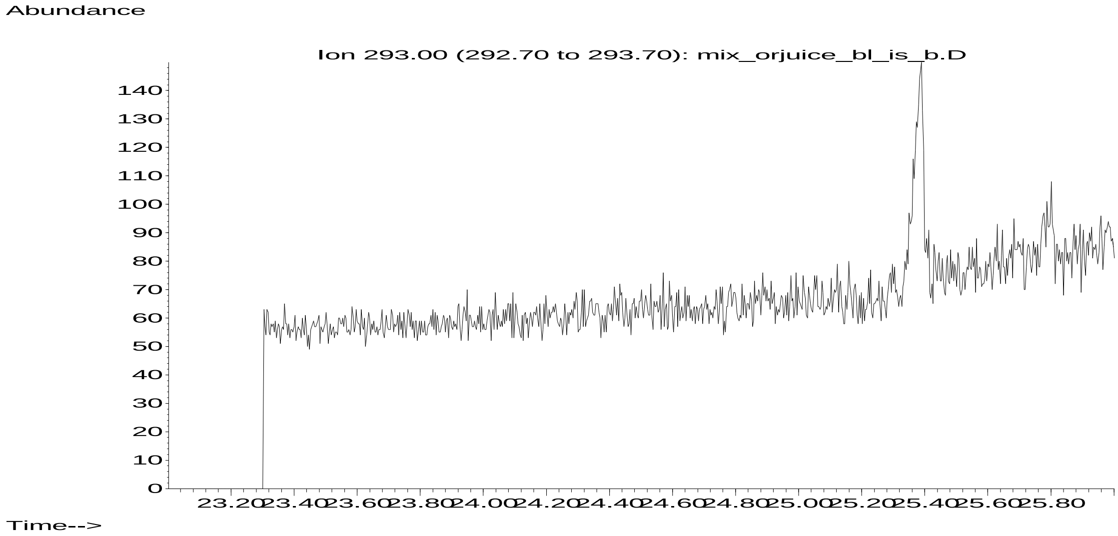
(e) Pork mince overspiked with diisononyl phthalate at 0.05 mg/kg



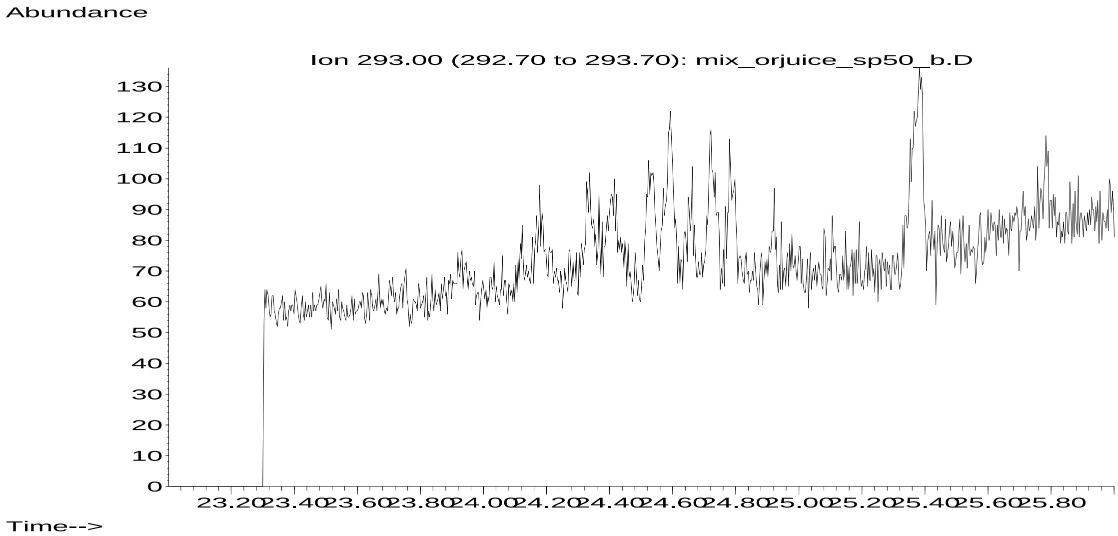
(f) Pork mince overspiked with diisononyl phthalate at 1 mg/kg



(g) Orange juice only



(h) Orange juice overspiked with diisononyl phthalate at 0.05 mg/kg



(i) Orange juice overspiked with diisononyl phthalate at 1 mg/kg

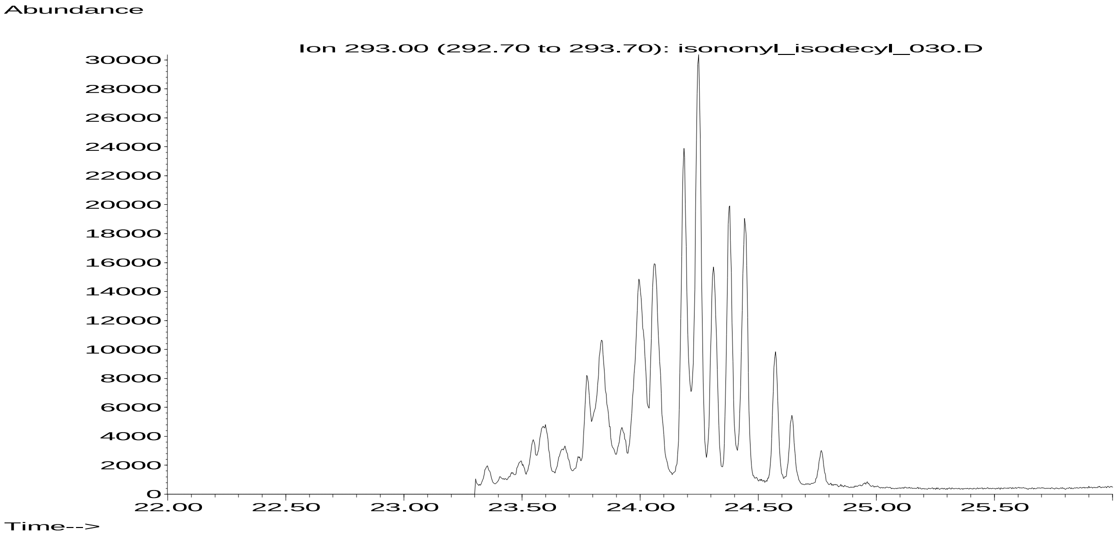
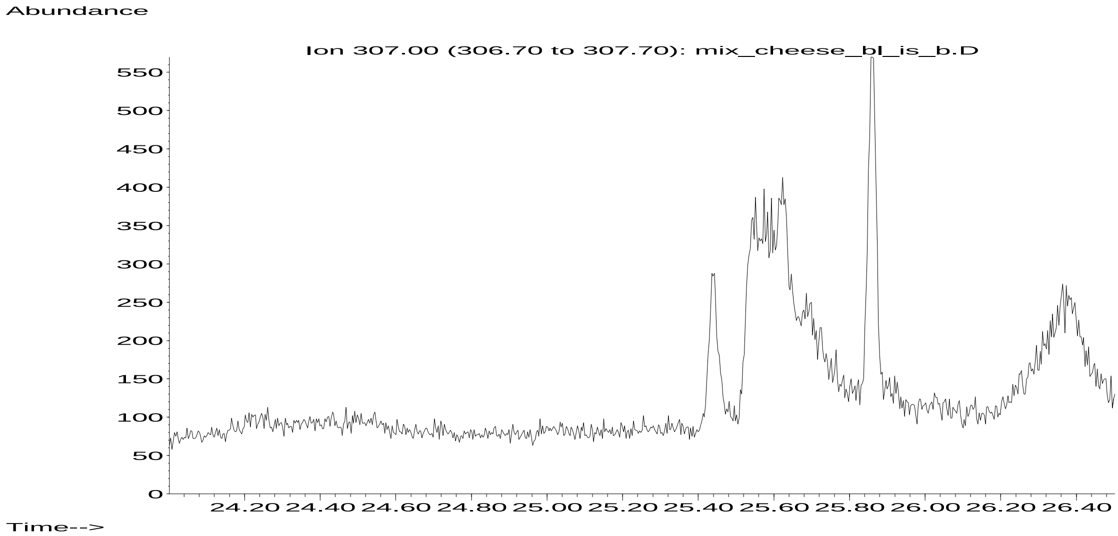
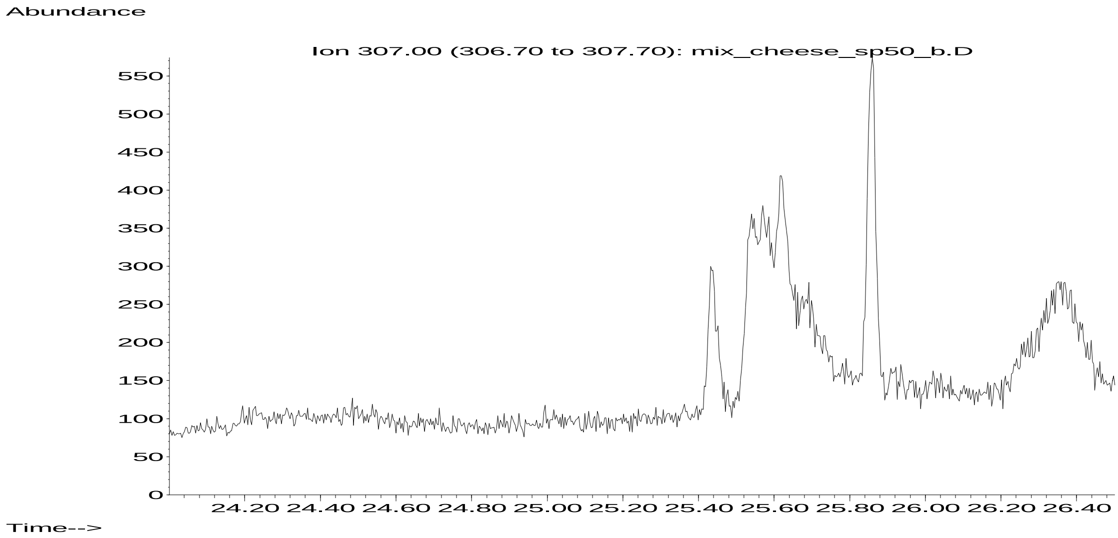


Figure 32. Diisodecyl phthalate spiked into foods at the target LOQ

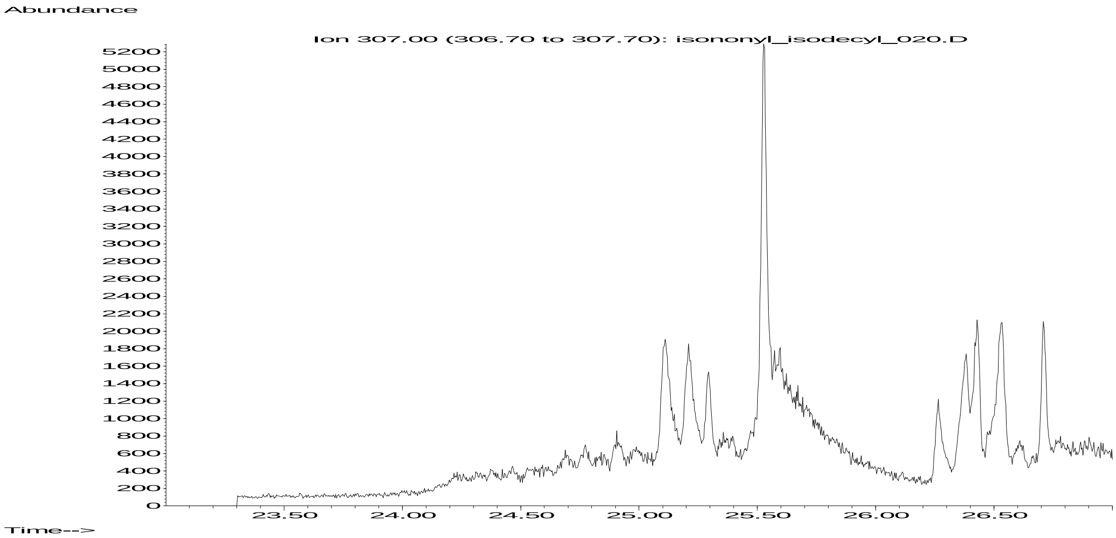
(a) Cheese only



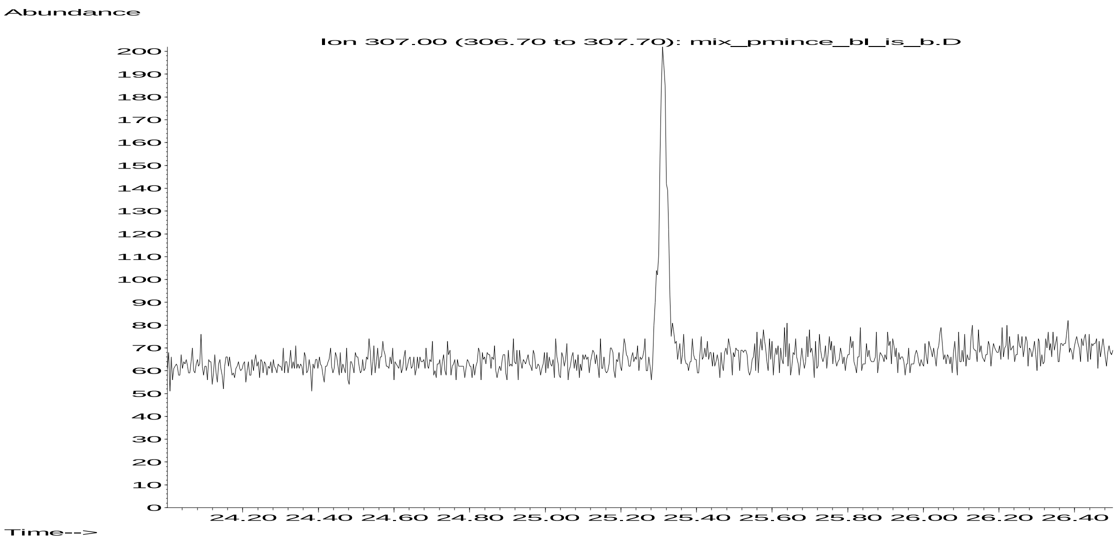
(b) Cheese overspiked with diisodecyl phthalate at 0.05 mg/kg



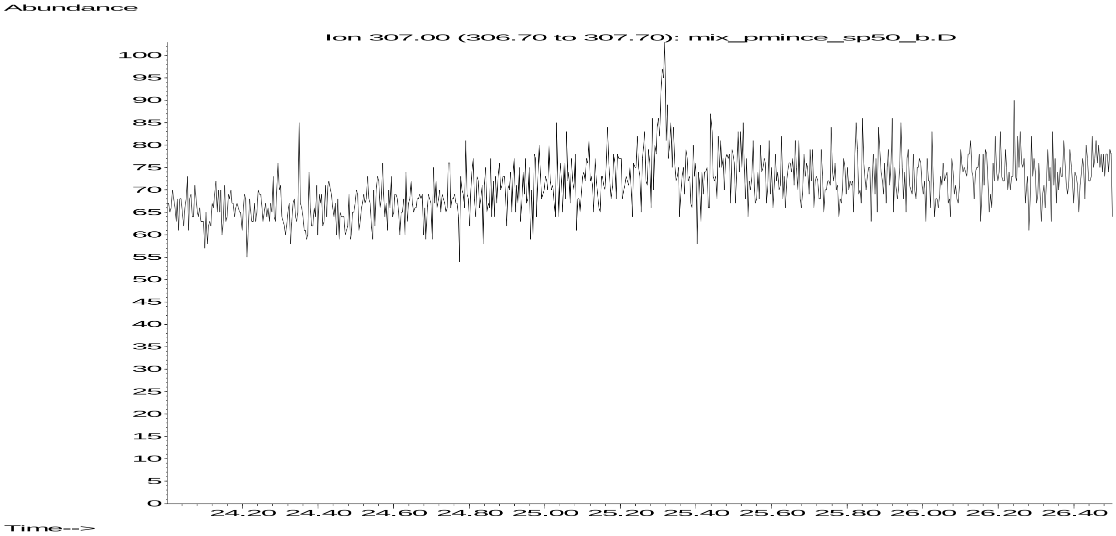
(c) Cheese overspiked with diisodecyl phthalate at 1 mg/kg



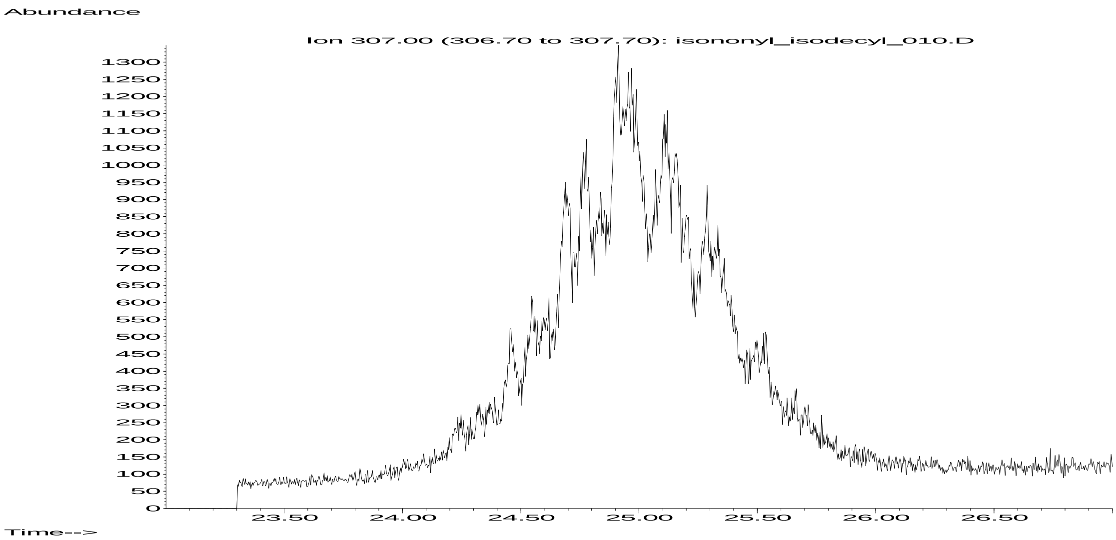
(d) Pork mince only



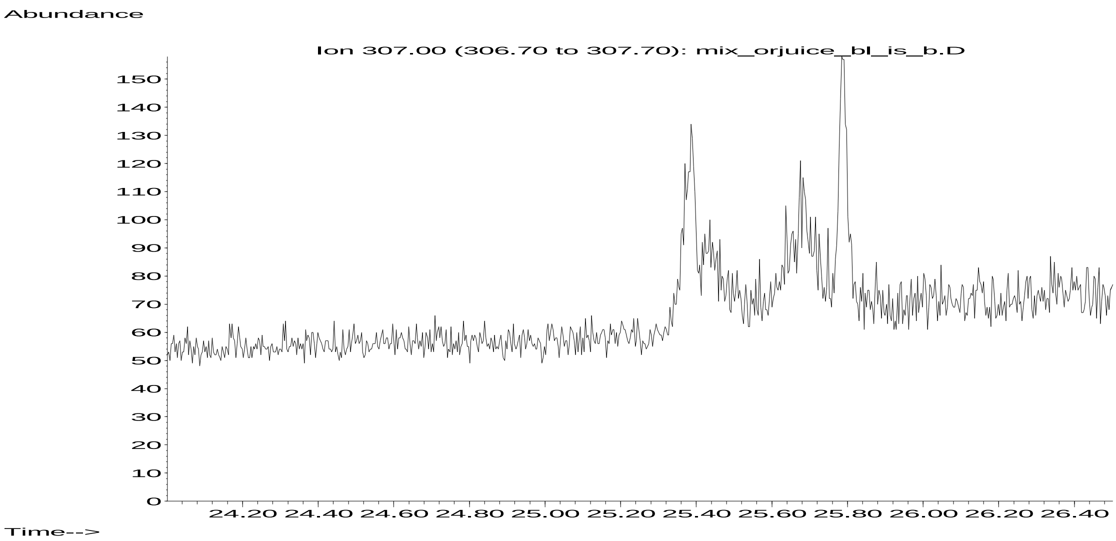
(e) Pork mince overspiked with diisodecyl phthalate at 0.05 mg/kg



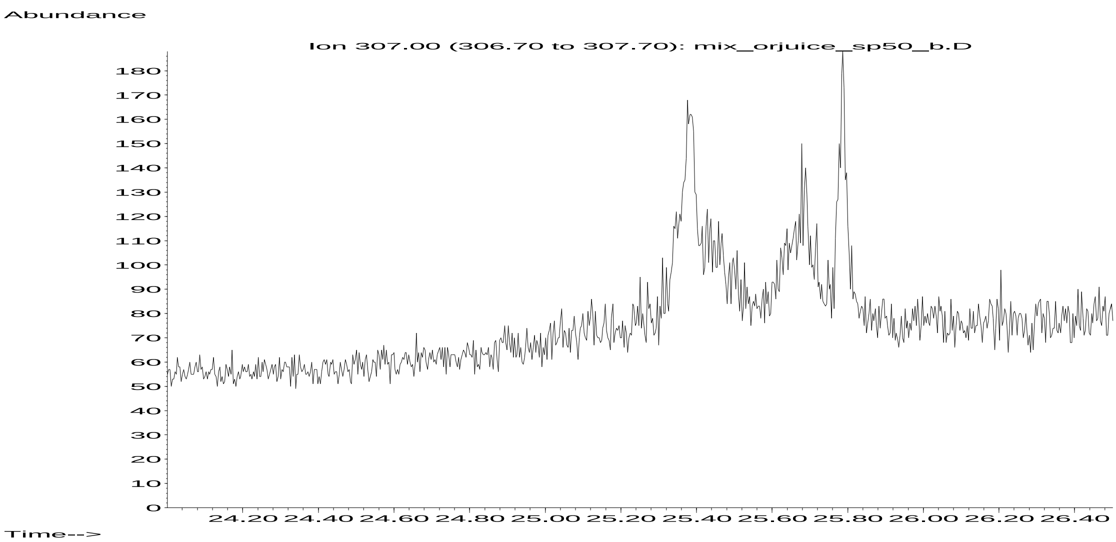
(f) Pork mince overspiked with diisodecyl phthalate at 1 mg/kg



(g) Orange juice only



(h) Orange juice overspiked with diisodecyl phthalate at 0.05 mg/kg



(i) Orange juice overspiked with diisodecyl phthalate at 1 mg/kg

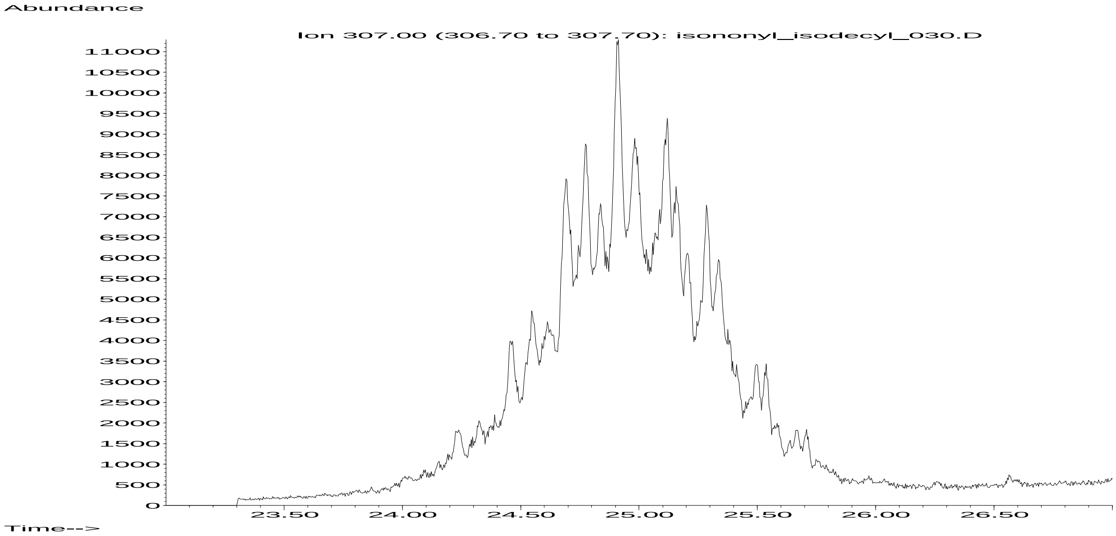
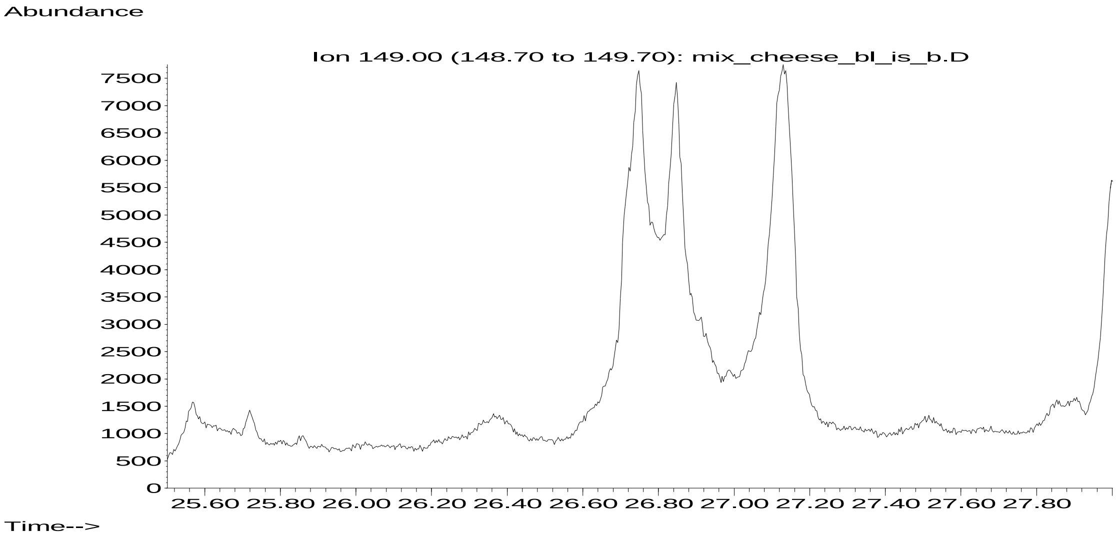
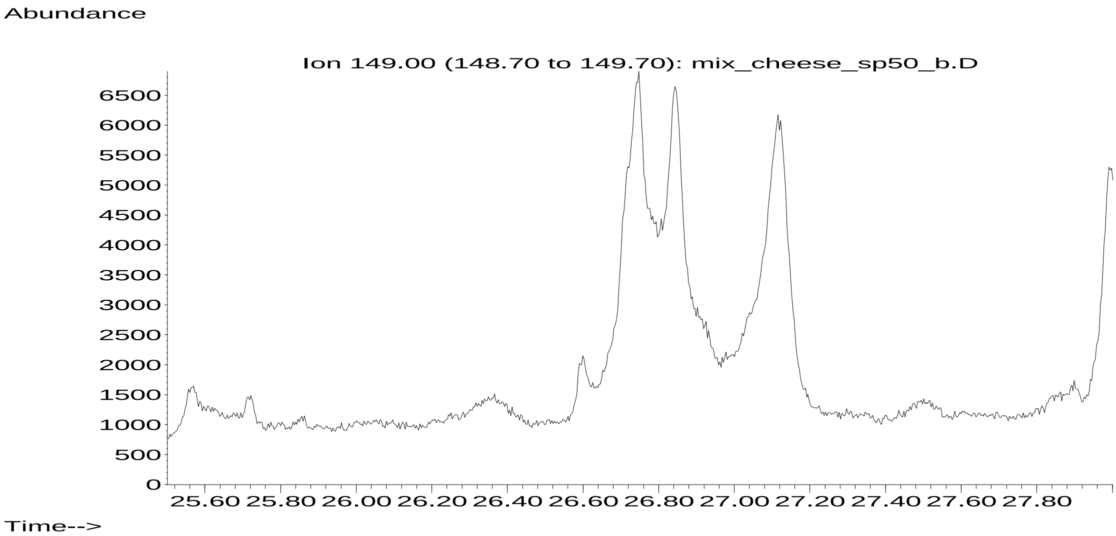


Figure 33. Di-n-decyl phthalate spiked into foods at the target LOQ

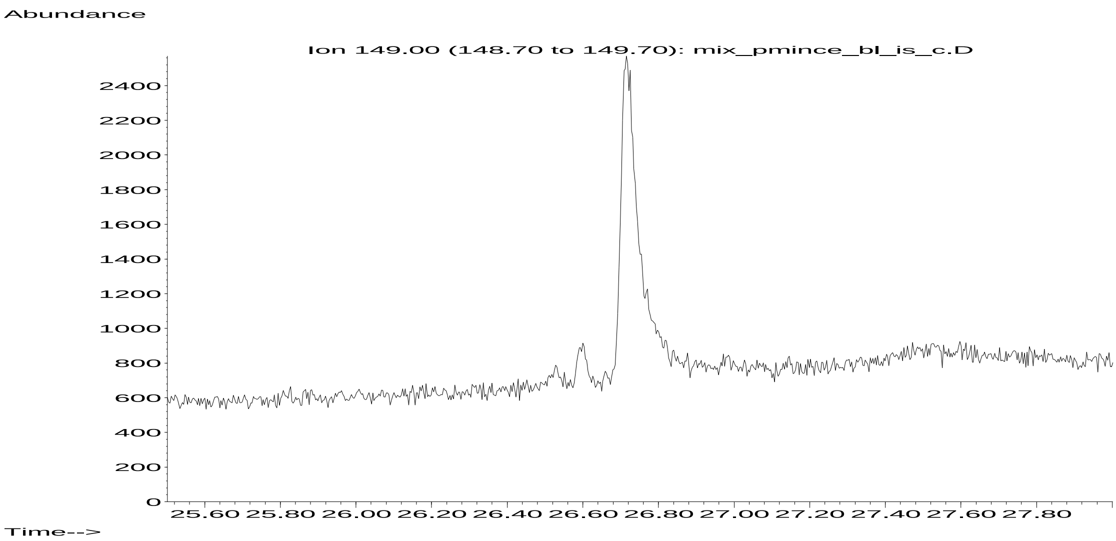
(a) Cheese only



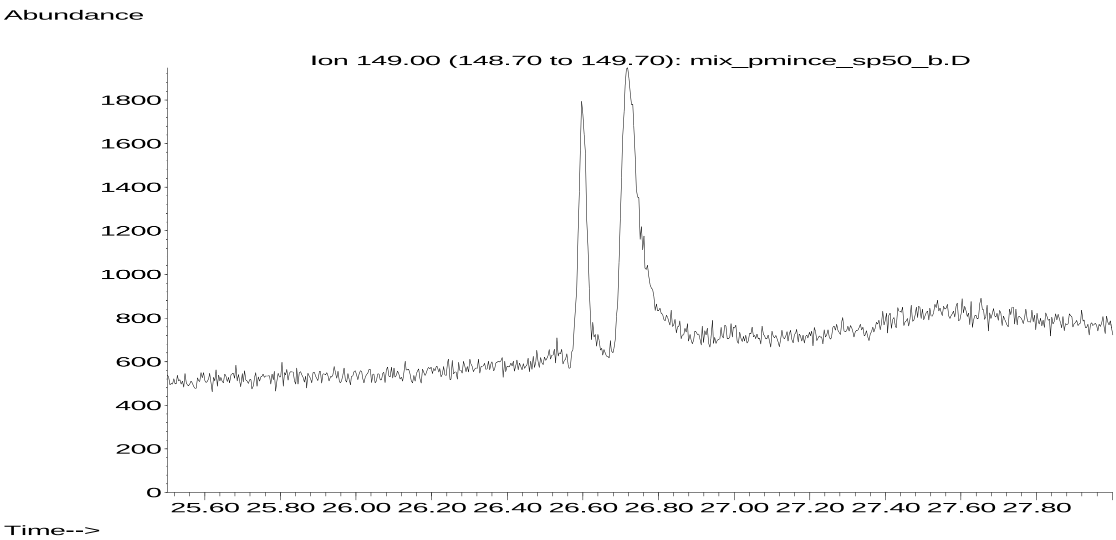
(b) Cheese overspiked with di-n-decyl phthalate at 0.05 mg/kg



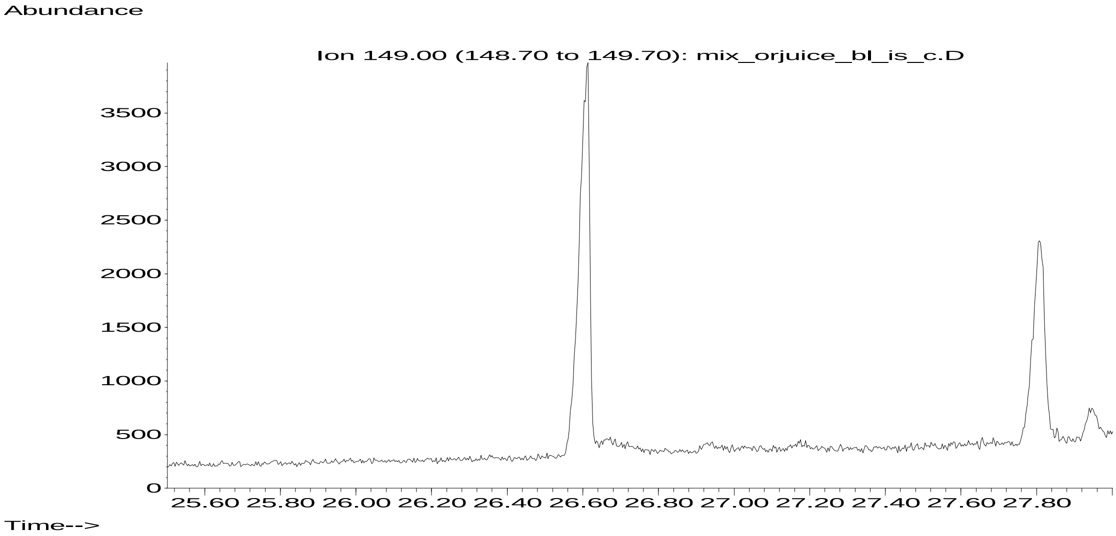
(c) Pork mince only



(d) Pork mince overspiked with di-n-decyl phthalate at 0.05 mg/kg



(e) Orange juice only



(f) Orange juice overspiked with di-n-decyl phthalate at 0.05 mg/kg

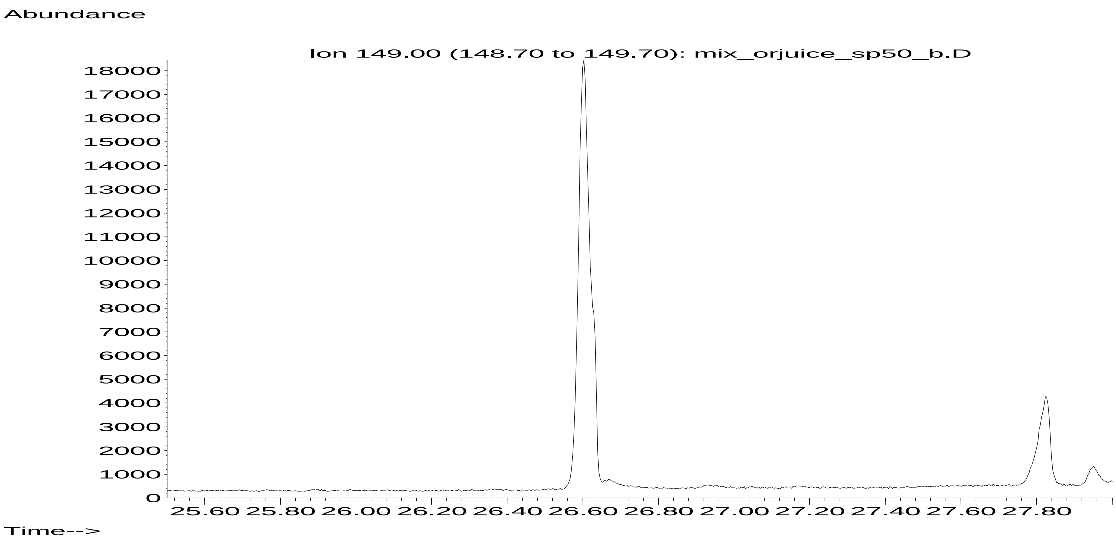


Figure 34. Calibration graph for dimethyl phthalate in acetonitrile

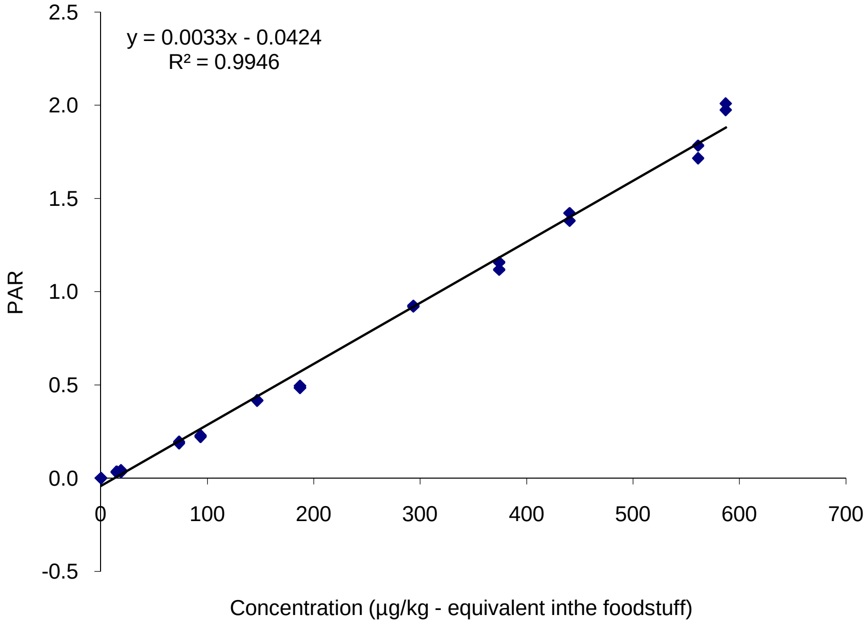


Figure 35. Calibration graph for diethyl phthalate in acetonitrile

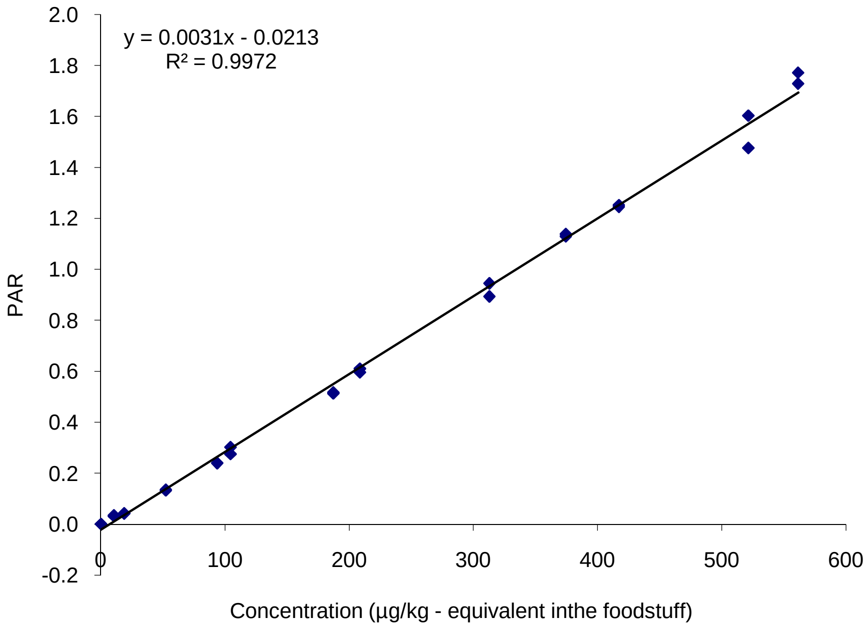


Figure 36. Calibration graph for diisopropyl phthalate in acetonitrile

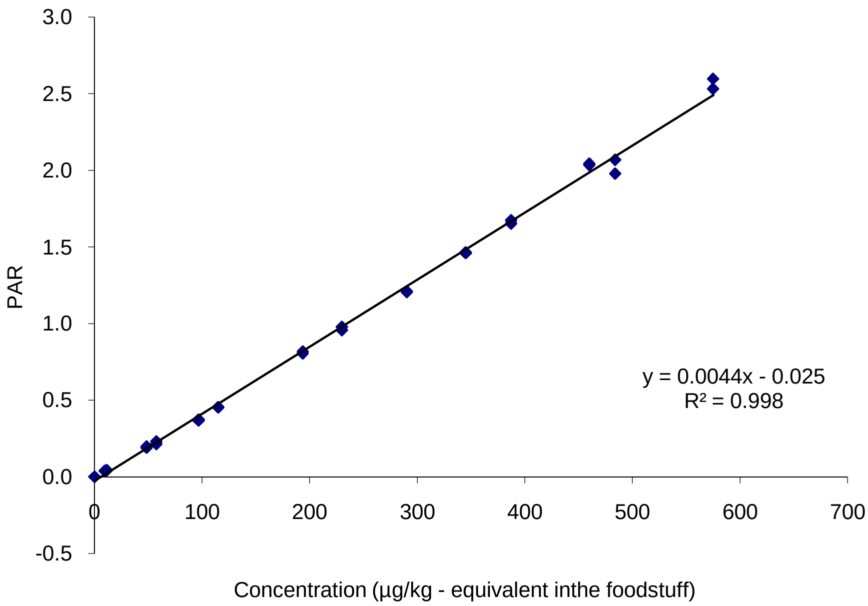


Figure 37. Calibration graph for diallyl phthalate in acetonitrile

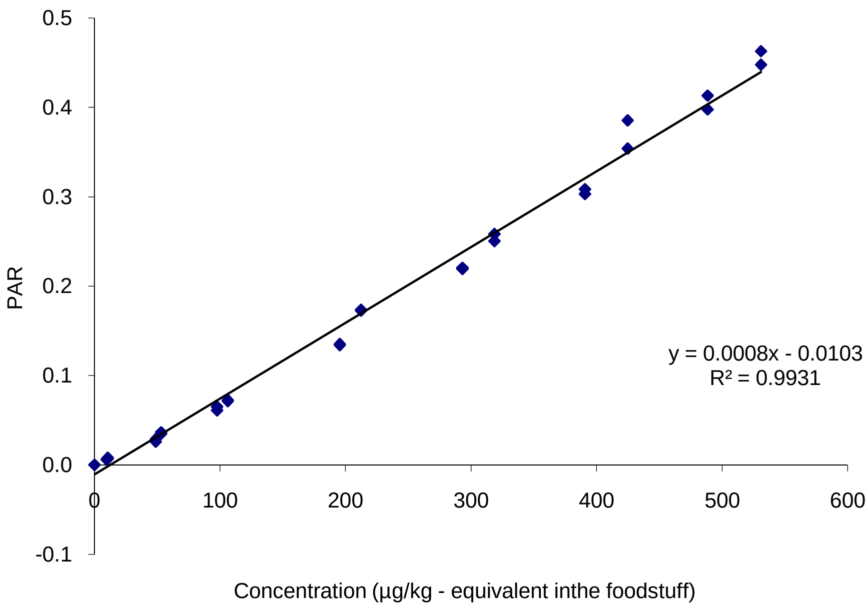


Figure 38. Calibration graph for diisobutyl phthalate in acetonitrile

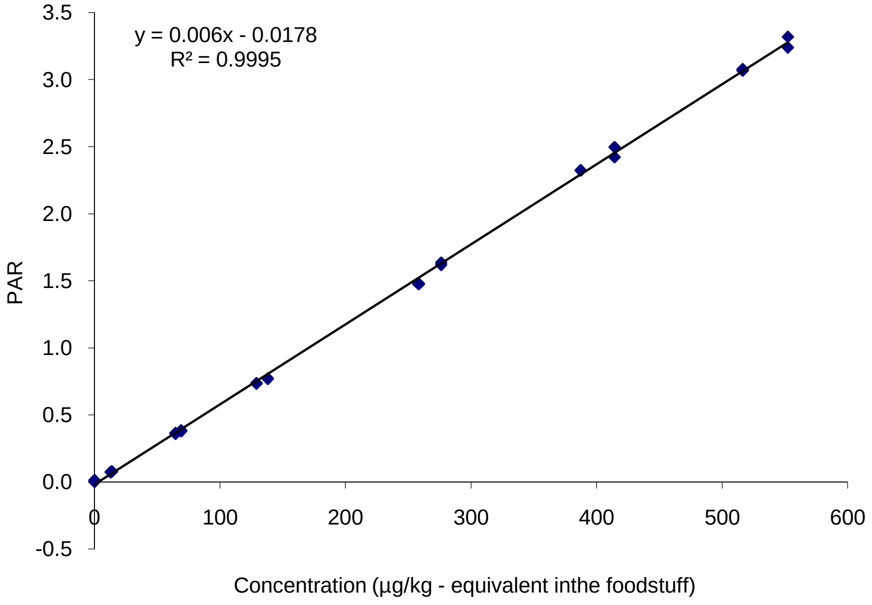


Figure 39. Calibration graph for dibutyl phthalate in acetonitrile

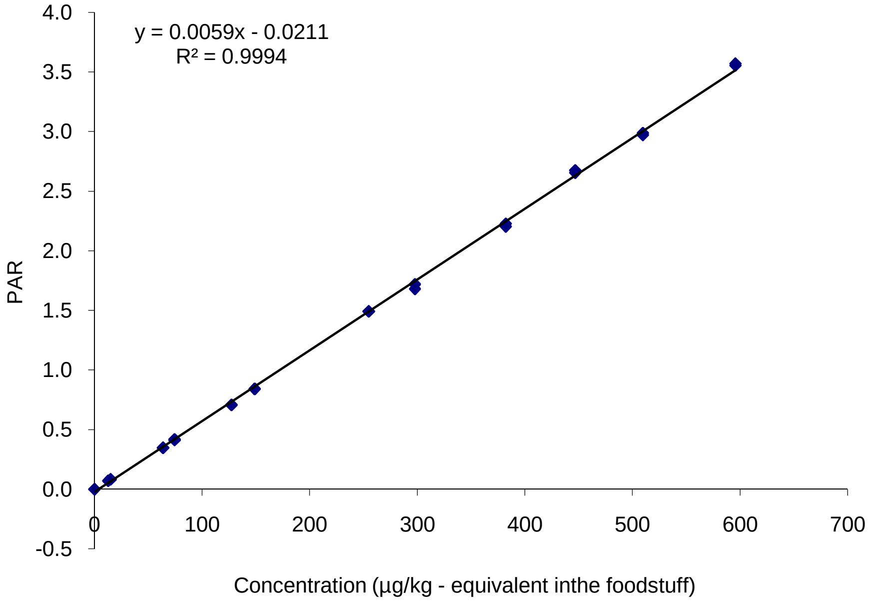


Figure 40. Calibration graph for dipentyl phthalate in acetonitrile

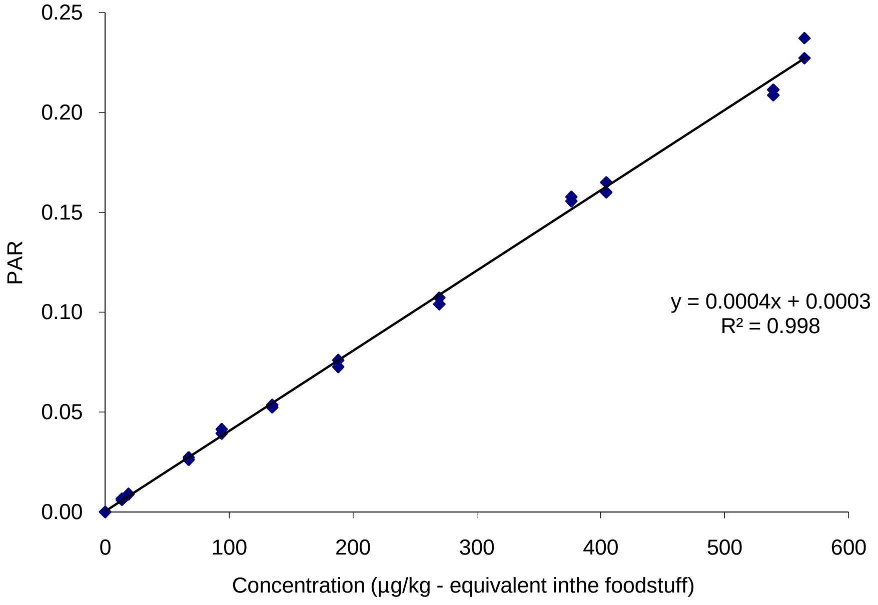


Figure 41. Calibration graph for di-n-hexyl phthalate in acetonitrile (concentration corrected to 17% of the total mass – see text)

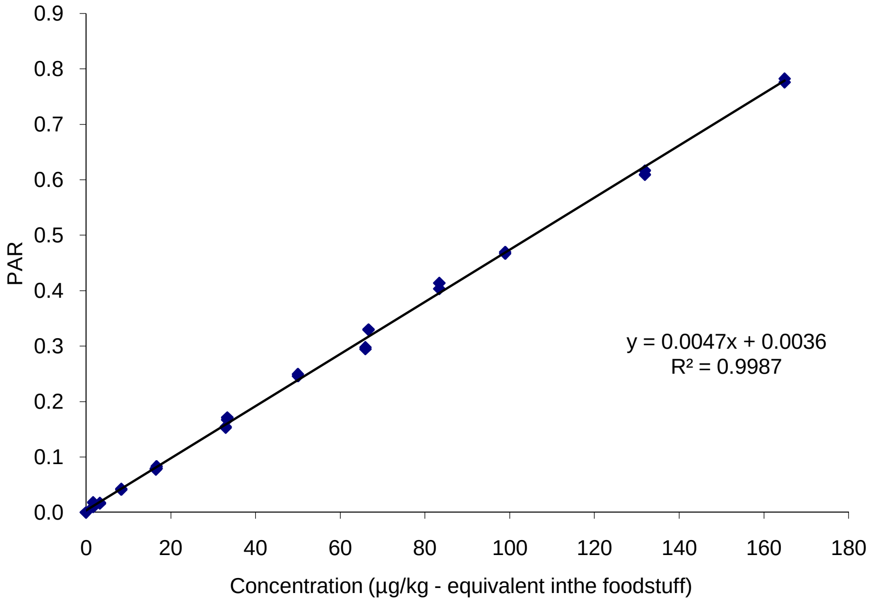


Figure 42. Calibration graph for benzyl butyl phthalate in acetonitrile

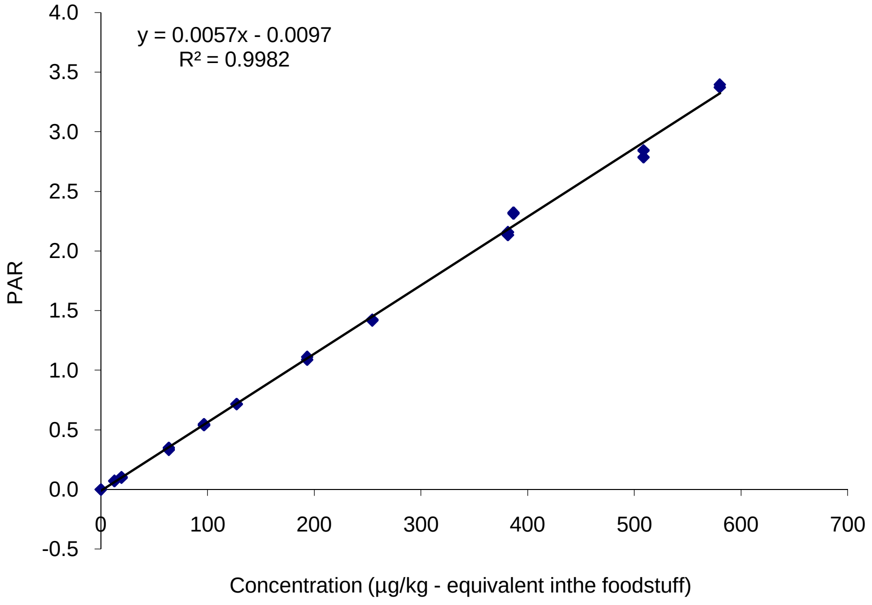


Figure 43. Calibration graph for dicyclohexyl phthalate in acetonitrile

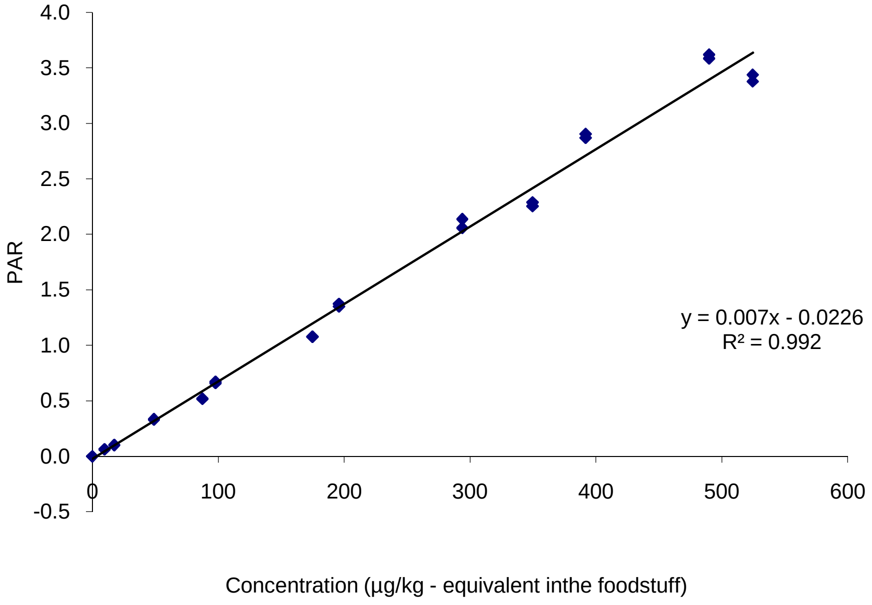


Figure 44. Calibration graph for di-(2-ethylhexyl) phthalate in acetonitrile

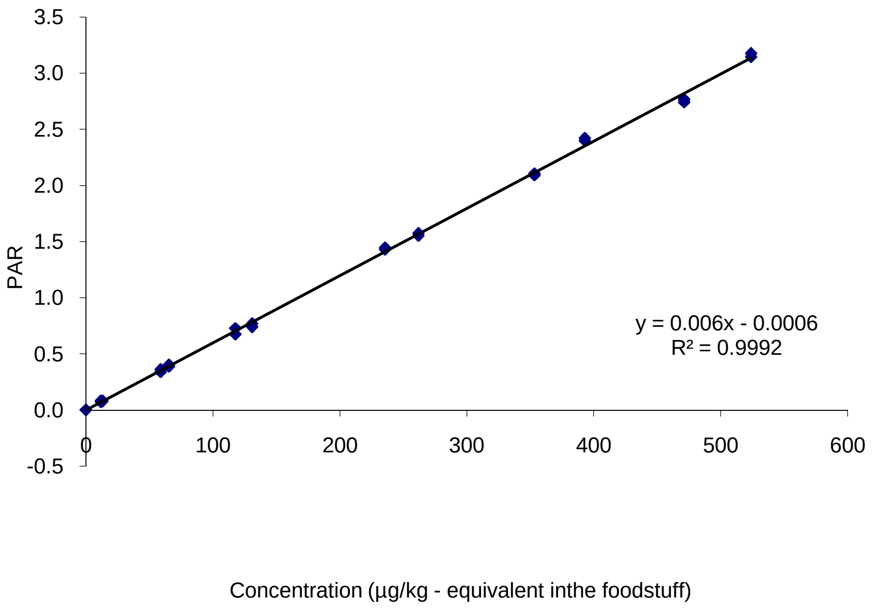


Figure 45. Calibration graph for di-n-heptyl phthalate in acetonitrile

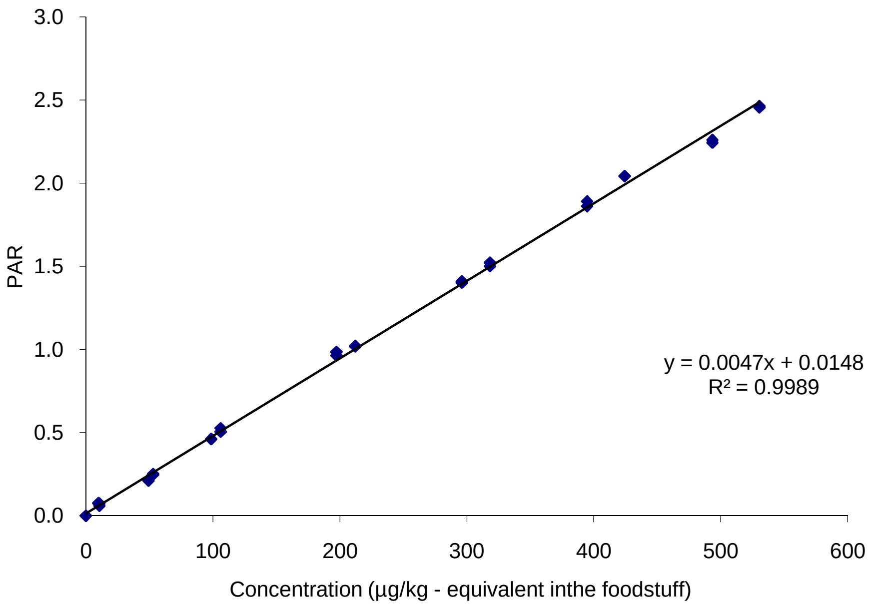


Figure 46. Calibration graph for di-n-octyl phthalate in acetonitrile

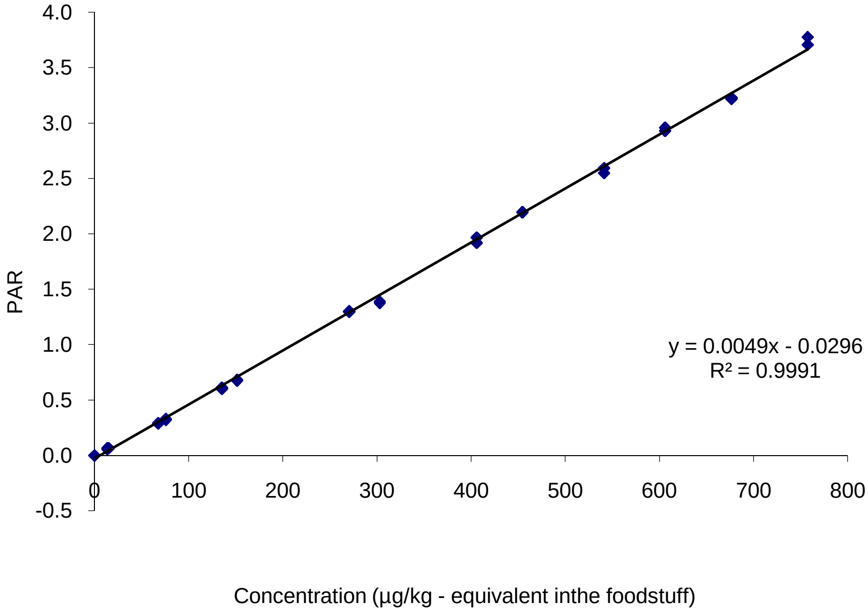


Figure 47. Calibration graph for n-octyl-n-decyl phthalate in acetonitrile (concentration corrected to 58% of the total mass – see text)

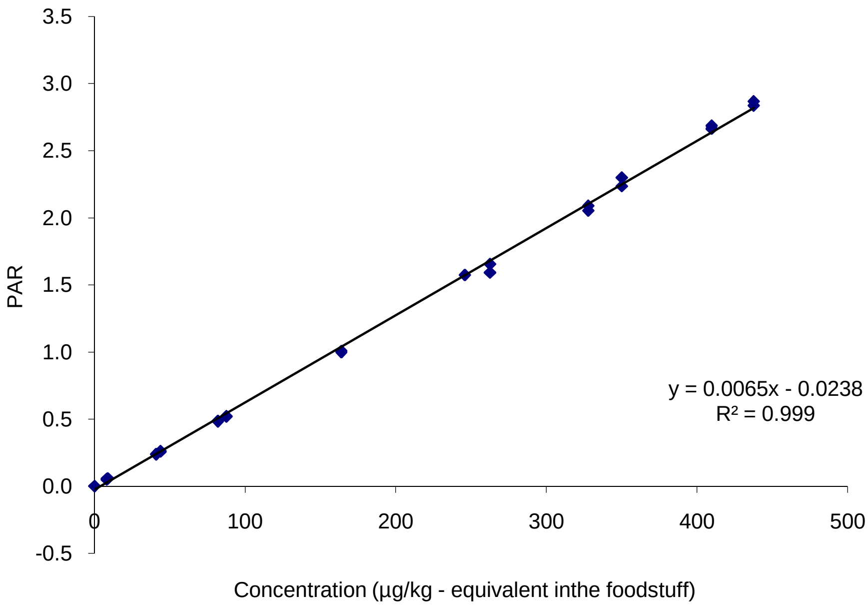


Figure 48. Calibration graph for diisononyl phthalate in acetonitrile

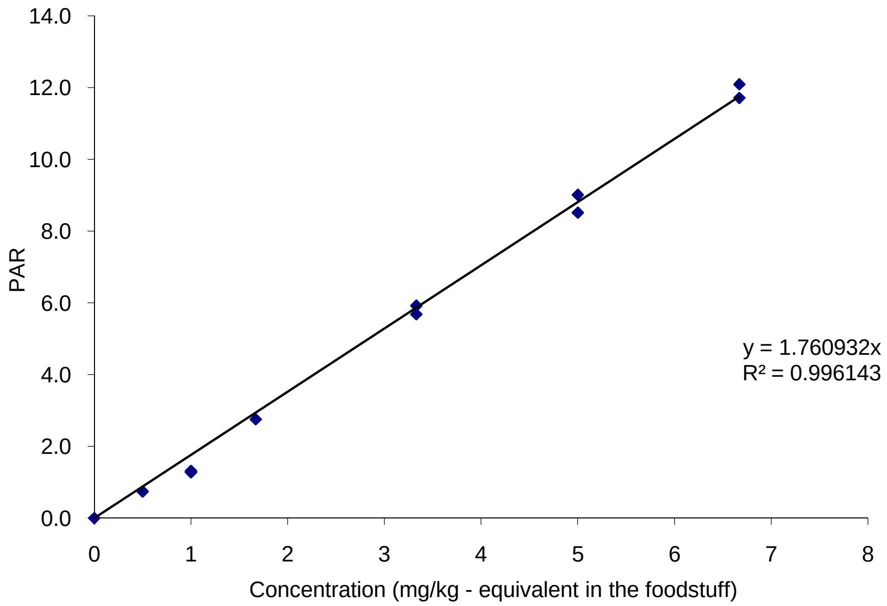


Figure 49. Calibration graph for diisodecyl phthalate in acetonitrile

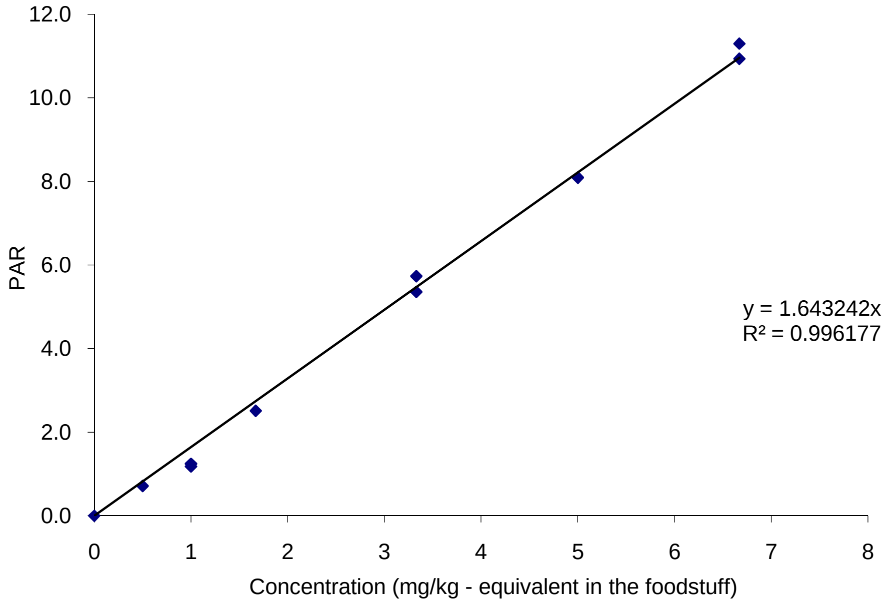


Figure 50. Calibration graph for di-n-decyl phthalate in acetonitrile

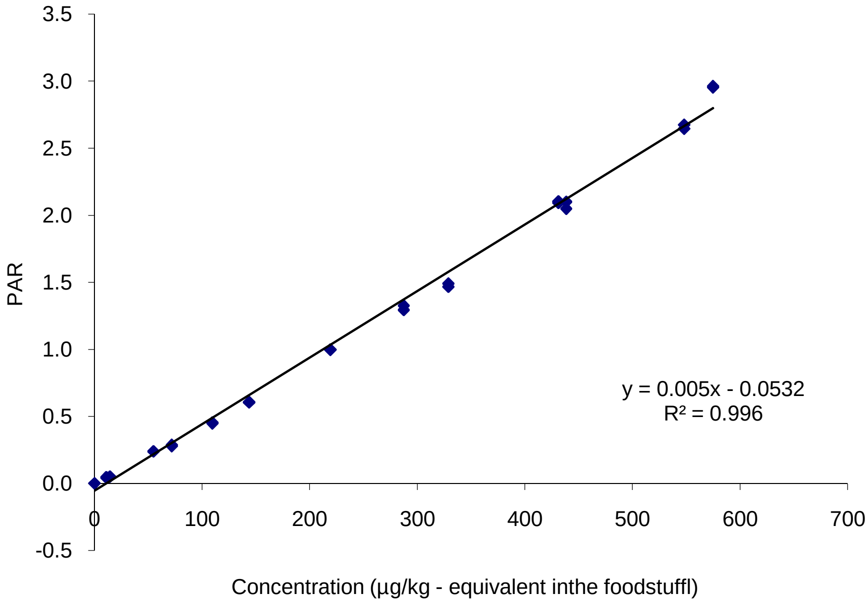
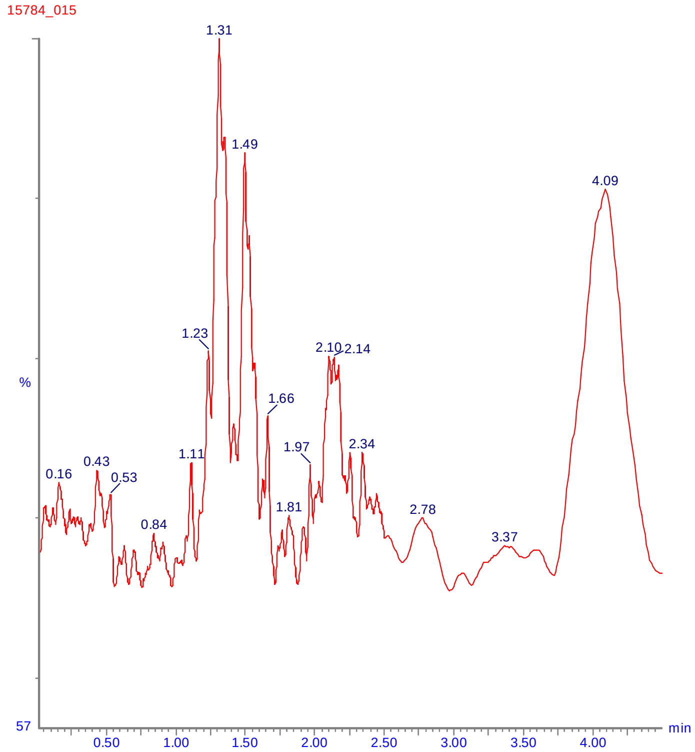
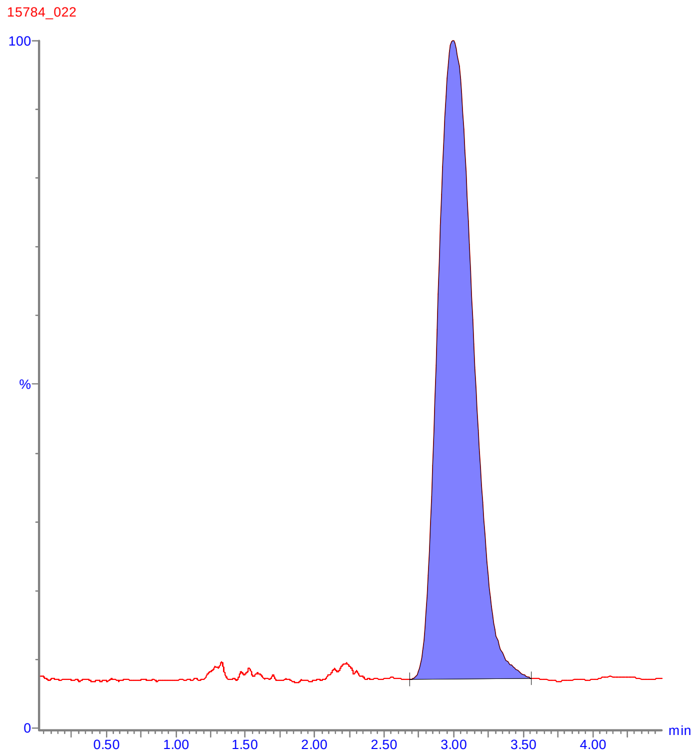


Figure 51. Monoisopropyl phthalate spiked into foods at the target LOQ

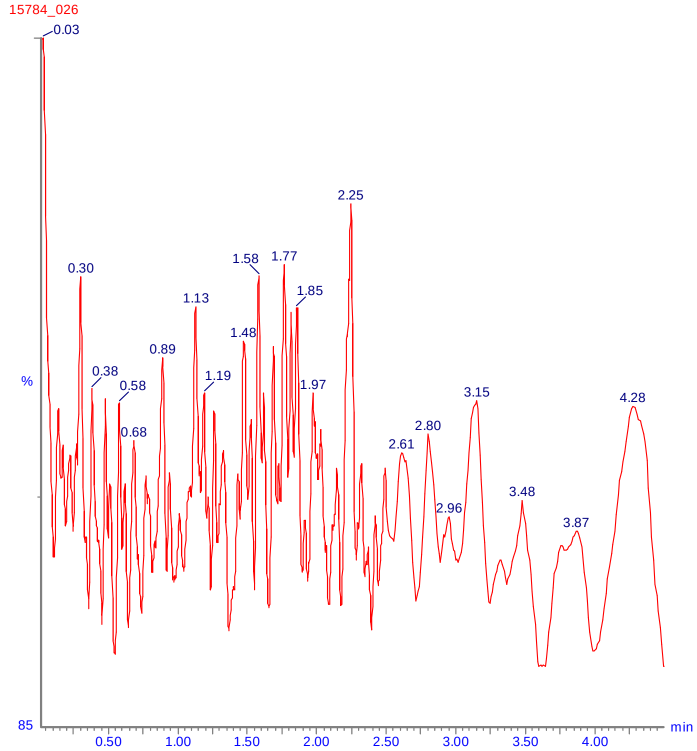
(a) Cheese blank



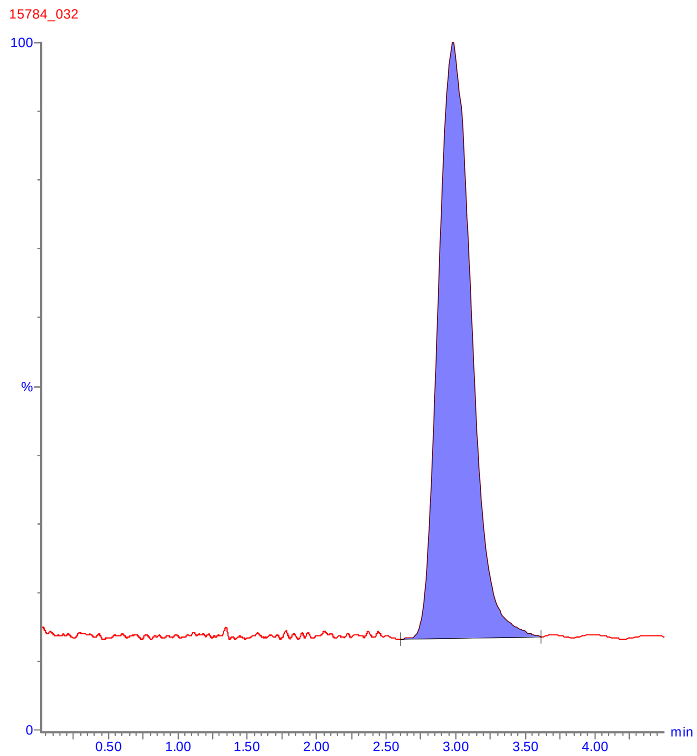
(b) Cheese + overspike at 0.05 mg/kg



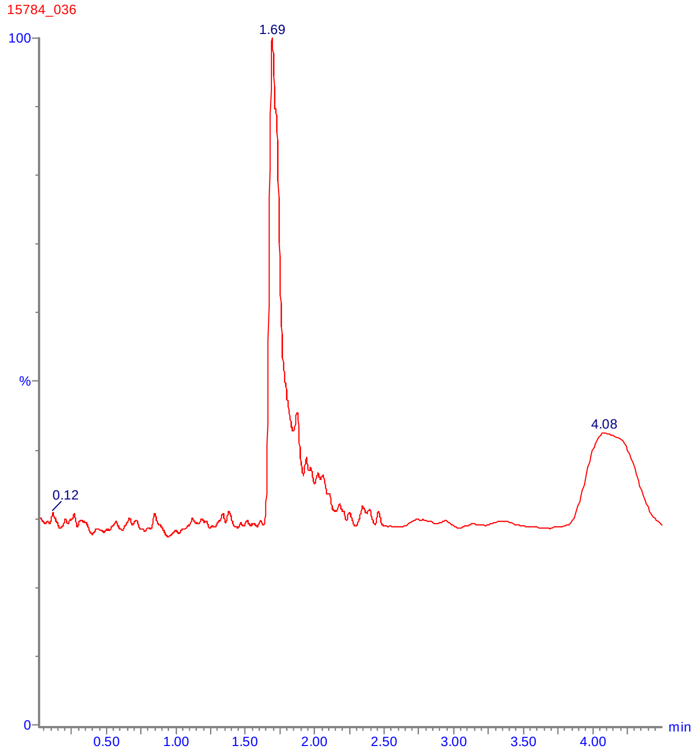
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

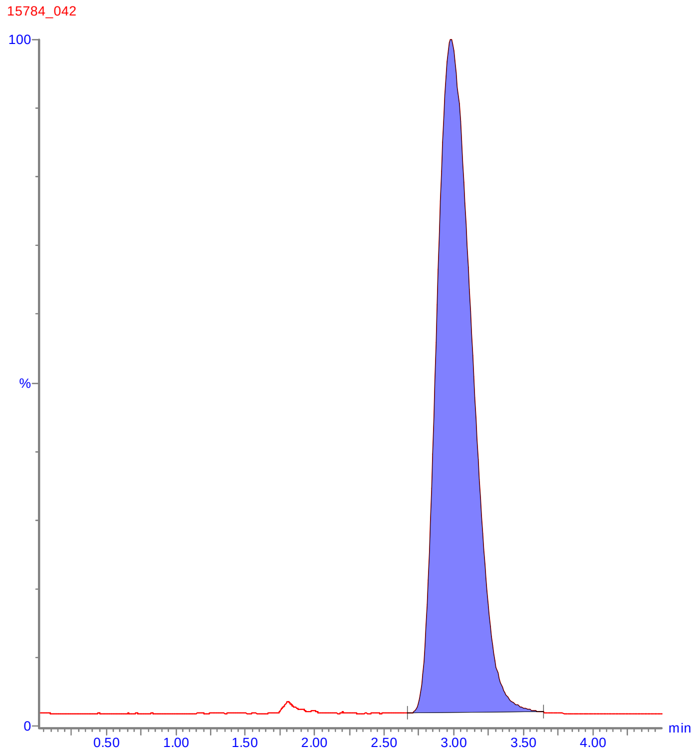
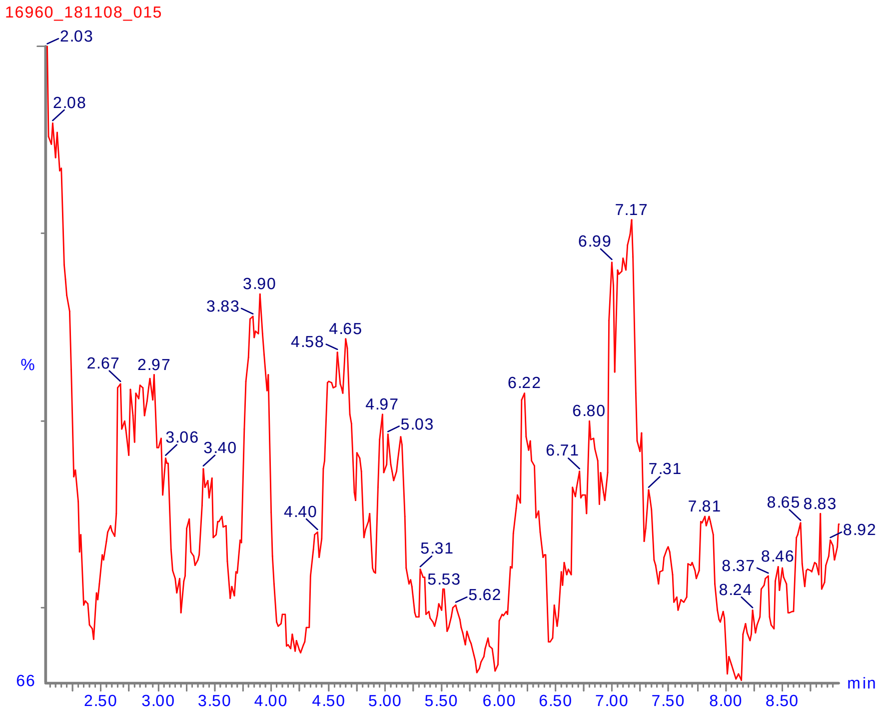
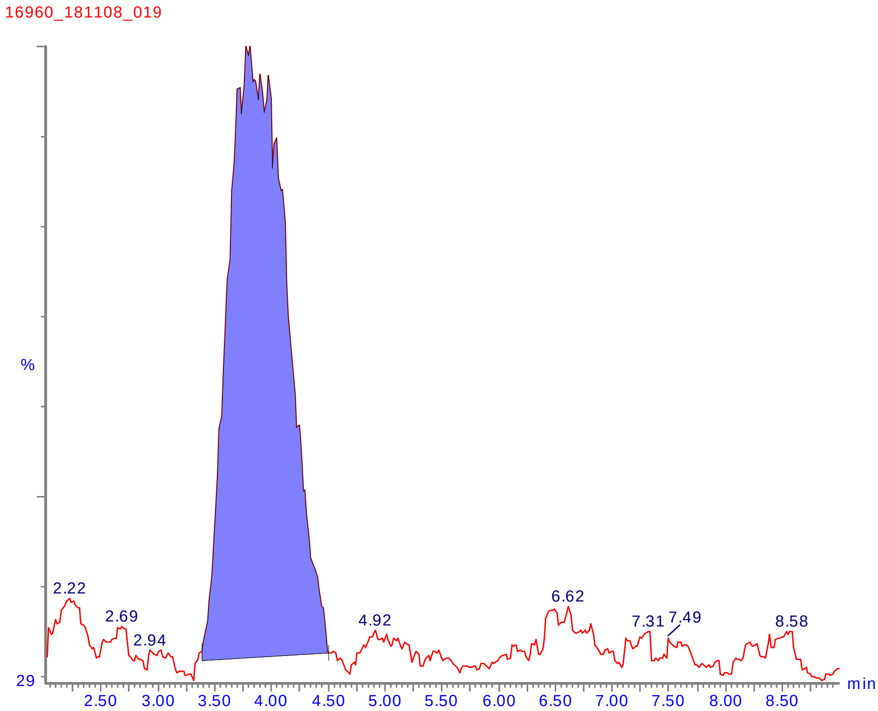


Figure 52. Total monoisobutyl phthalate and mono-n-butyl phthalate spiked into foods at the target LOQ

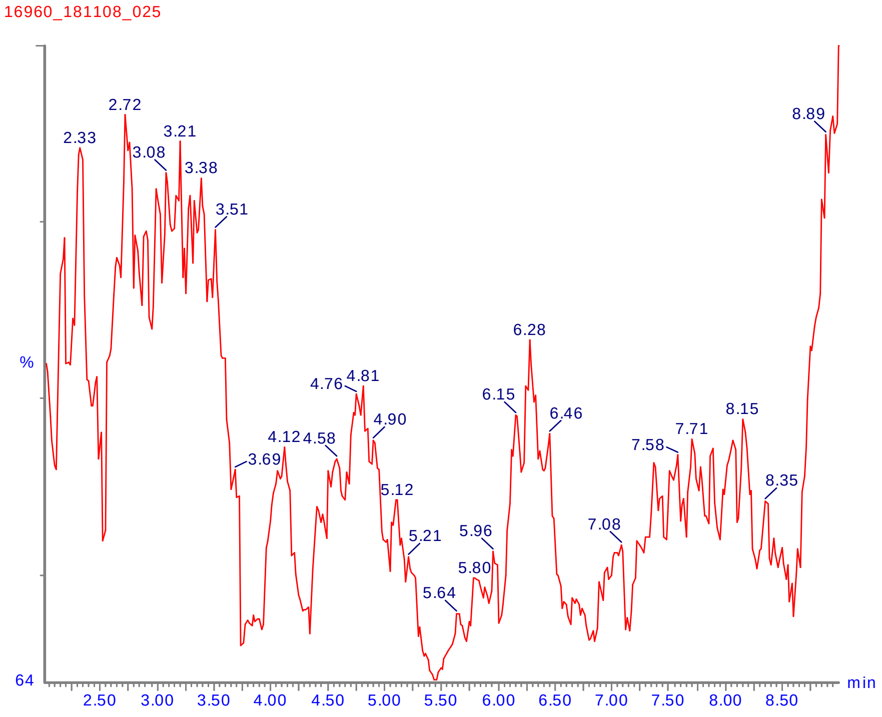
(a) Cheese blank



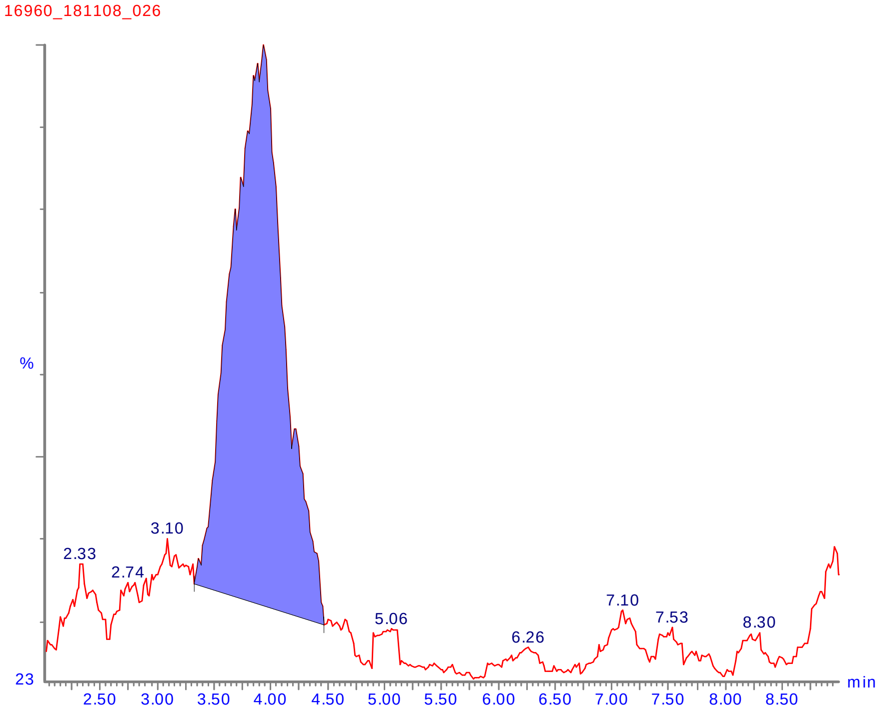
(b) Cheese + overspike at 0.05 mg/kg monoisobutyl phthalate and 0.05 mg/kg mono-n-butyl phthalate



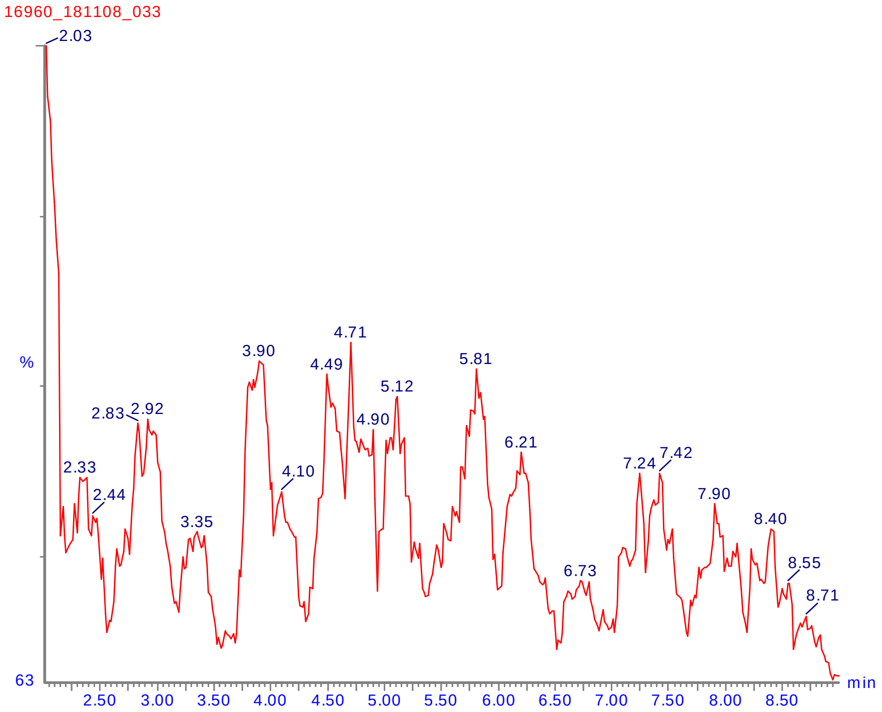
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg monoisobutyl phthalate and 0.05 mg/kg mono-n-butyl phthalate



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg monoisobutyl phthalate and 0.05 mg/kg mono-n-butyl phthalate

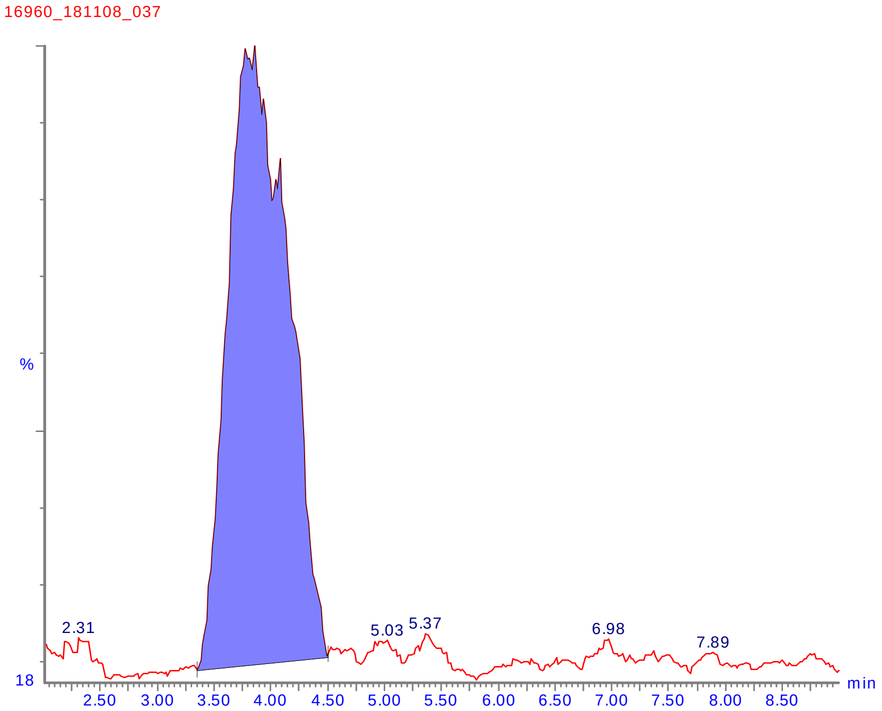
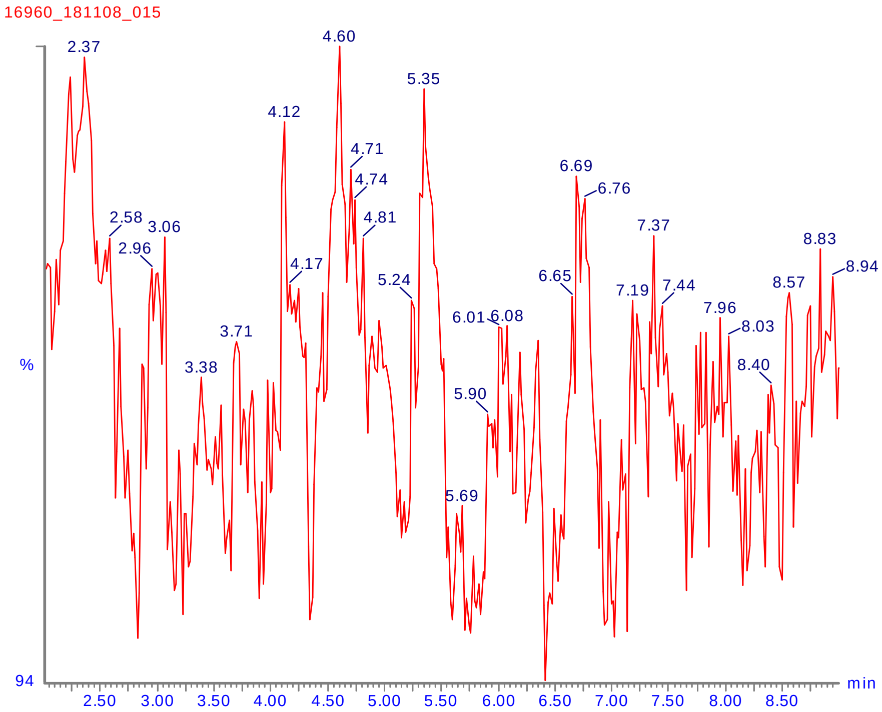
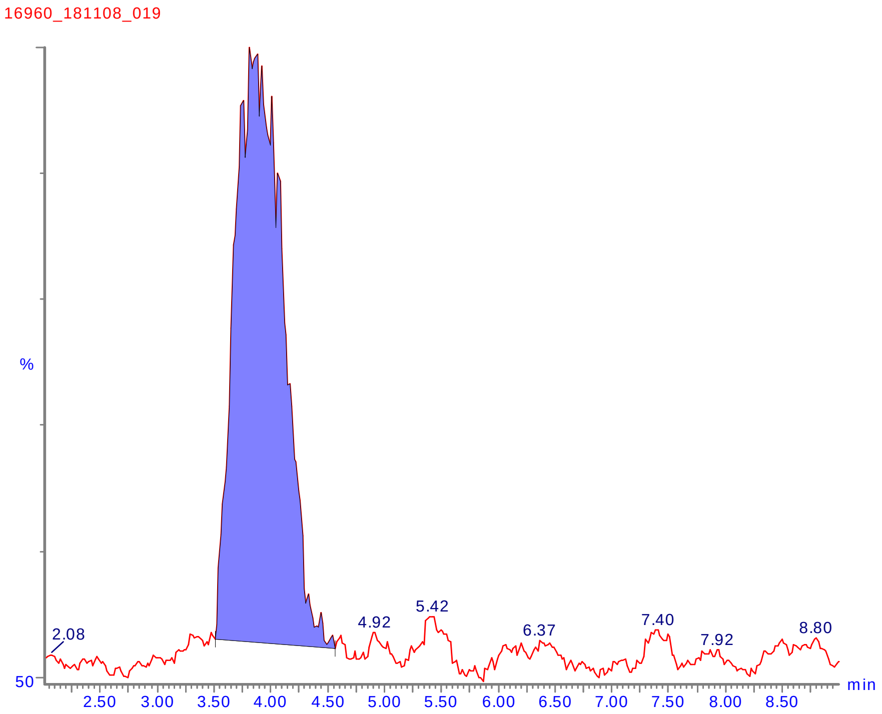


Figure 53. Mono-n-butyl phthalate spiked into foods at the target LOQ

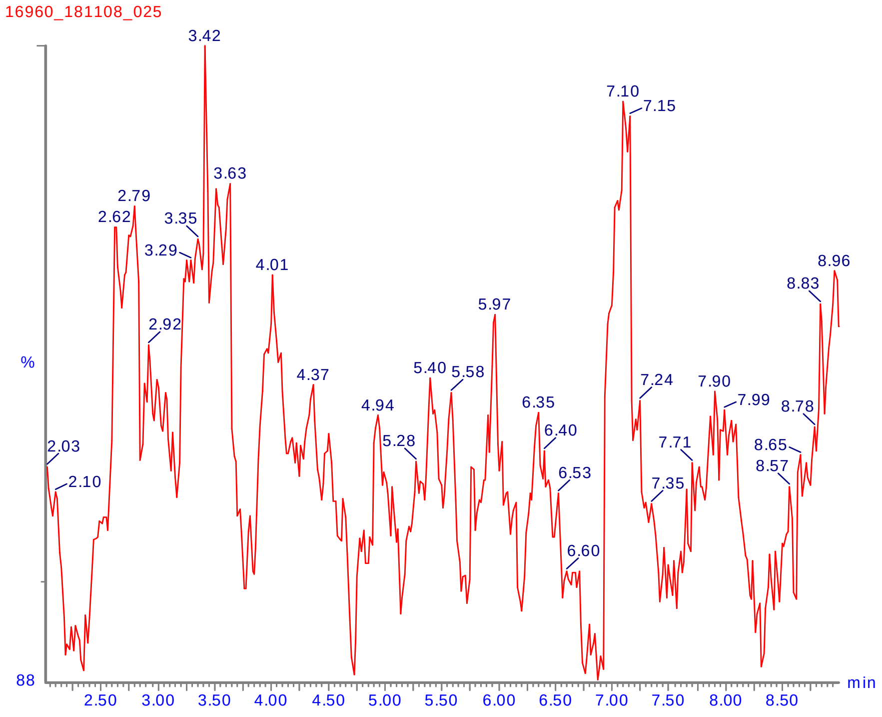
(a) Cheese blank



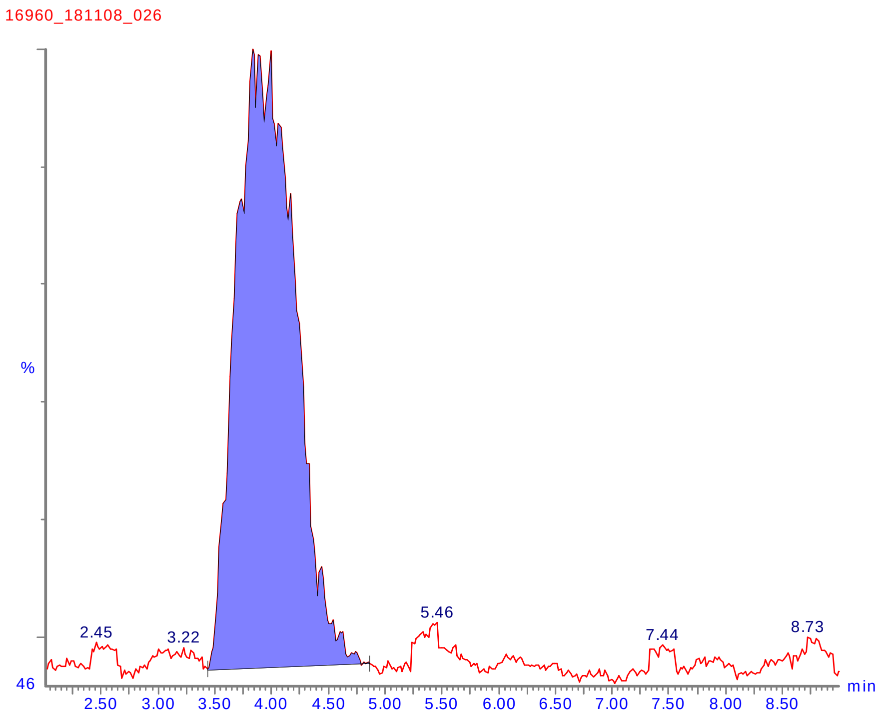
(b) Cheese + overspike at 0.05 mg/kg



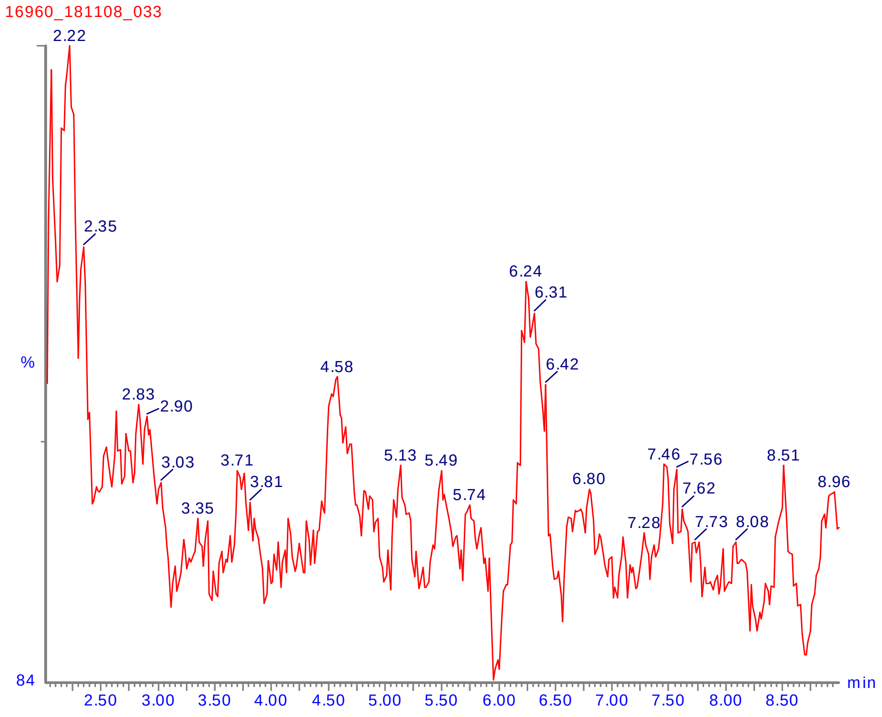
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

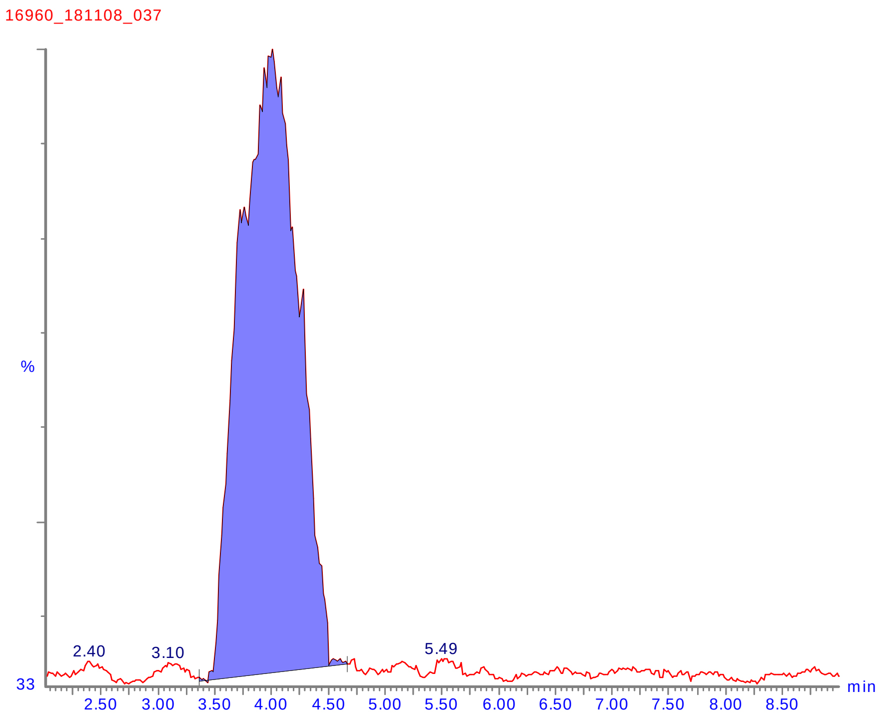
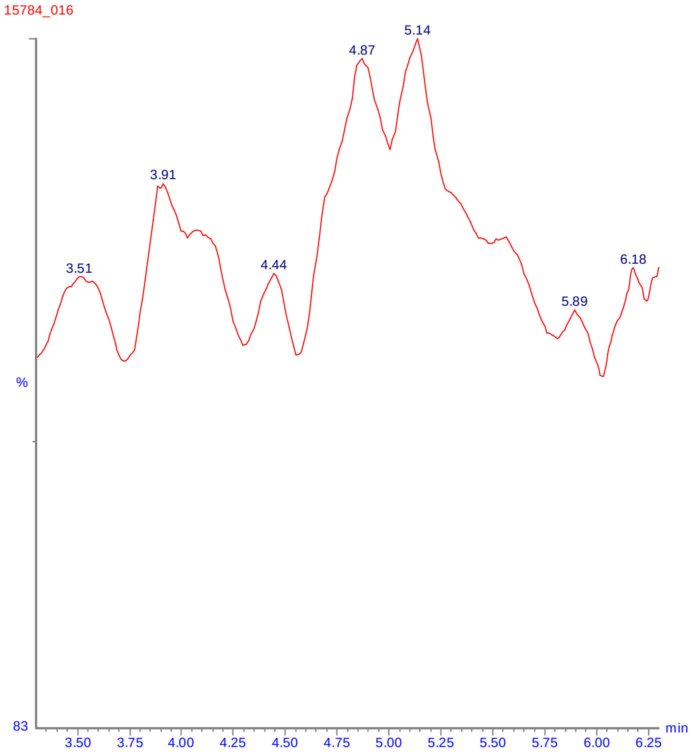
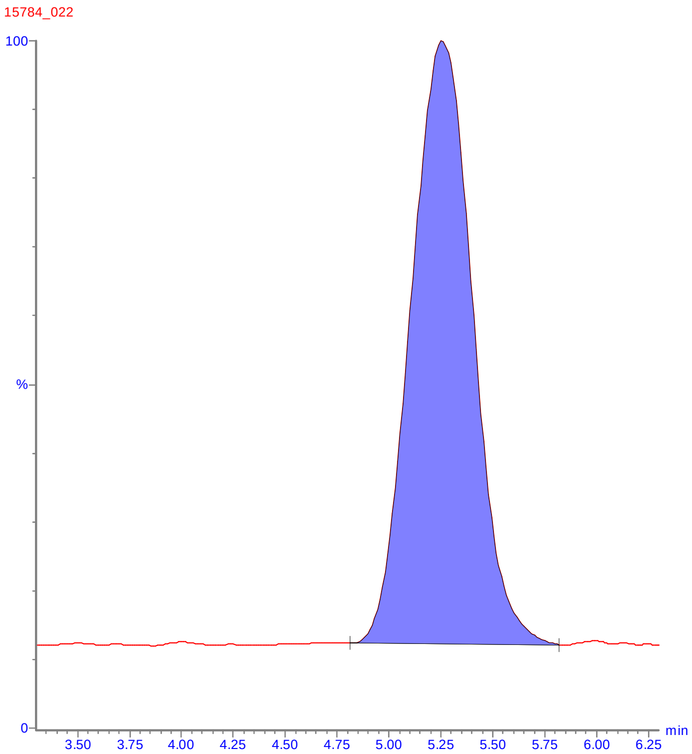


Figure 54. Monobenzyl phthalate spiked into foods at the target LOQ

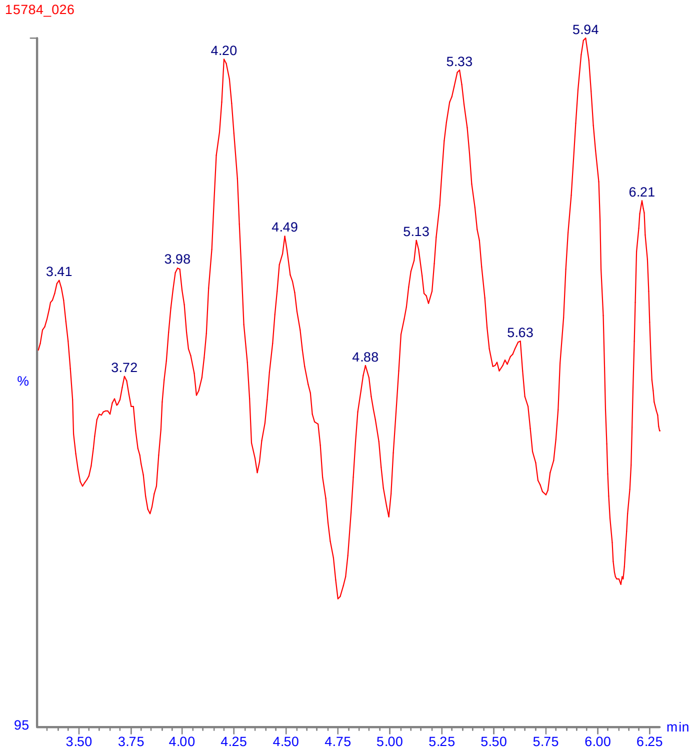
(a) Cheese blank



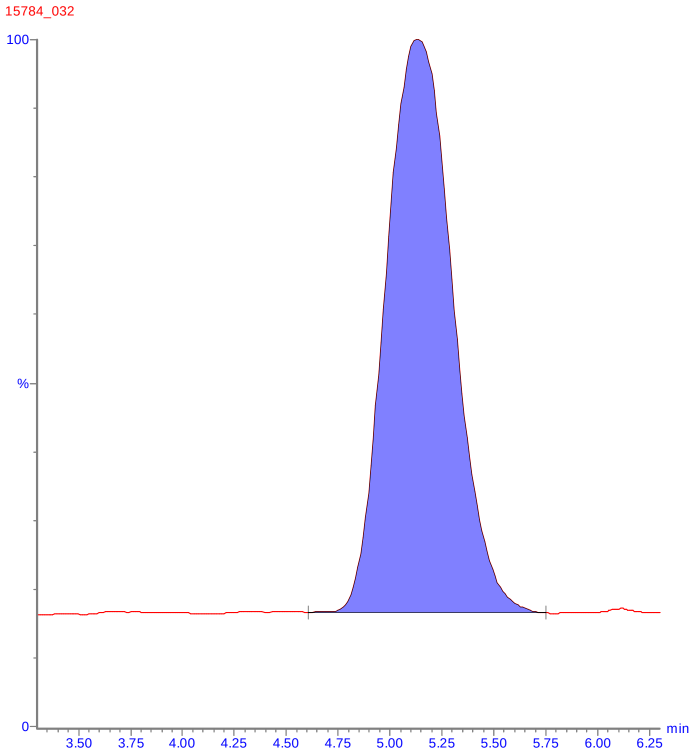
(b) Cheese + overspike at 0.05 mg/kg



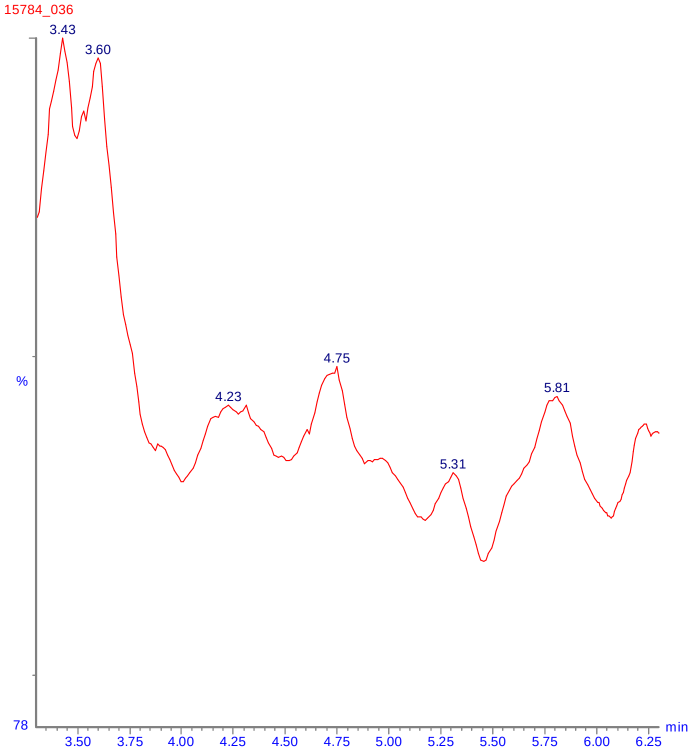
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

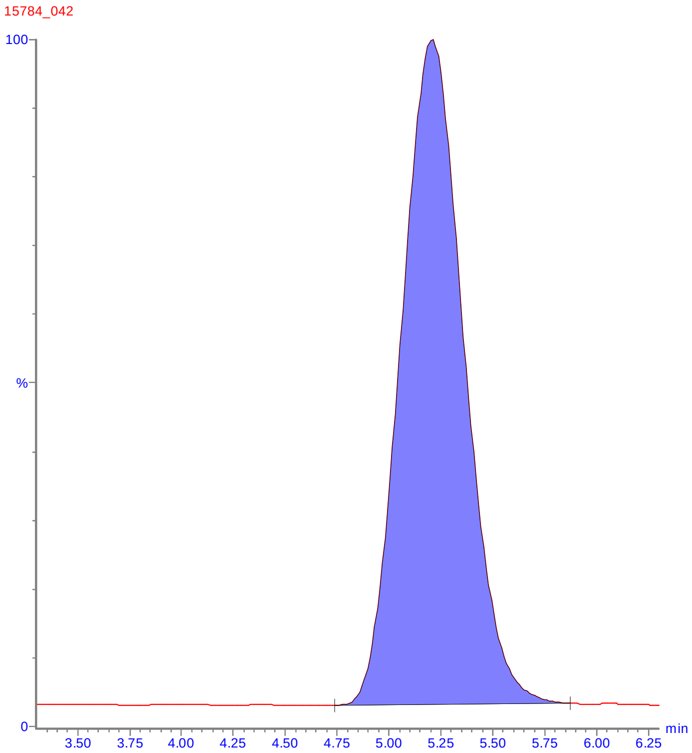
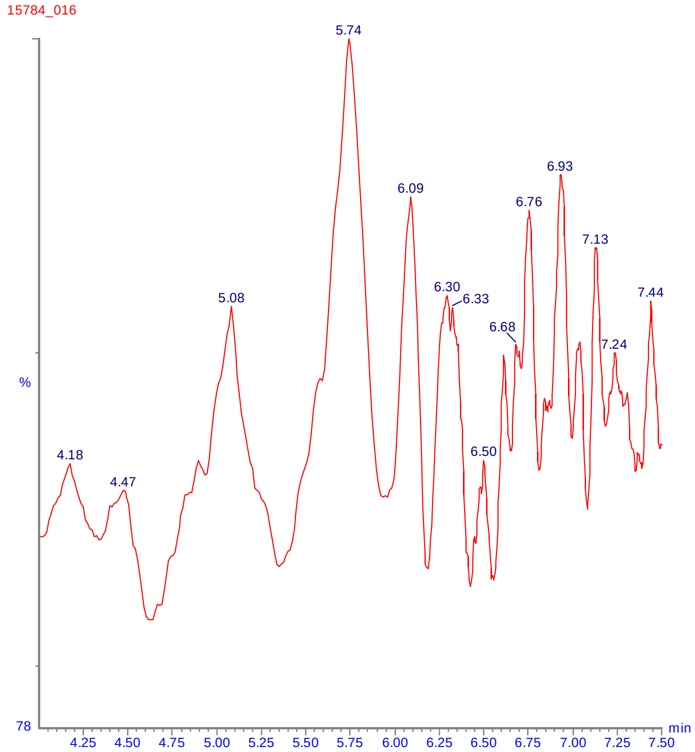
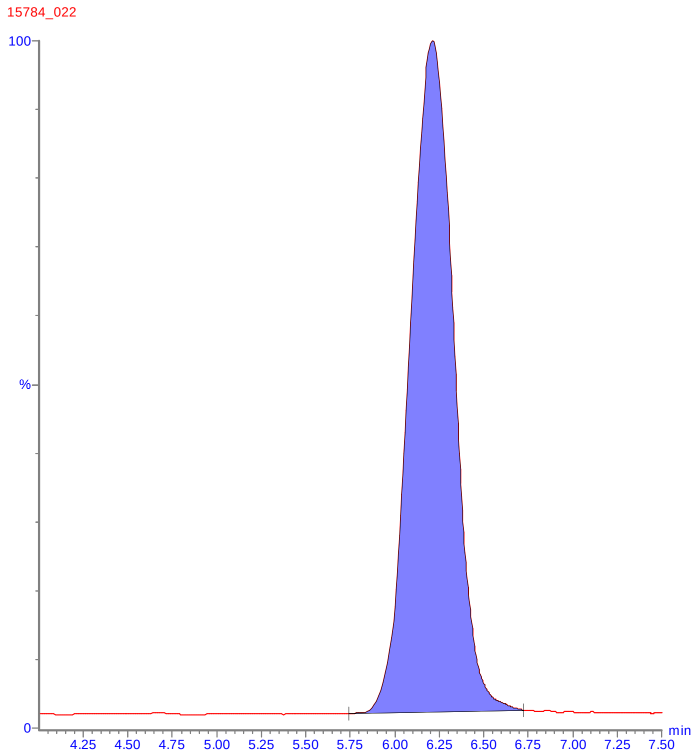


Figure 55. Monocyclohexyl phthalate spiked into foods at the target LOQ

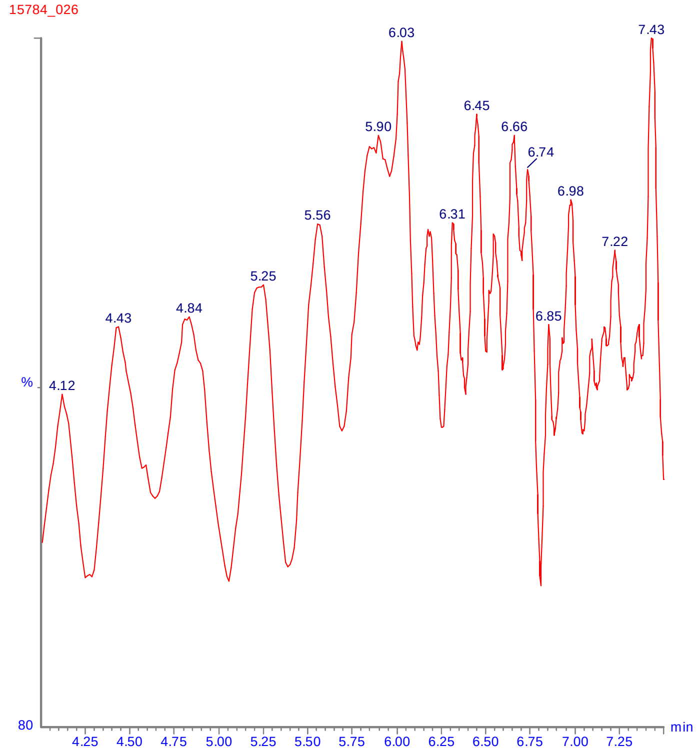
(a) Cheese blank



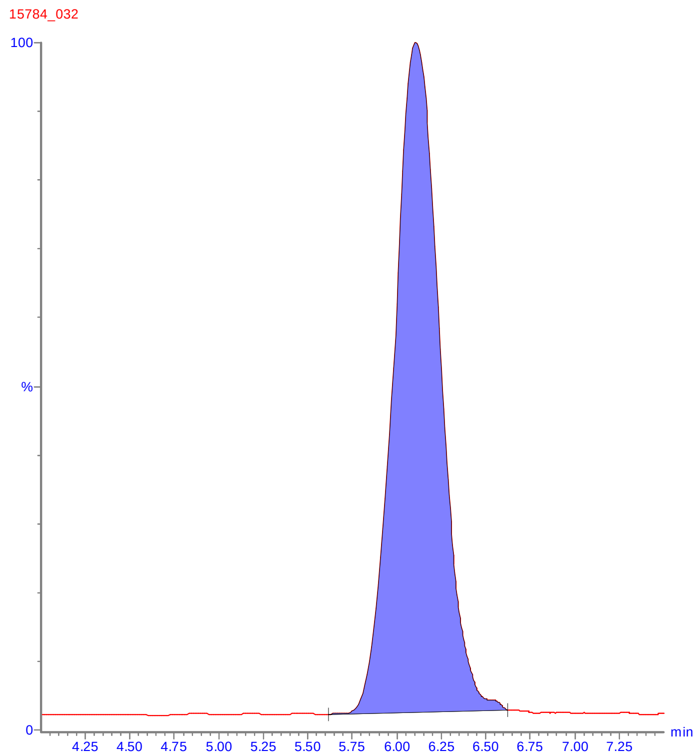
(b) Cheese + overspike at 0.05 mg/kg



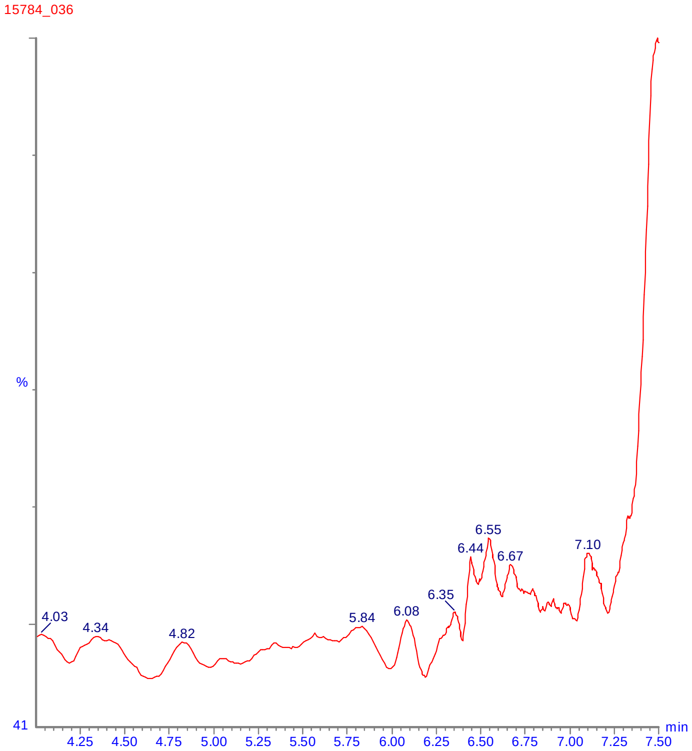
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

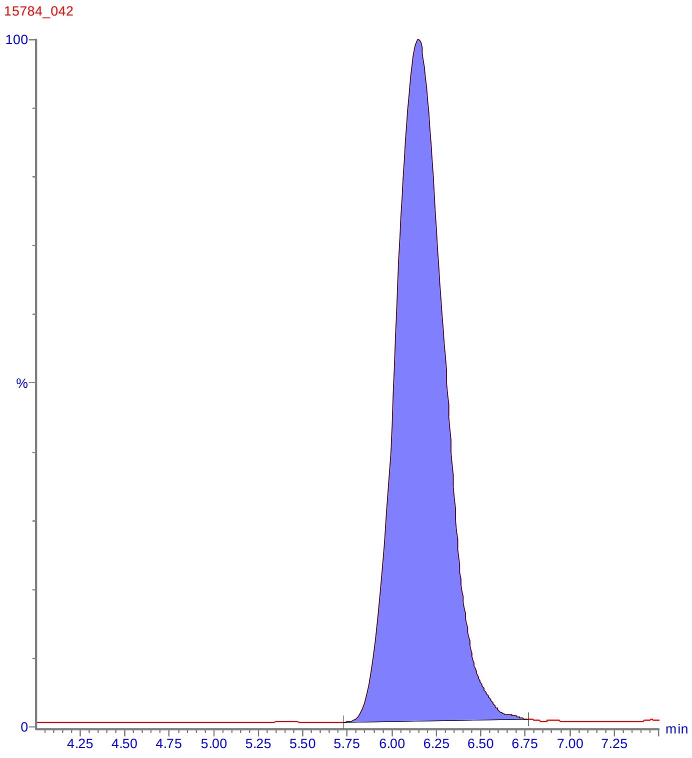
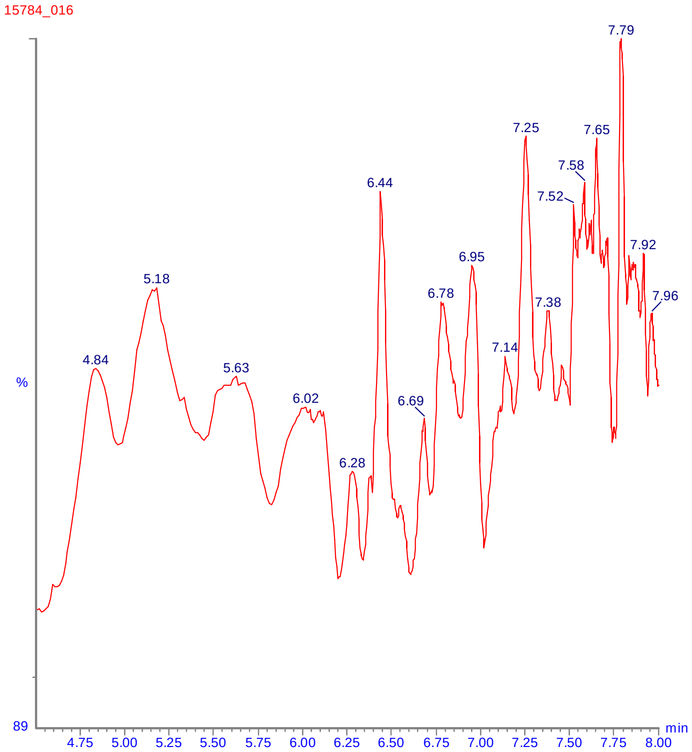
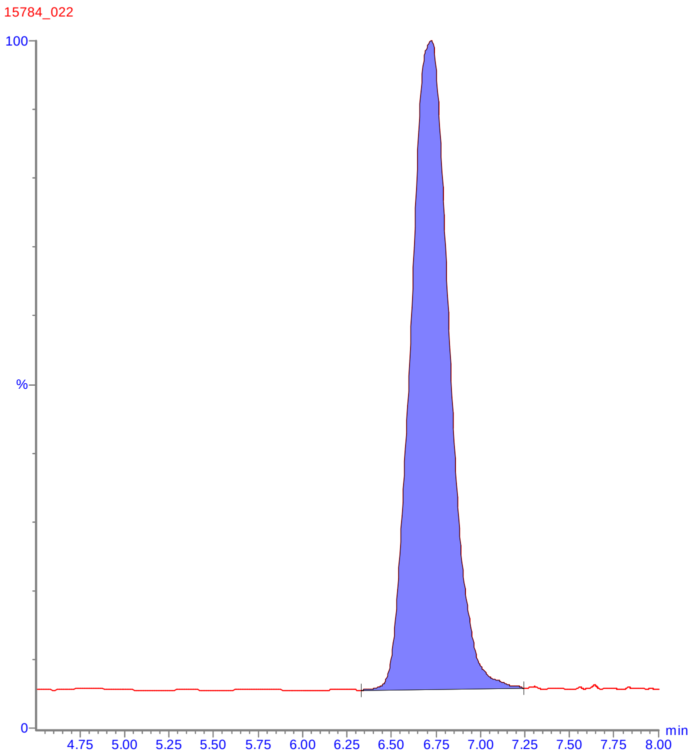


Figure 56. Mono-n-pentyl phthalate spiked into foods at the target LOQ

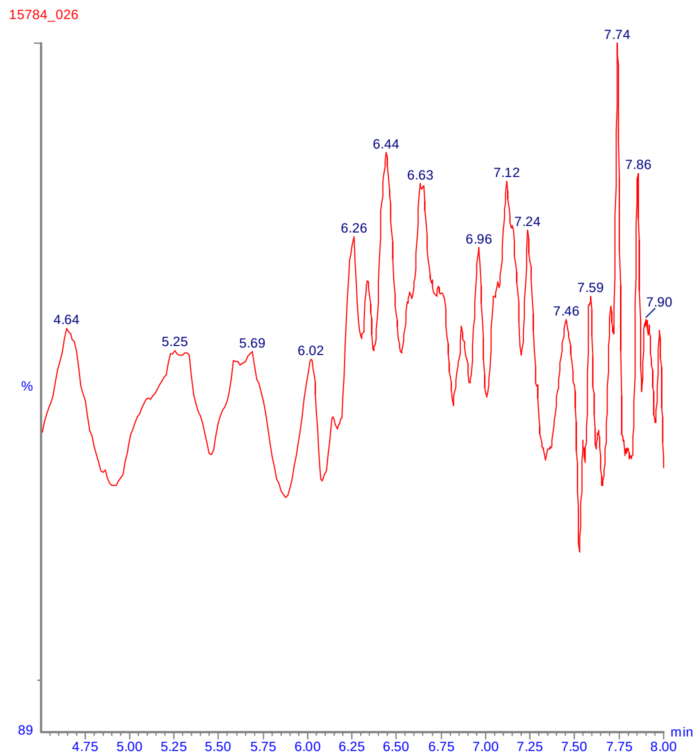
(a) Cheese blank



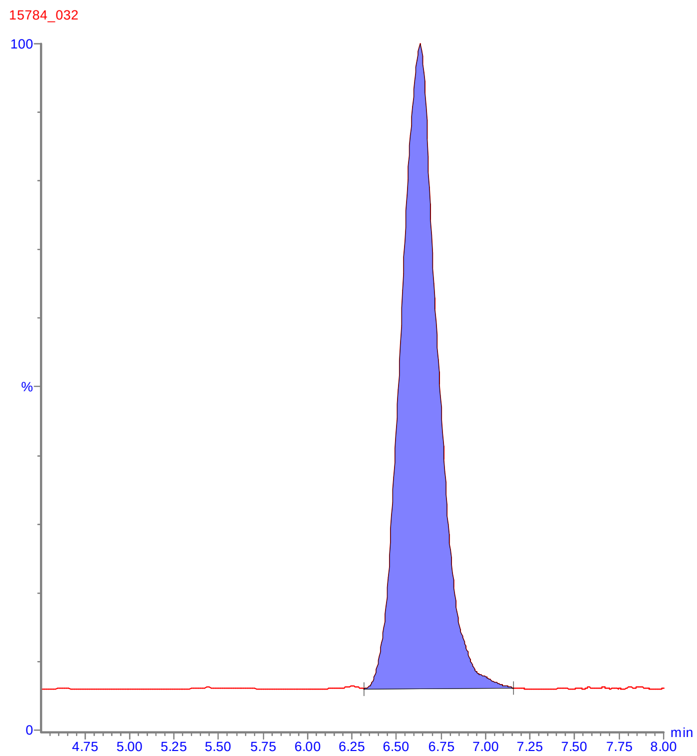
(b) Cheese + overspike at 0.05 mg/kg



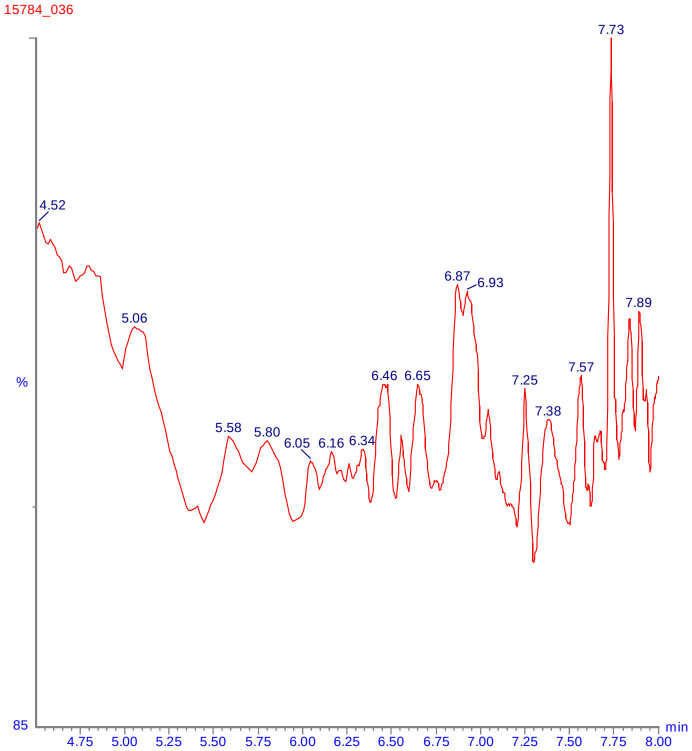
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

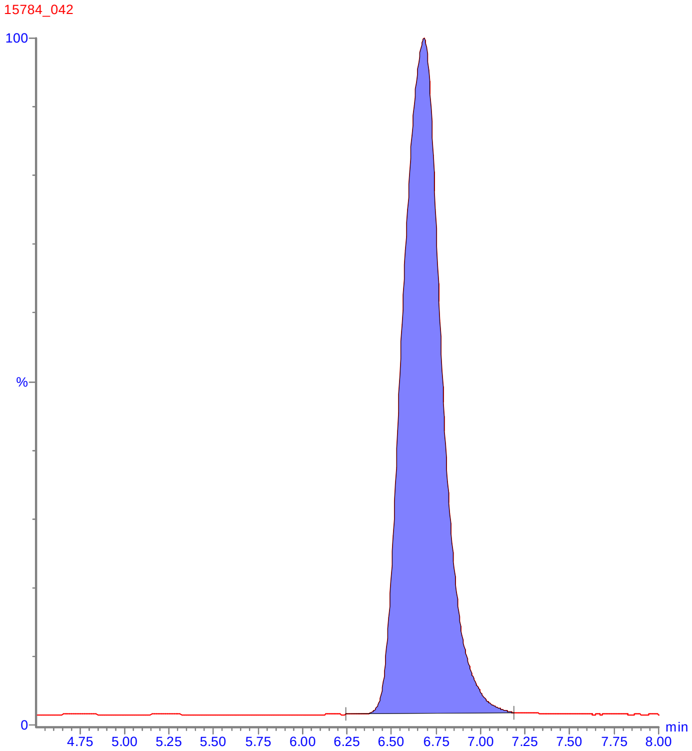
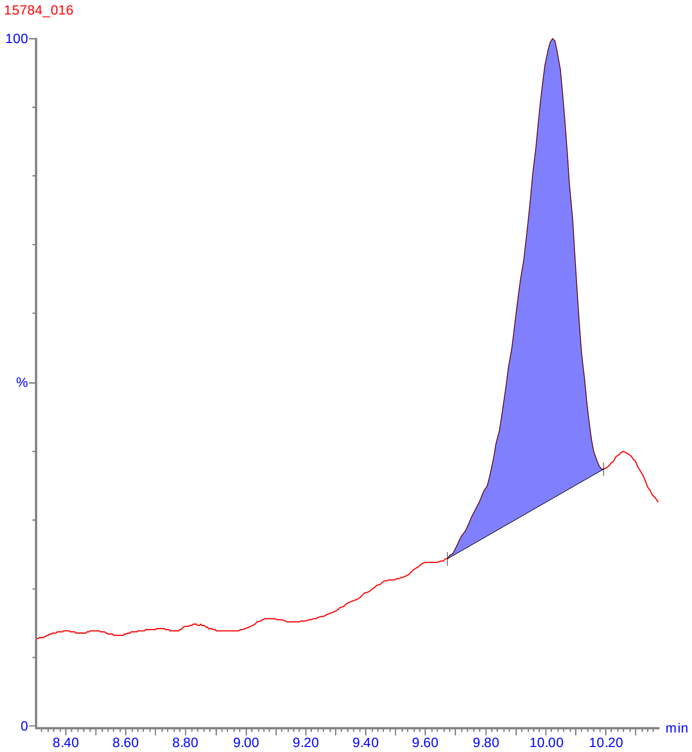
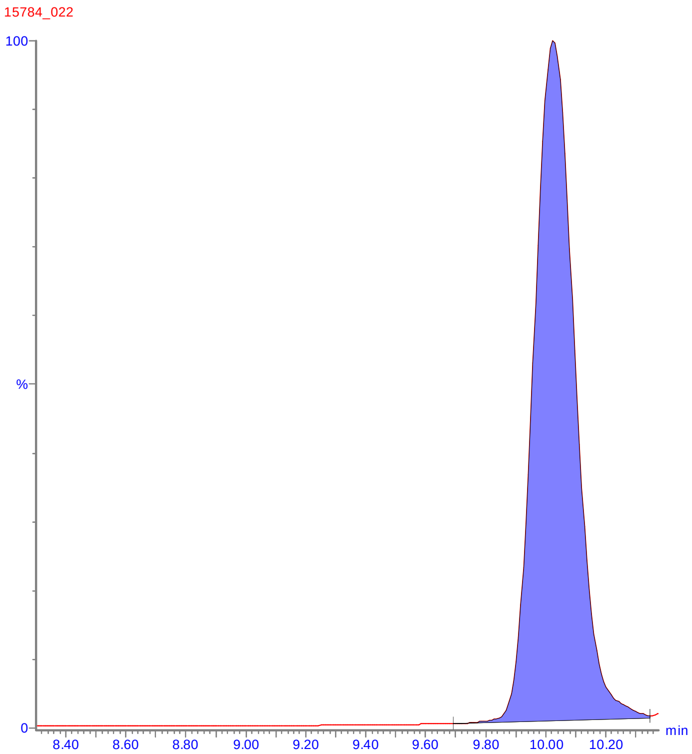


Figure 57. Mono-2-ethylhexyl phthalate spiked into foods at the target LOQ

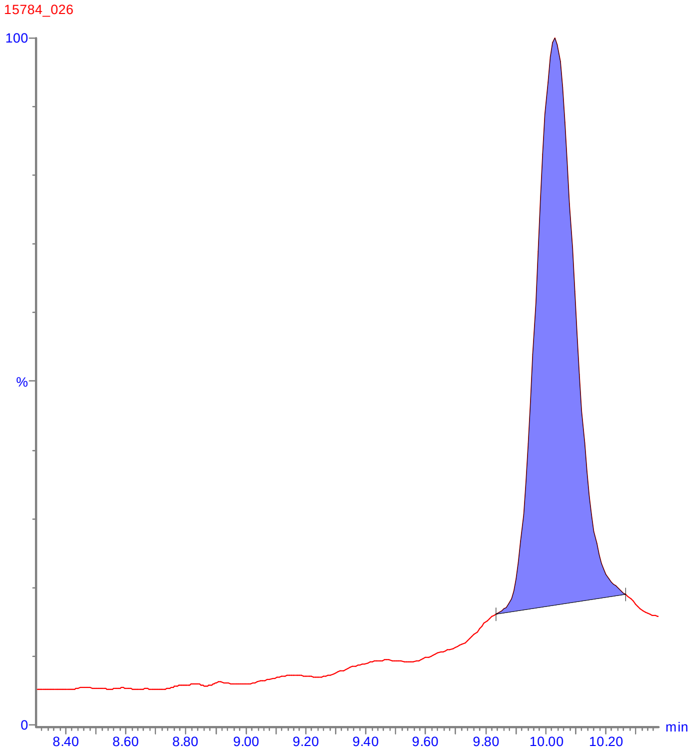
(a) Cheese blank



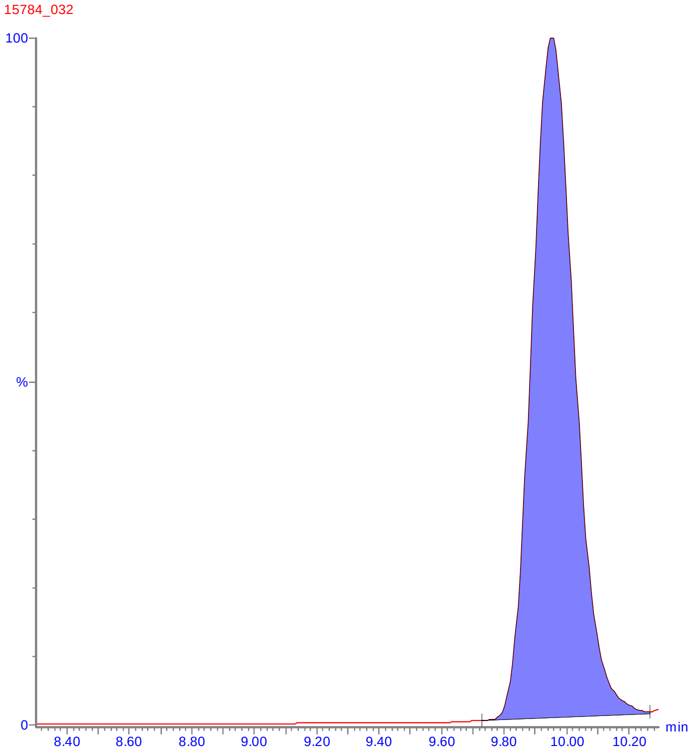
(b) Cheese + overspike at 0.05 mg/kg



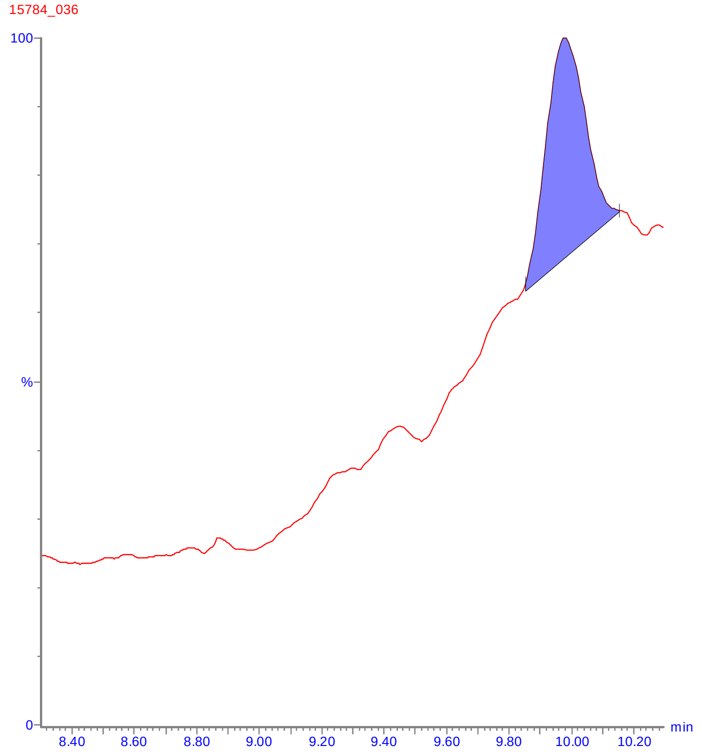
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

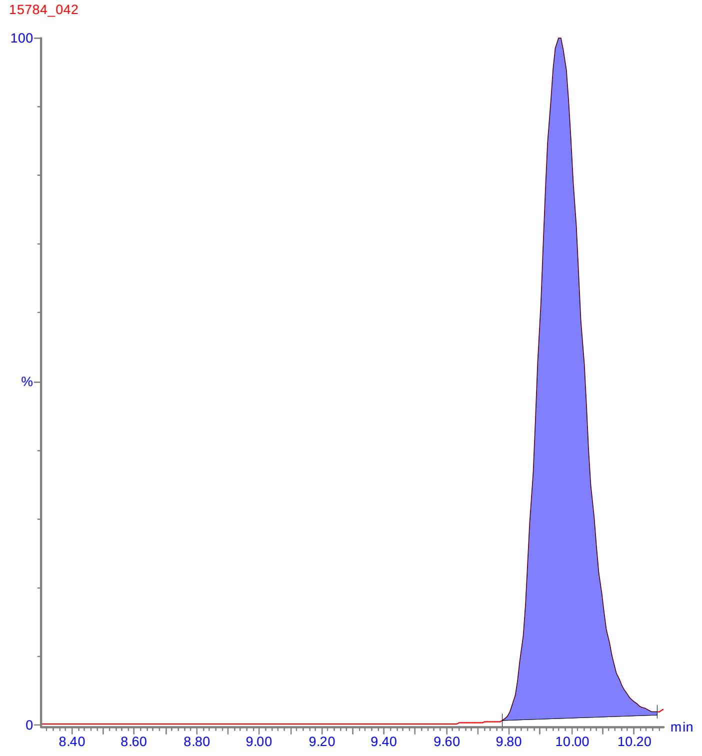
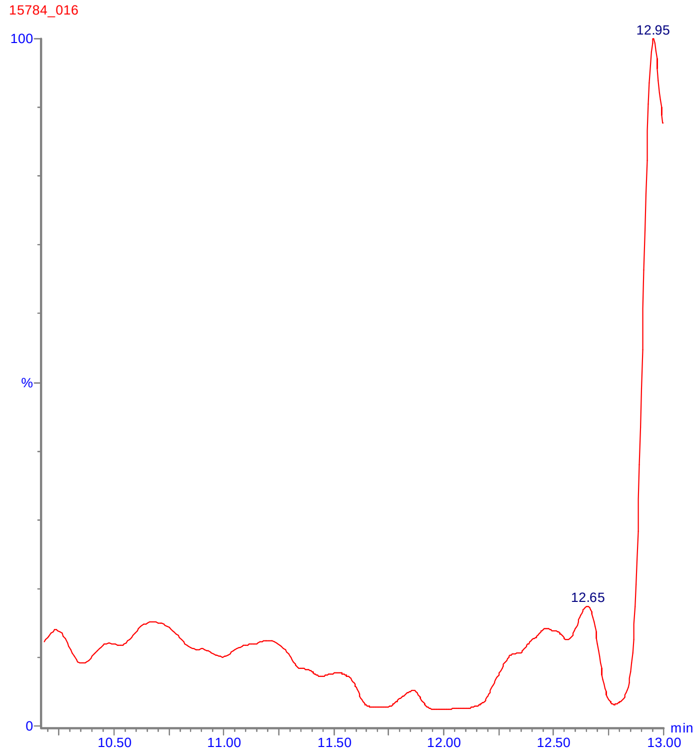
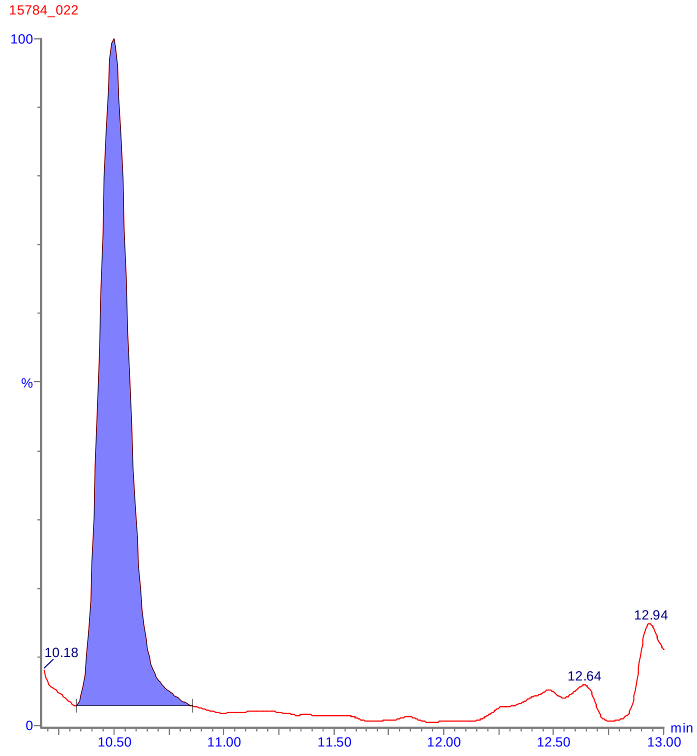


Figure 58. Mono-n-octyl phthalate spiked into foods at the target LOQ

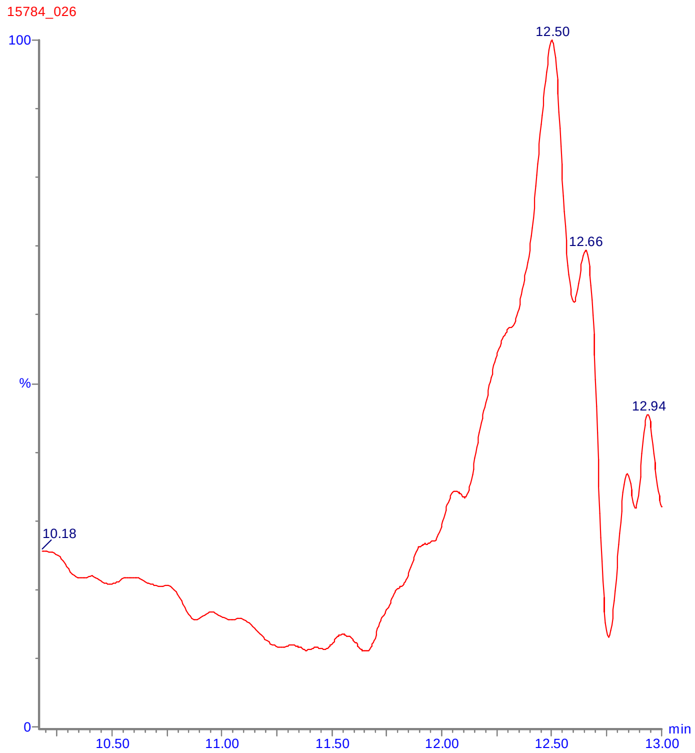
(a) Cheese blank



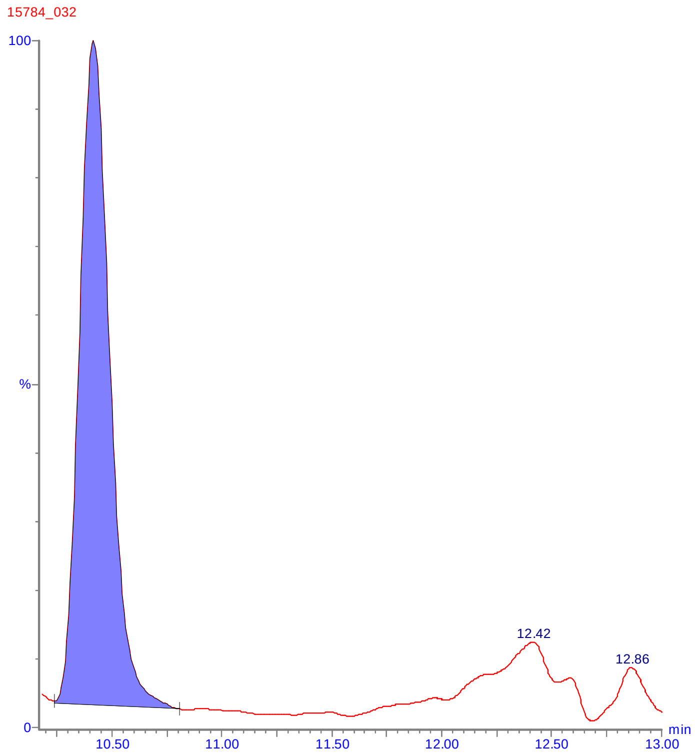
(b) Cheese + overspike at 0.05 mg/kg



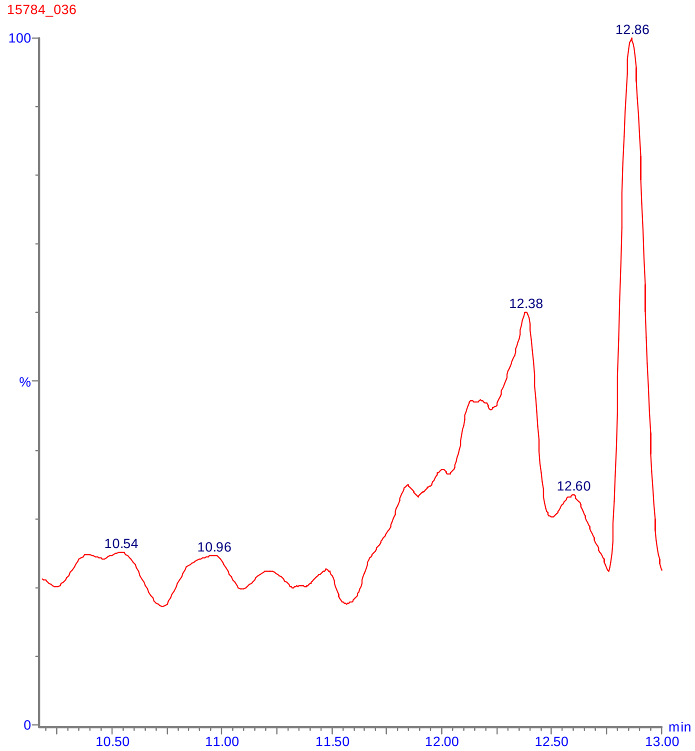
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

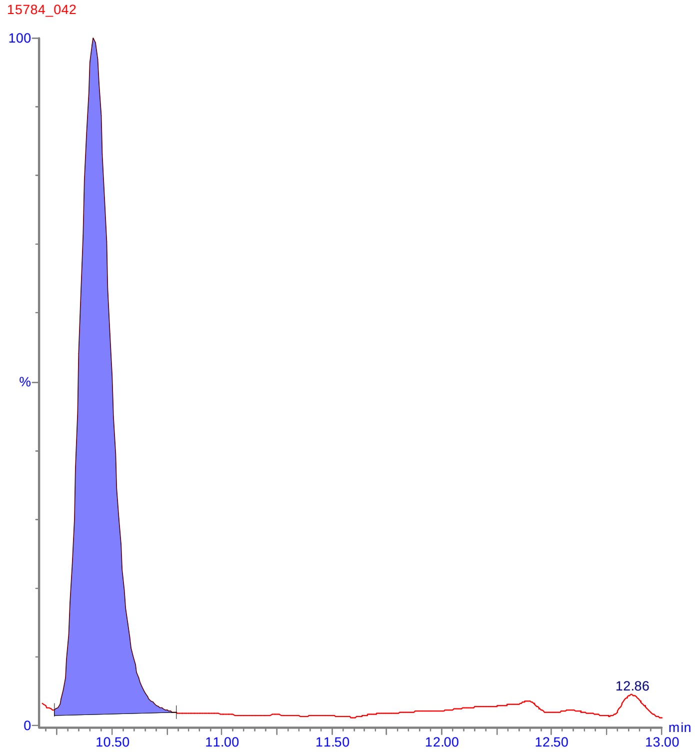
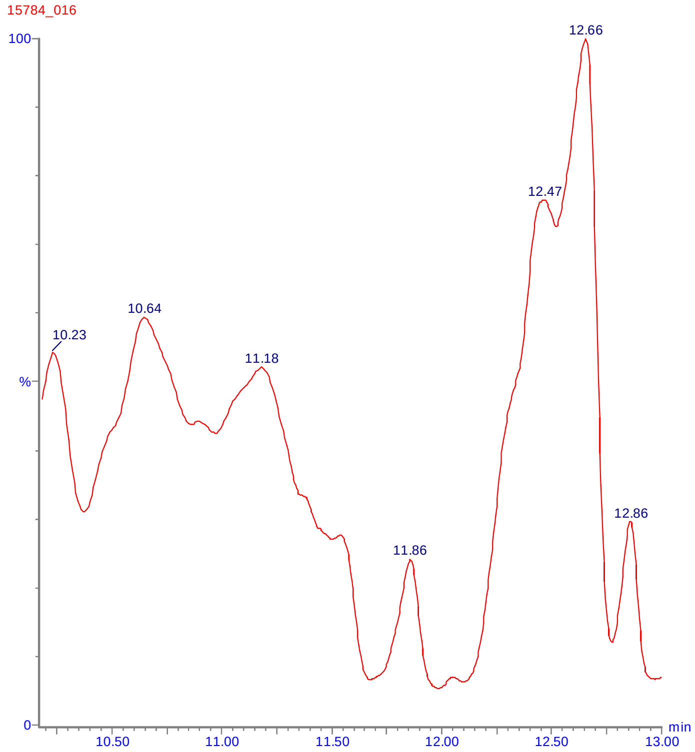
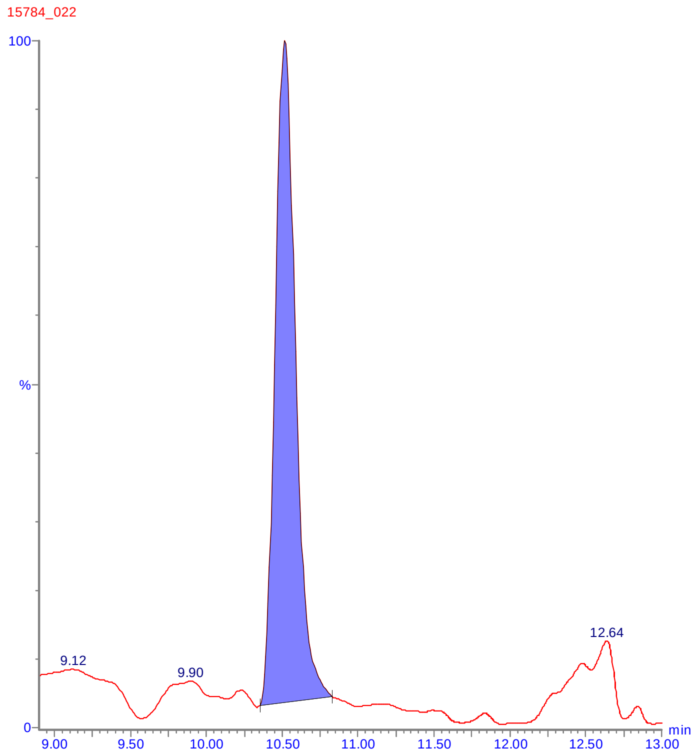


Figure 59. Monoisononyl phthalate spiked into foods at the target LOQ

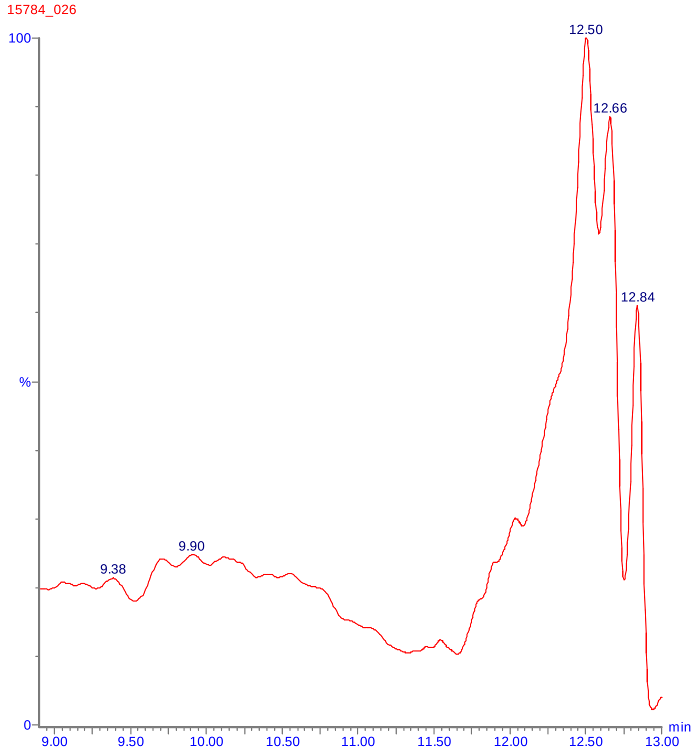
(a) Cheese blank



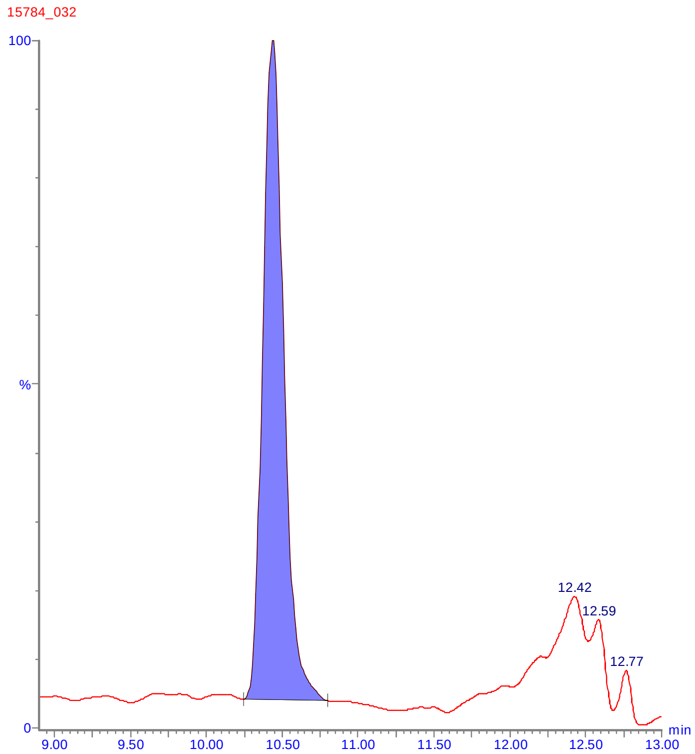
(b) Cheese + overspike at 0.05 mg/kg



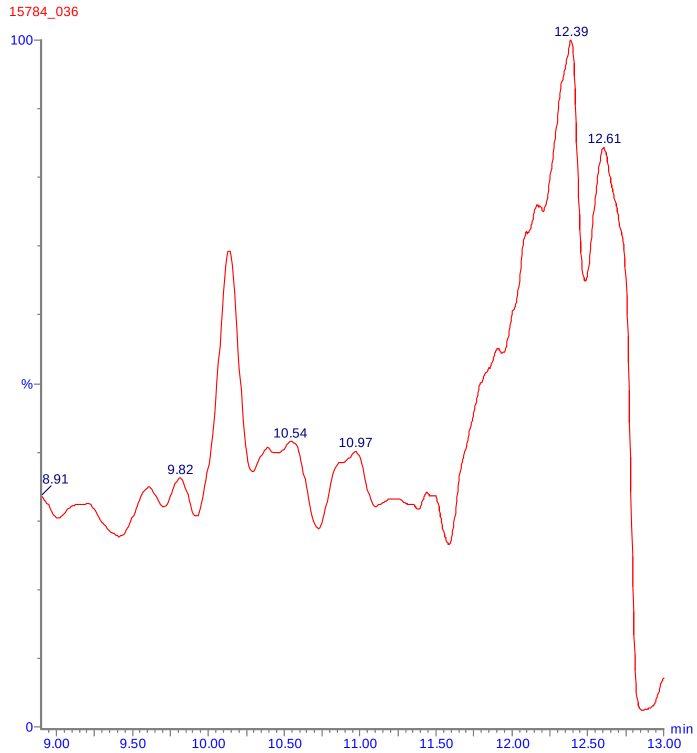
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

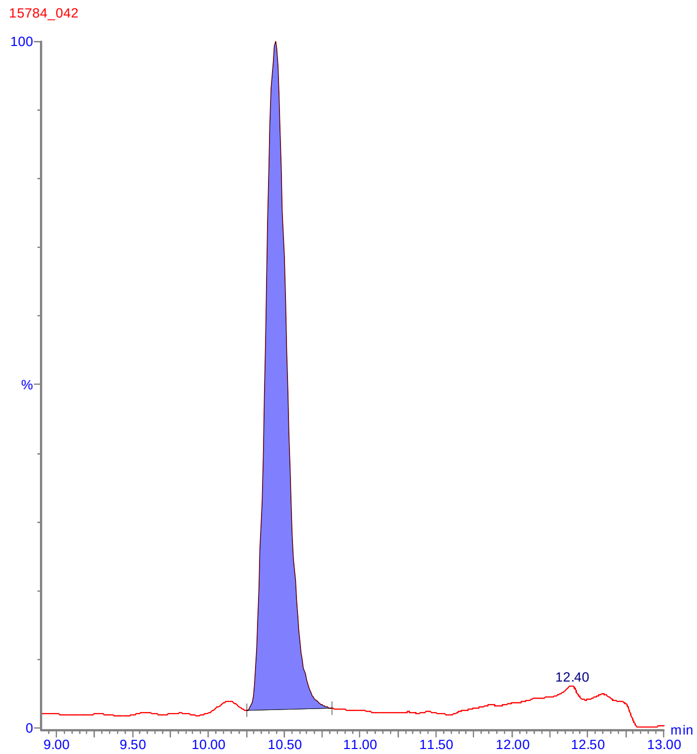


Figure 60. Calibration graph for monoisopropyl phthalate in acetonitrile

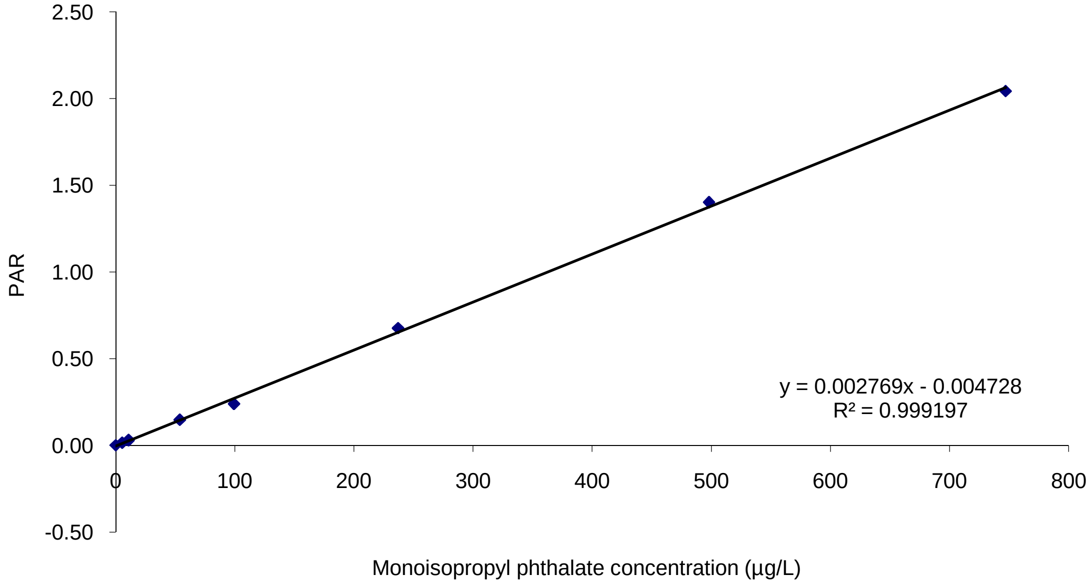


Figure 61. Calibration graph for total monoisobutyl phthalate and mono-n-butyl phthalate in acetonitrile

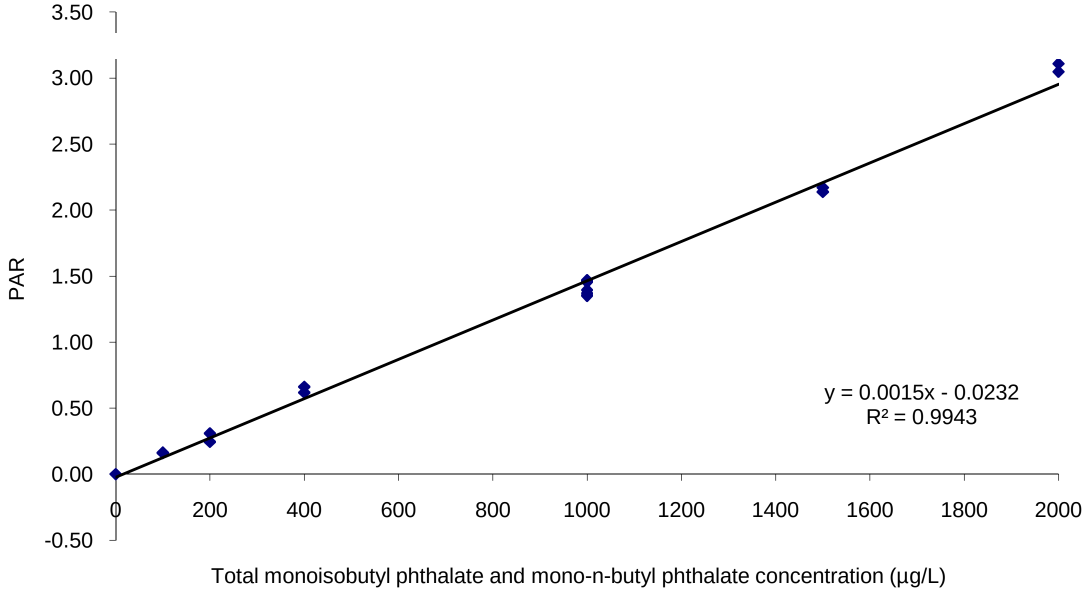


Figure 62. Calibration graph for mono-n-butyl phthalate in acetonitrile

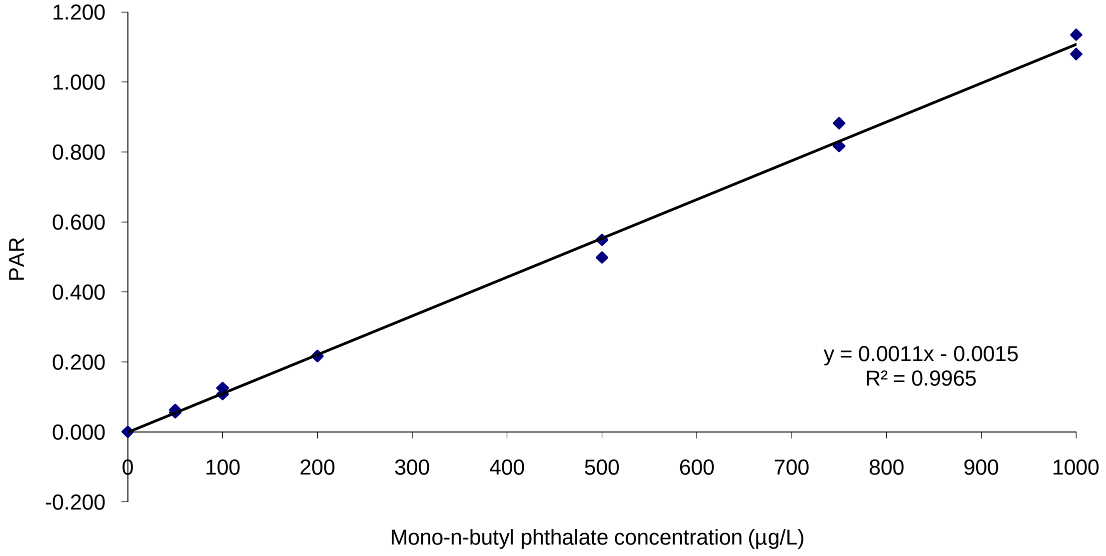


Figure 63. Calibration graph for monobenzyl phthalate in acetonitrile

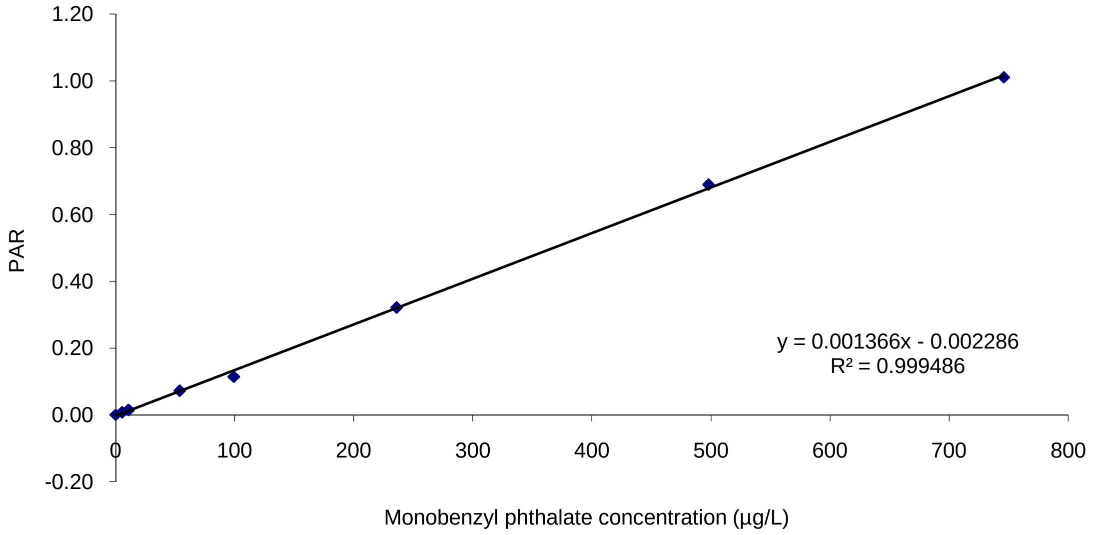


Figure 64. Calibration graph for monocyclohexyl phthalate in acetonitrile

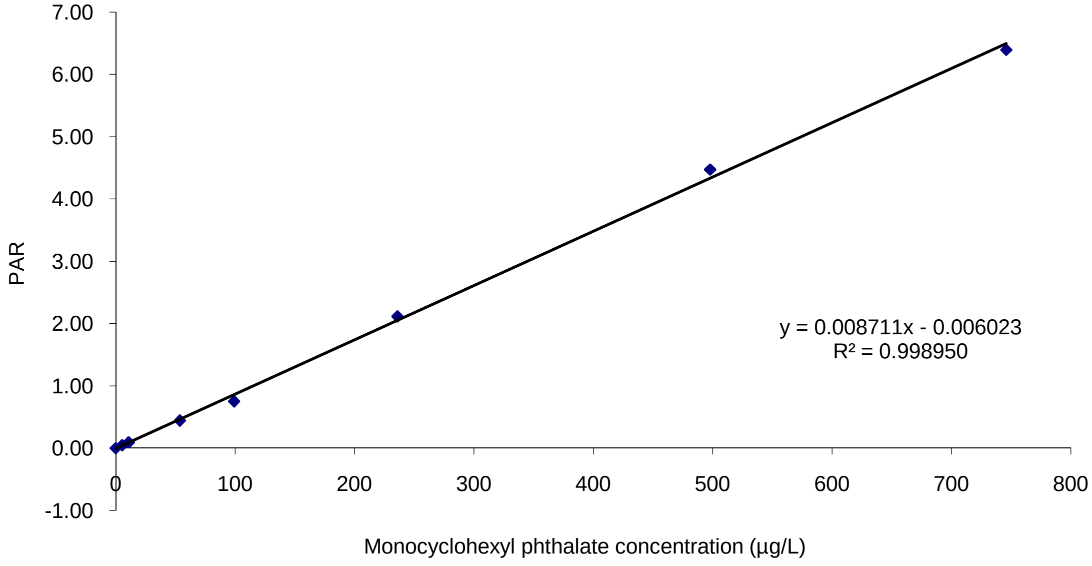


Figure 65. Calibration graph for mono-n-pentyl phthalate in acetonitrile

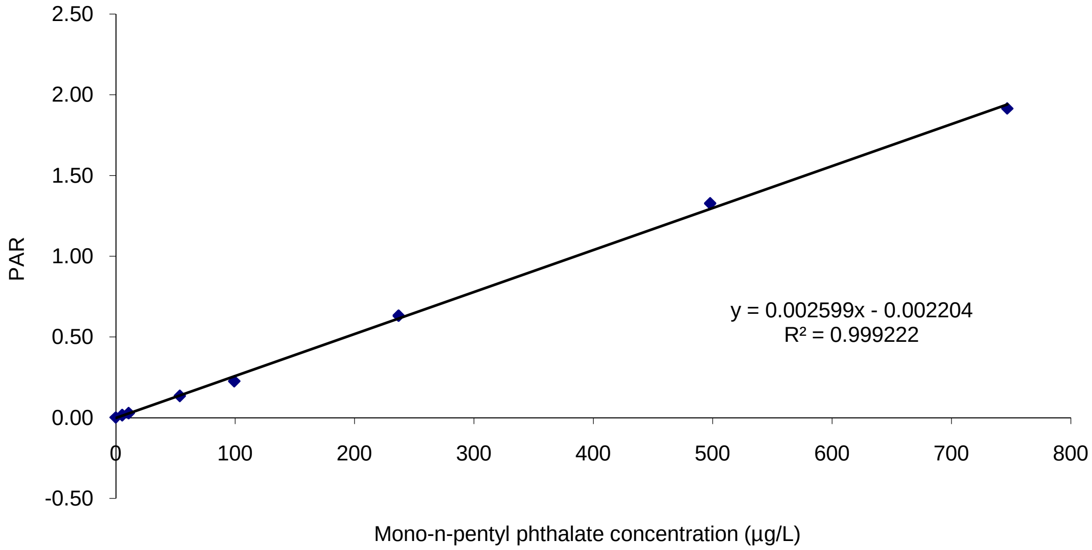


Figure 66. Calibration graph for mono-(2-ethylhexyl) phthalate in acetonitrile

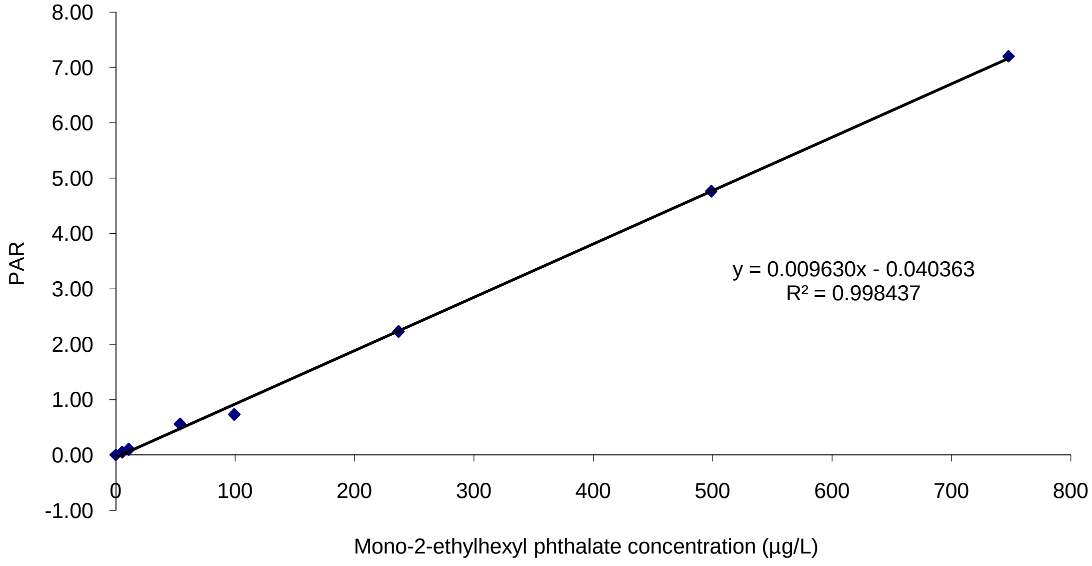


Figure 67. Calibration graph for mono-n-octyl phthalate in acetonitrile

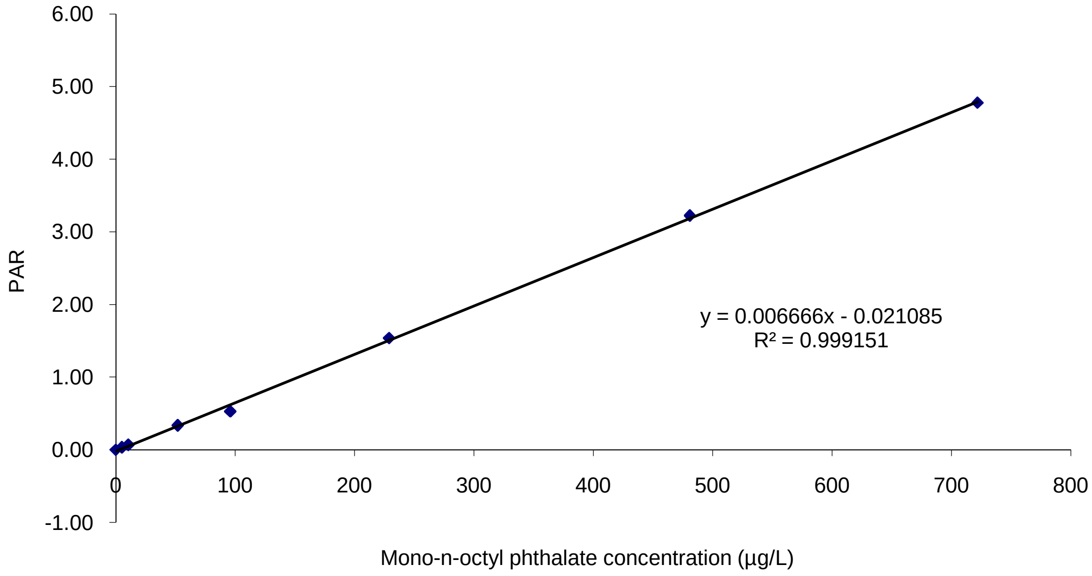


Figure 68. Calibration graph for monoisononyl phthalate in acetonitrile

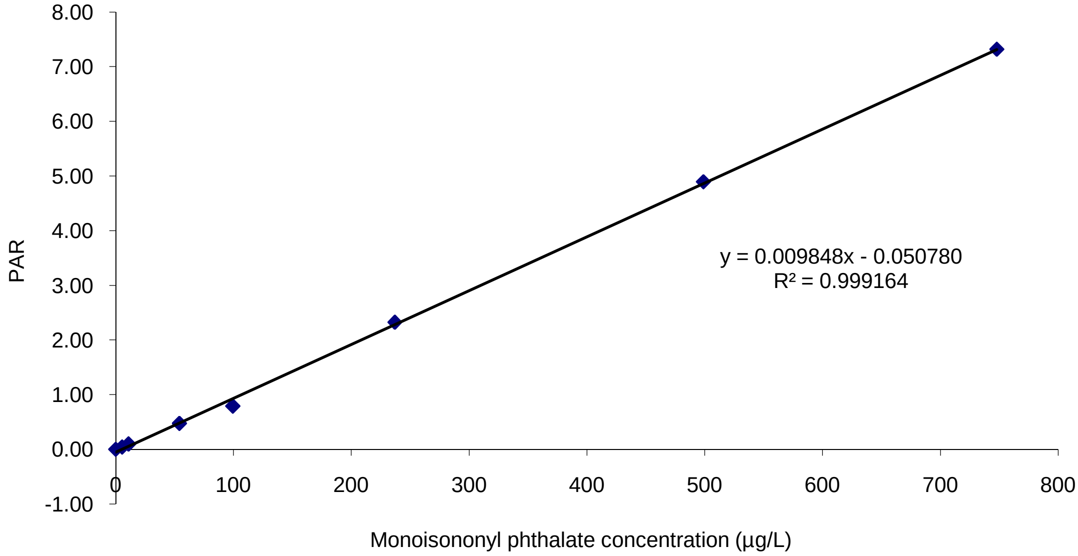
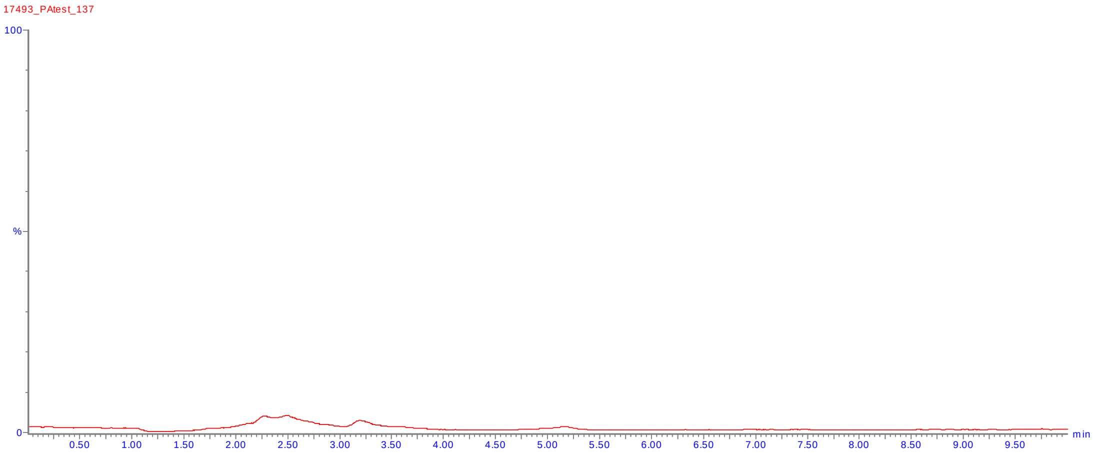
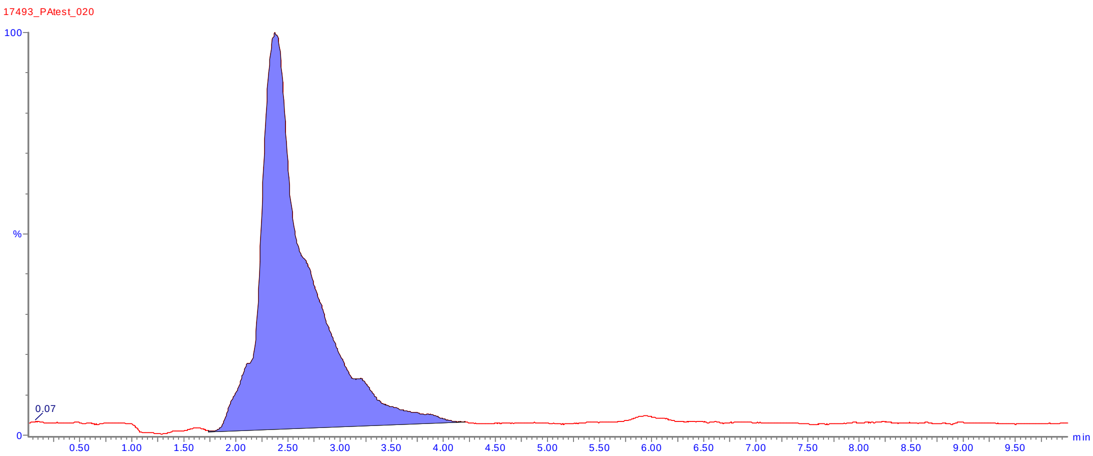


Figure 69. Phthalic acid spiked into foods at the target LOQ

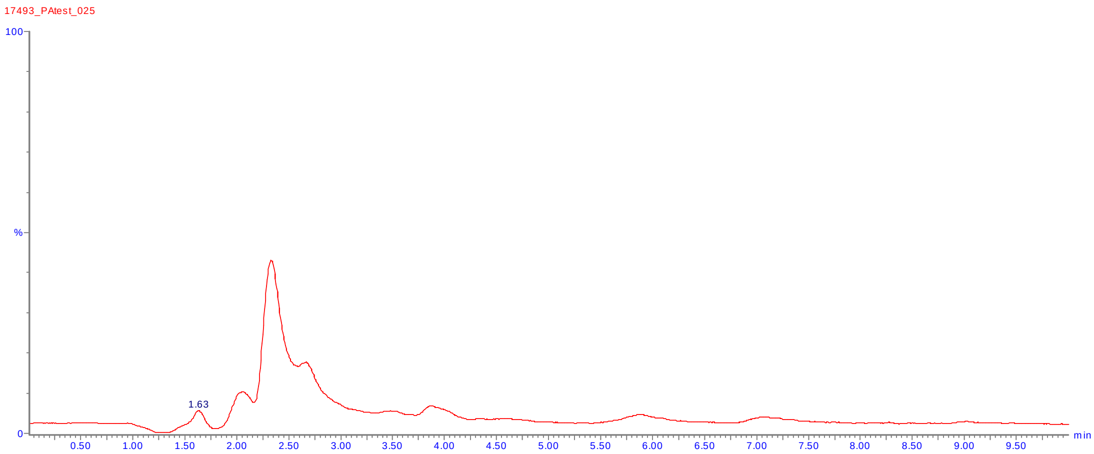
(a) Cheese blank



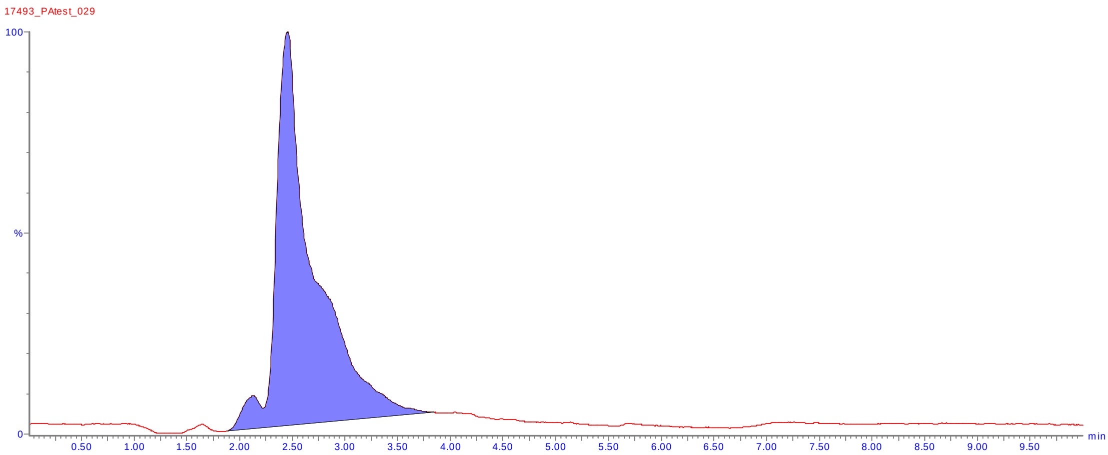
(b) Cheese + overspike at 0.05 mg/kg



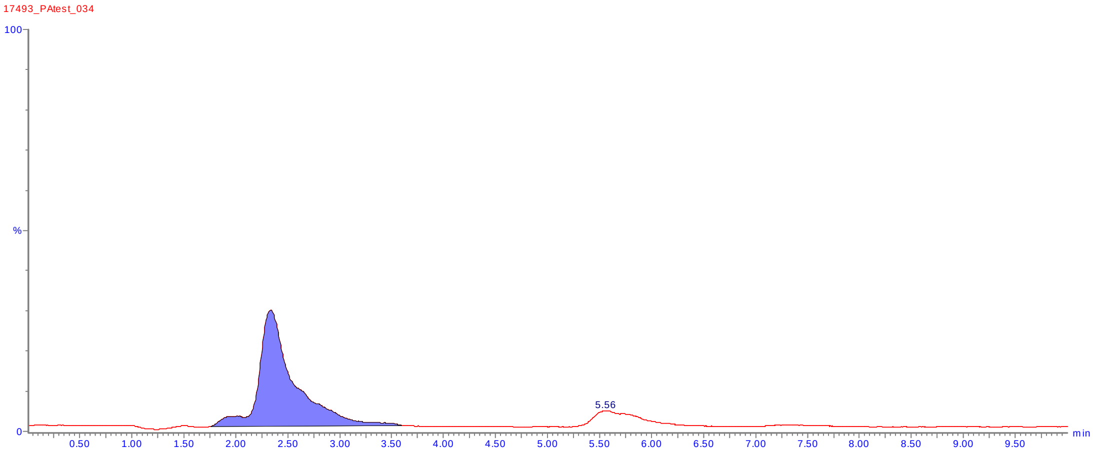
(c) Pork mince blank



(d) Pork mince + overspike at 0.05 mg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 0.05 mg/kg

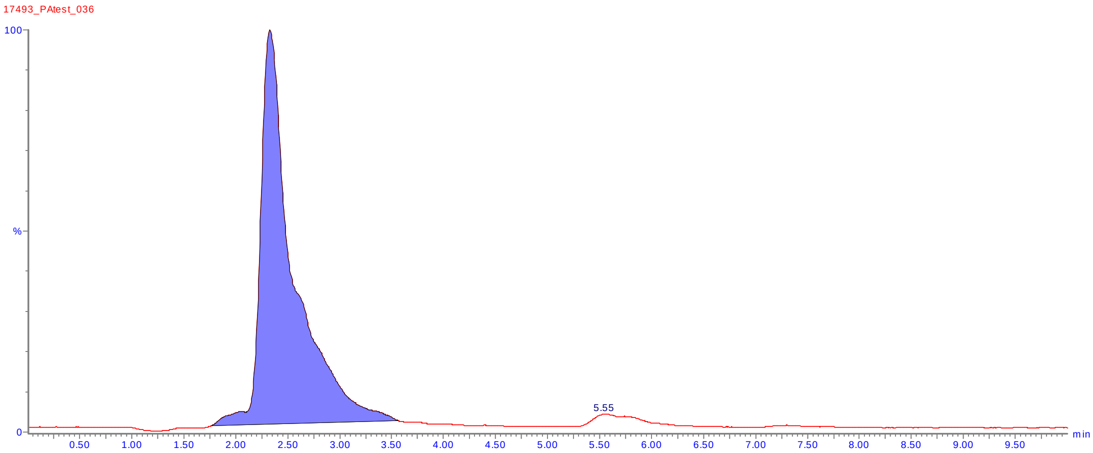


Figure 70. Calibration graph for phthalic acid in acetonitrile

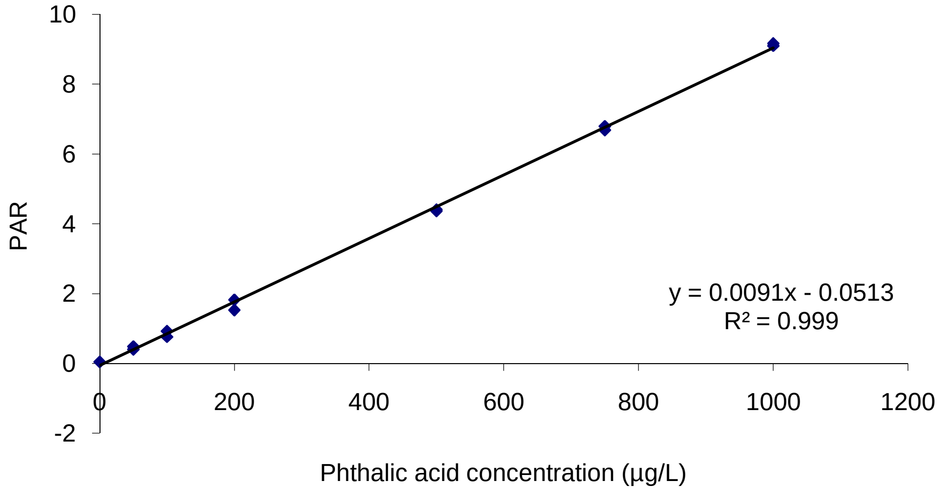
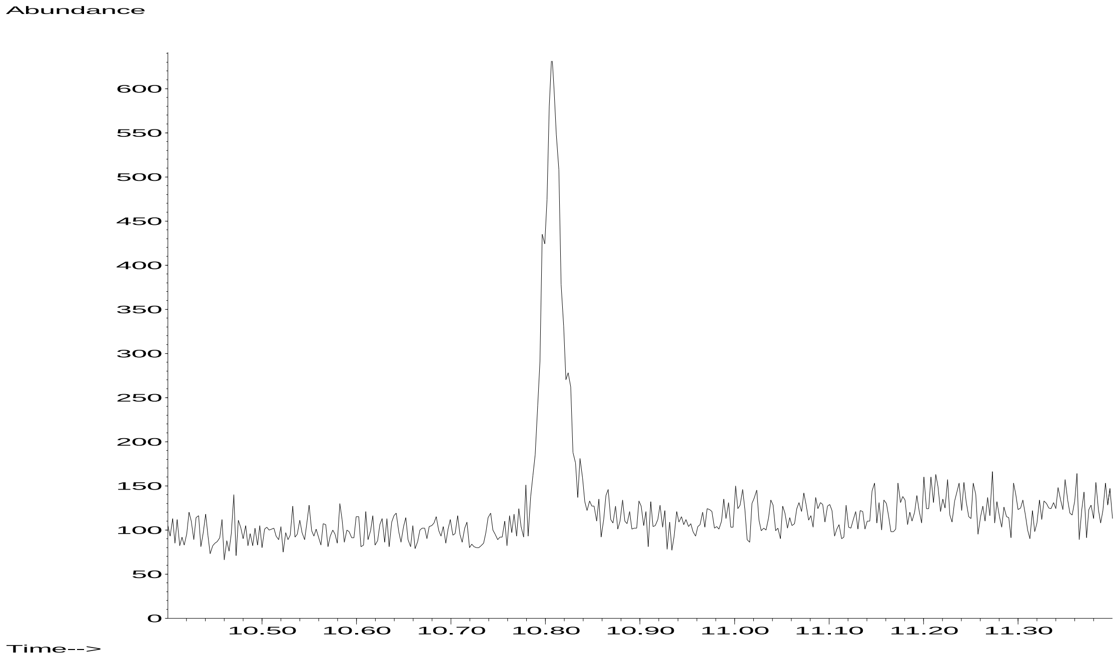
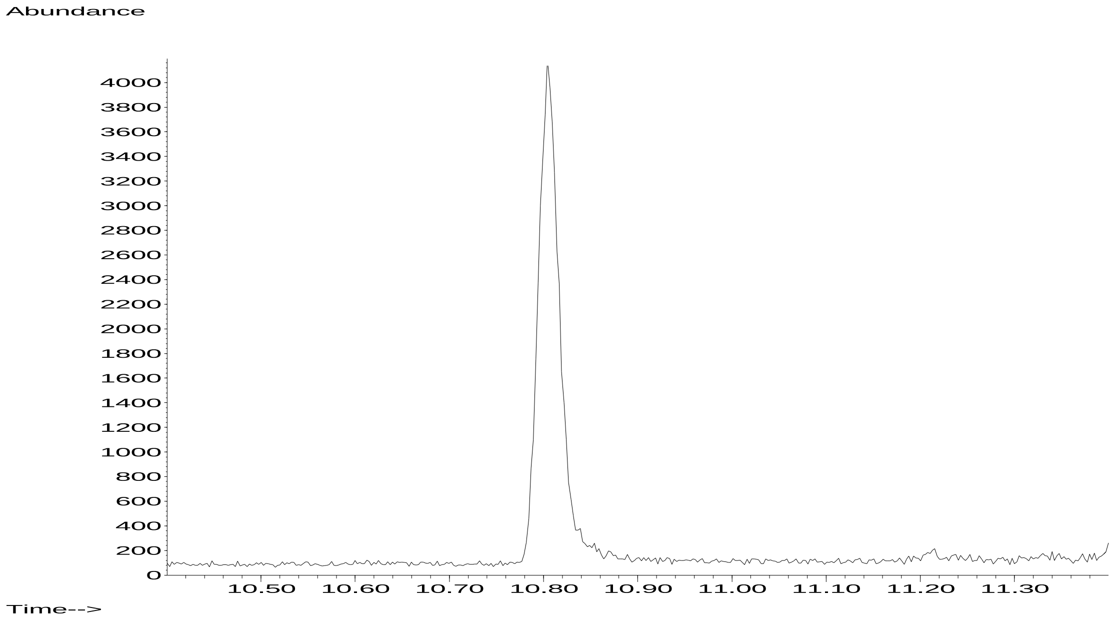


Figure 71. GC-MS selected ion chromatogram for dimethyl phthalate

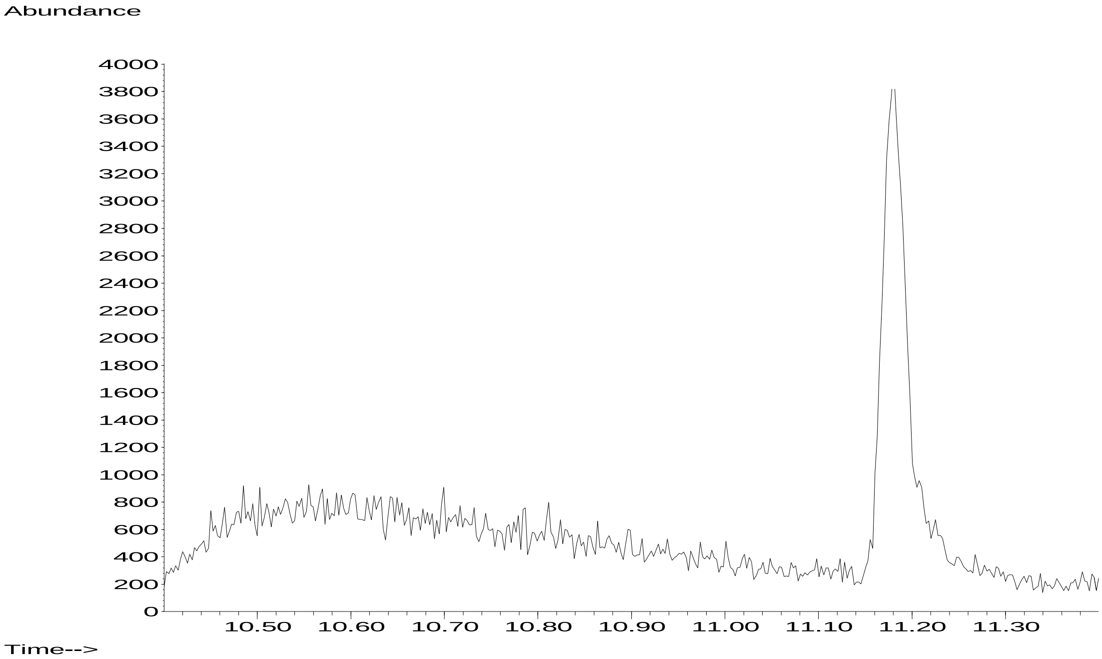
(a) Cheese blank



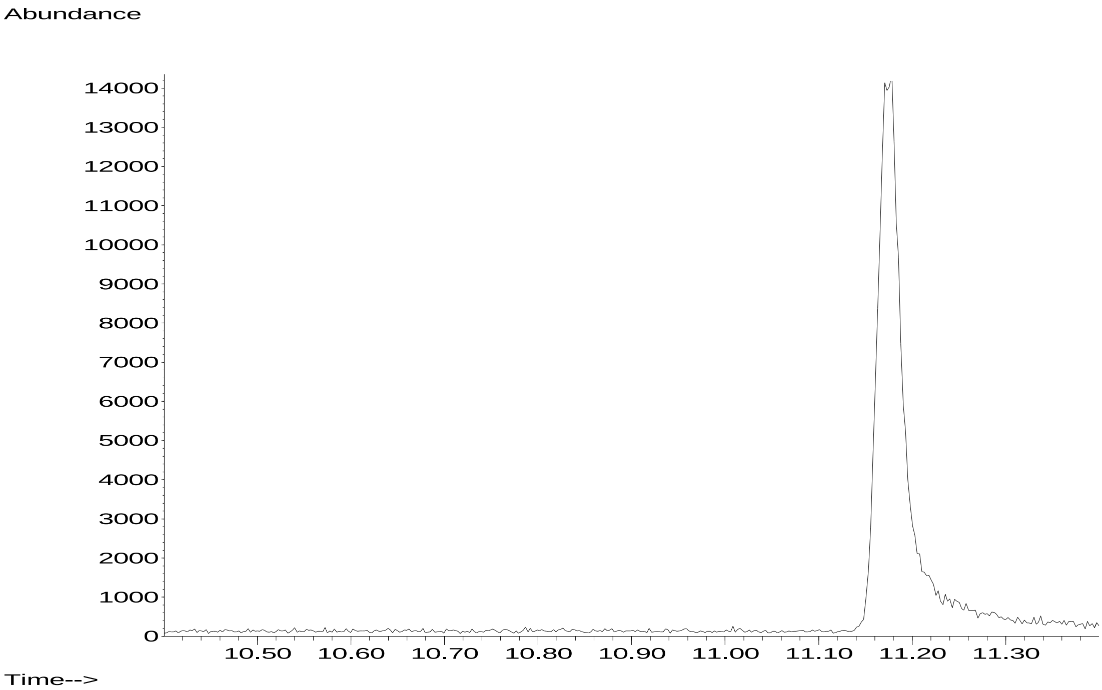
(b) Cheese + overspike at 420 µg/kg



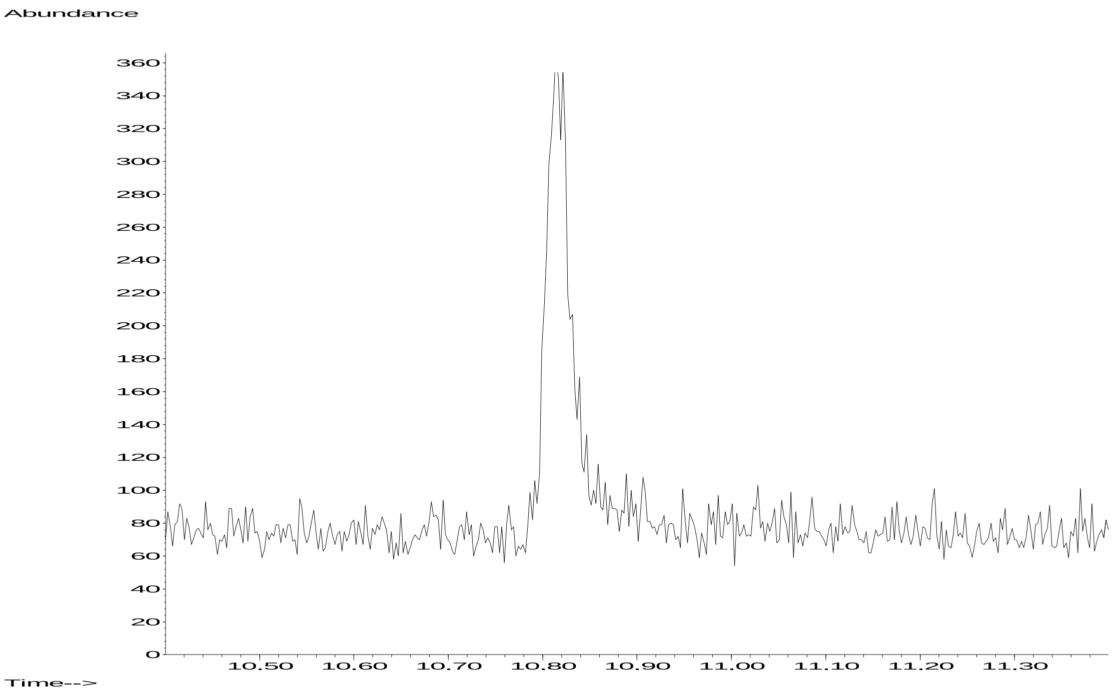
(c) Pork mince blank



(d) Pork mince + overspike at 420 µg/kg



(e) Orange juice blank



(f) Orange juice + overspike at 420 µg/kg

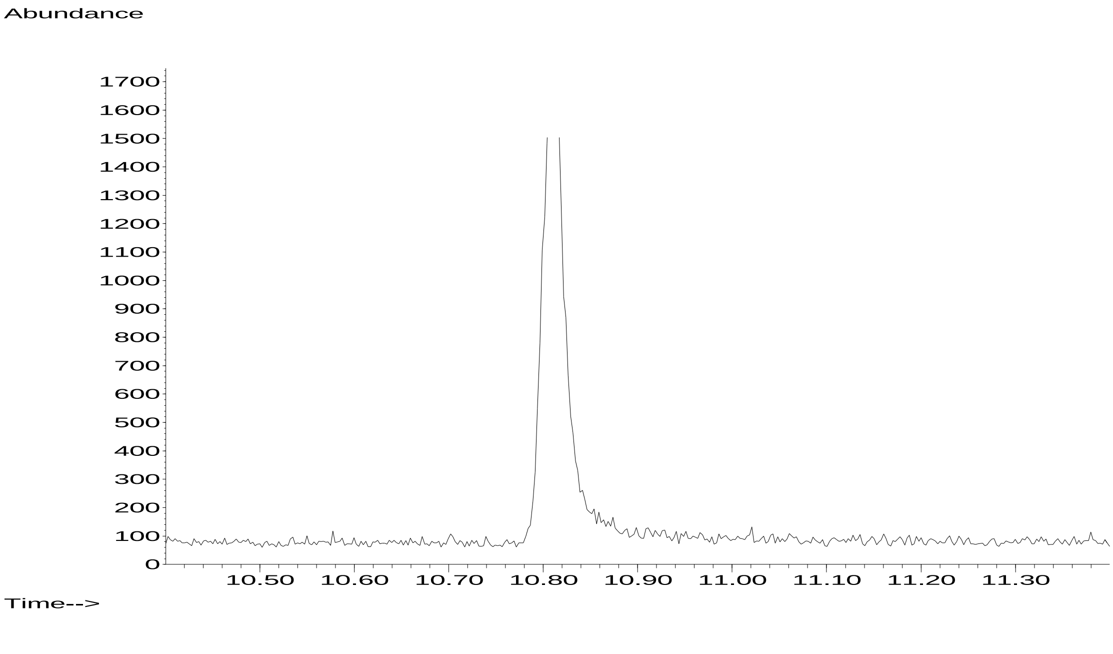


Figure 72. Calibration graph for dimethyl phthalate in acetonitrile

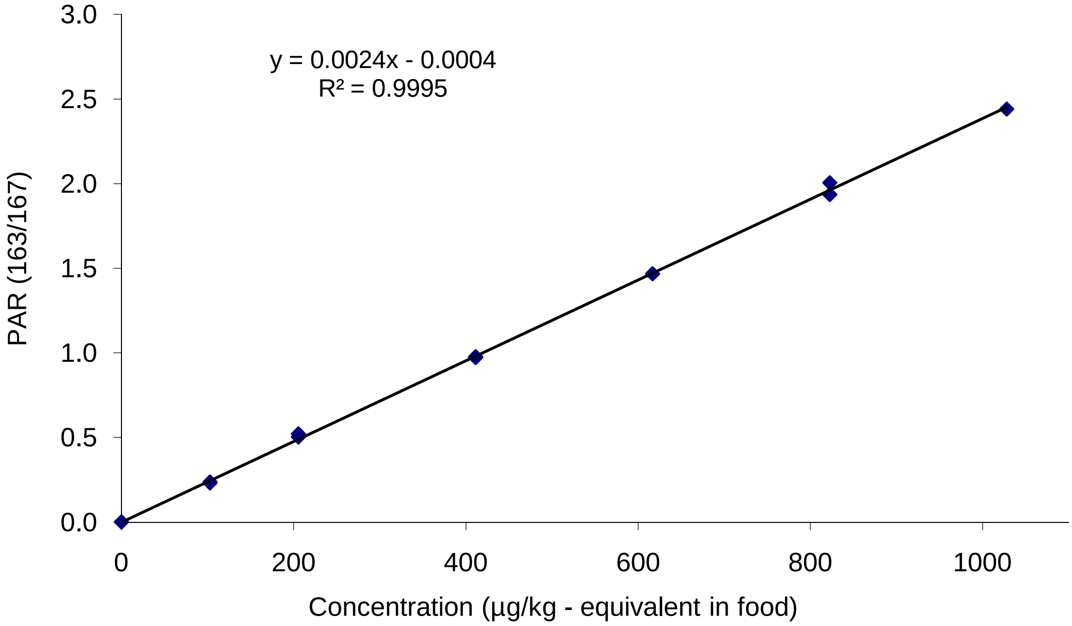


Figure 73. Diisobutyl phthalate concentrations in the lasagne sheets taken at different distances from the packaging

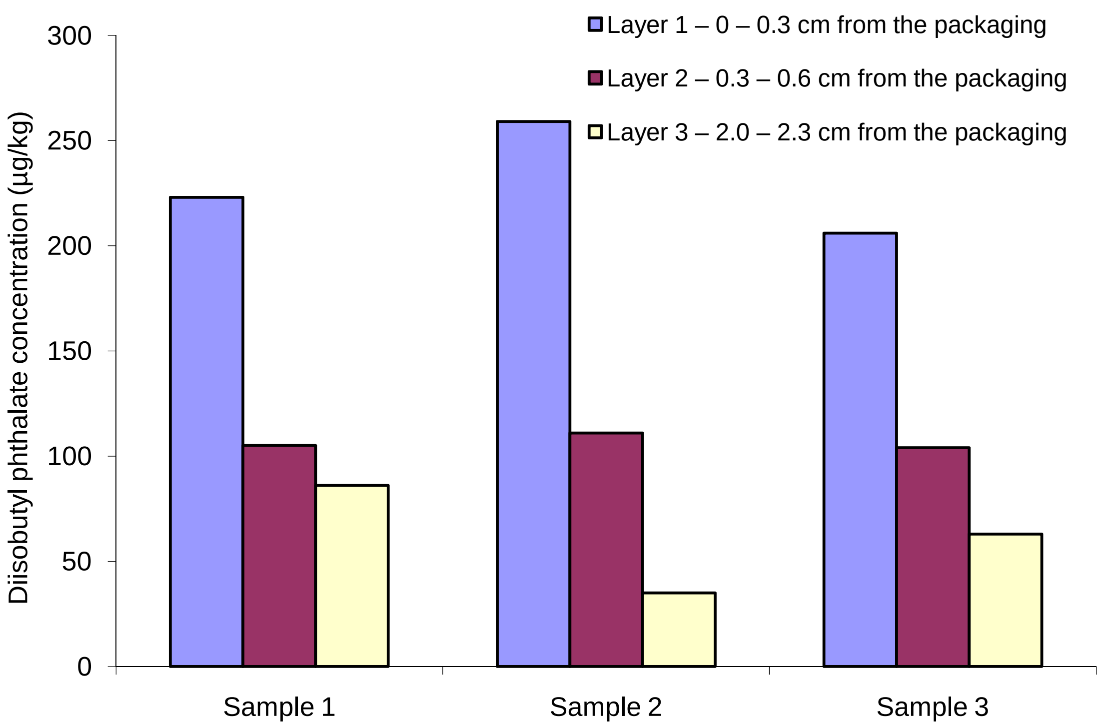


Table 1. Phthalate diester - ions monitored

Phthalate diester	Ions monitored (m/z)
Dimethyl phthalate	77,163, 194
Diethyl phthalate	149, 177, 222
Diisopropyl phthalate	149, 192, 209
Diallyl phthalate	104, 149, 189
Diisobutyl phthalate	149, 205, 223
Di-n-butyl phthalate	149, 205, 223
Di-n-pentyl phthalate	149, 219, 237
Di-n-hexyl phthalate	149, 233, 251
Benzyl butyl phthalate	149, 206
Dicyclohexyl phthalate	149, 167, 249
Di-(2-ethylhexyl) phthalate	149, 167, 279
Di-n-heptyl phthalate	149, 167, 265
Di-n-octyl phthalate	149, 167, 279
n-Octyl-n-decyl phthalate	149, 279, 307
Diisononyl phthalate	149, 167, 293
Diisodecyl phthalate	149, 167, 307
Di-n-decyl phthalate	149, 167, 307

Table 2. Limits of detection and quantification of the phthalate diesters in the three food matrices tested

ANALYTE	LOD (µg/kg) in cheese	LOQ (µg/kg) in cheese	LOD (µg/kg) in pork mince	LOQ (µg/kg) in pork mince	LOD (µg/kg) in orange juice	LOQ (µg/kg) in orange juice
Dimethyl phthalate	2.0	6.8	2.0	6.6	3.4	11.5
Diethyl phthalate	1.5	5.1	1.6	5.4	1.7	5.5
Diisopropyl phthalate	0.9	2.7	0.9	2.7	1.6	5.2
Diallyl phthalate	11.1 *	37.0 *	7.3	24.3	7.1	23.7
Diisobutyl phthalate	11.1 [#]	37.0 [#]	11.1 [#]	37.0 [#]	11.1 [#]	37.0 [#]
Di-n-butyl phthalate	4.2 [#]	14.0 [#]	4.2 [#]	14.0 [#]	4.2 [#]	14.0 [#]
Dipentyl phthalate	4.8 ◄	16.0 ◄	1.3 *◄	4.3 *◄	1.1 ◄	3.7 ◄
Di-n-hexyl phthalate	3.2	10.6	0.8	2.5	3.8	12.8
Benzyl butyl phthalate	11.5	38.4	13.3	44.3	7.3	24.3
Dicyclohexyl phthalate	5.8	19.3	5.1	17.0	1.3	4.3
Di-(2-ethylhexyl) phthalate	6.0 *	20.0 *	6.0 *	20.0 *	6.0 *	20.0 *
Di-n-heptyl phthalate	12.4	41.2	7.1	23.8	13.4	44.7
Di-n-octyl phthalate	10.7	35.7	2.1 *	7.0 *	2.1	7.0
n-Octyl-n-decyl phthalate	8.7	29.0	1.8	6.0	2.2	7.3
Diisononyl phthalate	115	385	55	182	26	88
Diisodecyl phthalate	214	714	105	351	50	167
Di-n-decyl phthalate	3.3 *	11.0 *	3.3 *	11.0 *	3.3 *	11.0 *

LOD = Limit of detection   LOQ = Limit of quantification

* Positive response in sample. LOD/LOQ calculated from procedural blanks and procedural spiked samples.

[#] A positive response was detected in the procedural blank samples. LOD and LOQ calculated as 3x and 10x the concentration detected in the procedural blank samples.

◄ LOD and LOQ values have been calculated assuming 10% of the standard is di-n-pentyl phthalate

Table 3. Repeatability and reproducibility data – dimethyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	Cheese		Pork mince		Orange juice	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	53.7	295.2	48.9	No data	49.6	257.9
Analyst 1 replicate 2	52.4	284.7	40.4	426.0	50.5	273.1
Analyst 1 replicate 3	50.5	292.6	57.1	391.6	44.1	284.1
Analyst 1 replicate 4	51.5	288.4	49.6	321.0	49.2	295.4
Analyst 1 replicate 5	67.0	278.2	445.8	365.0	52.1	295.5
Analyst 1 replicate 6	56.9	297.4	44.2	293.3	47.9	268.9
Analyst 1 replicate 7	76.3	287.6	59.1	292.8	51.2	292.8
Analyst 1 replicate 8	74.8	293.2	51.3	260.3	48.8	295.4
Average	**60.4**	**289.7**	**49.5**	**335.7**	**49.2**	**282.9**
RSD (%)	**17.7**	**2.2**	**12.7**	**17.9**	**5.0**	**5.2**
Analyst 2 replicate 1	47.6	265.1	44.1	296.3	45.0	274.1
Analyst 2 replicate 2	45.9	274.8	49.9	304.5	50.9	276.4
Analyst 2 replicate 3	48.2	273.5	47.4	287.4	43.1	268.8
Average	**47.2**	**271.1**	**47.1**	**296.1**	**46.3**	**273.1**
Recovery (%)	**81.8**	**88.3**	**93.8**	**98.6**	**92.6**	**91.0**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 4. Repeatability and reproducibility data – diethyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	53.5	299.8	54.8	249.7	45.9	285.9
Analyst 1 replicate 2	52.3	289.0	No data	339.9	55.1	291.1
Analyst 1 replicate 3	48.8	299.3	54.9	228.8	No data	290.2
Analyst 1 replicate 4	50.6	292.5	39.8	353.8	50.8	289.8
Analyst 1 replicate 5	57.4	294.0	49.5	301.2	59.8	298.2
Analyst 1 replicate 6	54.8	293.3	50.7	287.4	48.0	278.4
Analyst 1 replicate 7	66.2	287.0	32.1	418.0	48.0	272.3
Analyst 1 replicate 8	48.3	295.0	43.9	319.1	47.0	294.7
Average	**54.0**	**293.7**	**46.5**	**312.2**	**50.7**	**287.6**
RSD (%)	**10.8**	**1.5**	**18.1**	**19.3**	**10.0**	**3.0**
Analyst 2 replicate 1	52.8	290.6	44.1	296.3	37.5	381.2
Analyst 2 replicate 2	45.9	310.6	49.9	304.5	68.2	238.1
Analyst 2 replicate 3	49.1	310.4	47.4	287.4	66.2	353.9
Average	**49.3**	**303.9**	**47.1**	**296.1**	**57.3**	**324.4**
Recovery (%)	**98.5**	**101.3**	**93.8**	**98.6**	**114.6**	**108.4**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 5. Repeatability and reproducibility data – diisopropyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	Cheese		Pork mince		Orange juice	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	39.4	249.5	44.7	254.2	28.8	217.5
Analyst 1 replicate 2	39.8	256.8	48.1	309.7	26.2	251.3
Analyst 1 replicate 3	41.3	257.6	55.7	302.3	33.0	167.2
Analyst 1 replicate 4	39.6	238.0	50.4	295.4	23.1	234.6
Analyst 1 replicate 5	43.5	233.1	48.3	321.6	34.6	No data
Analyst 1 replicate 6	46.4	259.0	48.1	280.8	No data	215.2
Analyst 1 replicate 7	44.5	277.8	51.7	307.9	33.5	No data
Analyst 1 replicate 8	45.8	271.7	52.3	286.6	31.7	230.7
Average	**42.5**	**255.5**	**49.9**	**294.8**	**30.1**	**219.4**
RSD (%)	**6.7**	**6.0**	**6.7**	**7.1**	**14.1**	**13.1**
Analyst 2 replicate 1	47.0	285.6	43.5	285.2	42.7	179.2
Analyst 2 replicate 2	47.6	286.3	48.0	306.6	38.4	296.8
Analyst 2 replicate 3	47.4	289.3	46.3	279.4	43.9	250.3
Average	**47.3**	**287.1**	**46.0**	**290.4**	**41.6**	**242.1**
Recovery (%)	**94.5**	**95.7**	**91.9**	**96.8**	**83.3**	**80.7**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 6. Repeatability and reproducibility data - diallyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	Cheese		Pork mince		Orange juice	
	50 µg/kg spike	300 µg/kg spike	50 µg/kg spike	300 µg/kg spike	50 µg/kg spike	300 µg/kg spike
Analyst 1 replicate 1	59.0	396.2	46.6	320.3	46.6	294.0
Analyst 1 replicate 2	63.3	400.1	51.6	321.5	52.5	307.3
Analyst 1 replicate 3	70.3	403.8	62.0	301.6	52.2	279.2
Analyst 1 replicate 4	75.1	404.1	53.7	313.0	49.7	298.8
Analyst 1 replicate 5	78.8	387.3	49.6	319.7	53.2	No data
Analyst 1 replicate 6	84.3	406.1	47.5	334.6	49.7	334.5
Analyst 1 replicate 7	82.0	413.3	49.0	355.6	51.4	273.3
Analyst 1 replicate 8	82.4	425.8	55.1	317.1	53.4	323.7
Average	**74.4**	**404.6**	**51.9**	**322.9**	**51.1**	**301.5**
RSD (%)	**12.6**	**2.8**	**9.7**	**5.0**	**4.5**	**7.4**
Analyst 2 replicate 1	57.6	353.0	44.9	302.6	53.9	285.6
Analyst 2 replicate 2	58.0	332.7	53.4	323.9	42.5	279.9
Analyst 2 replicate 3	57.9	355.3	47.8	293.8	41.5	273.9
Average	**57.8**	**347.0**	**48.7**	**306.8**	**46.0**	**273.9**
Recovery (%)	**115.6**	**115.7**	**97.4**	**102.3**	**91.9**	**93.3**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 7. Repeatability and reproducibility data – diisobutyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	51.3	297.3	45.5	292.5	50.9	300.3
Analyst 1 replicate 2	56.3	283.0	51.3	295.4	50.4	295.0
Analyst 1 replicate 3	50.7	282.7	54.6	288.1	53.5	291.9
Analyst 1 replicate 4	51.7	296.0	52.7	299.4	52.3	301.4
Analyst 1 replicate 5	51.0	283.2	47.3	324.6	50.0	No data
Analyst 1 replicate 6	51.9	288.6	49.9	286.9	50.2	291.0
Analyst 1 replicate 7	50.4	291.7	50.5	336.4	48.9	292.9
Analyst 1 replicate 8	52.6	282.2	49.9	295.7	49.8	294.5
Average	**52.0**	**288.1**	**50.2**	**302.4**	**50.8**	**295.3**
RSD (%)	**3.6**	**2.2**	**5.7**	**6.0**	**2.9**	**1.4**
Analyst 2 replicate 1	42.1	273.4	43.1	268.6	44.1	276.6
Analyst 2 replicate 2	46.3	275.7	45.5	299.2	45.3	291.7
Analyst 2 replicate 3	44.9	275.9	45.5	262.5	45.5	276.1
Average	**44.4**	**275.0**	**44.7**	**276.7**	**45.0**	**281.5**
Recovery (%)	**88.9**	**91.7**	**89.4**	**92.2**	**89.9**	**93.8**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 8. Repeatability and reproducibility data – di-n-butyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	50.6	311.4	48.5	284.6	51.9	307.1
Analyst 1 replicate 2	52.0	299.6	54.9	294.9	51.6	301.9
Analyst 1 replicate 3	54.7	289.9	58.6	288.6	56.1	298.3
Analyst 1 replicate 4	61.9	302.5	57.5	300.9	53.4	307.4
Analyst 1 replicate 5	55.2	288.6	49.5	331.2	52.7	No data
Analyst 1 replicate 6	58.0	289.1	52.1	286.0	50.9	297.4
Analyst 1 replicate 7	59.4	305.2	55.7	336.3	51.0	306.5
Analyst 1 replicate 8	57.6	302.4	52.2	300.7	52.8	294.4
Average	**56.2**	**298.6**	**53.6**	**302.9**	**52.6**	**301.9**
RSD (%)	**6.8**	**2.8**	**6.8**	**6.6**	**3.2**	**1.8**
Analyst 2 replicate 1	43.4	269.9	45.2	265.7	48.3	286.6
Analyst 2 replicate 2	46.4	279.5	44.5	300.5	48.4	296.9
Analyst 2 replicate 3	45.6	270.2	44.3	263.0	48.3	288.5
Average	**45.1**	**273.2**	**44.6**	**276.4**	**48.3**	**290.6**
Recovery (%)	**90.3**	**91.1**	**89.3**	**92.1**	**96.7**	**96.9**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 9. Repeatability and reproducibility data - dipentyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	57.9	318.8	50.1	313.6	53.9	309.9
Analyst 1 replicate 2	64.4	293.7	57.3	298.0	54.9	312.4
Analyst 1 replicate 3	50.8	376.1	67.0	294.8	68.7	306.2
Analyst 1 replicate 4	52.2	325.8	74.8	301.2	57.8	309.3
Analyst 1 replicate 5	46.2	337.6	60.9	358.9	58.5	No data
Analyst 1 replicate 6	49.2	360.5	47.4	360.3	53.6	314.8
Analyst 1 replicate 7	49.0	305.5	56.5	349.2	52.4	301.7
Analyst 1 replicate 8	55.5	306.1	61.7	296.3	52.2	315.1
Average	**53.2**	**328.0**	**59.5**	**321.5**	**56.5**	**309.9**
RSD (%)	**11.1**	**8.7**	**14.9**	**9.1**	**9.6**	**1.5**
Analyst 2 replicate 1	51.6	313.4	38.8	227.9	47.0	302.4
Analyst 2 replicate 2	59.1	327.6	47.4	251.1	49.7	312.0
Analyst 2 replicate 3	63.8	322.9	44.7	269.7	50.7	297.0
Average	**58.2**	**321.3**	**43.7**	**247.9**	**49.1**	**303.8**
Recovery (%)	**116.3**	**107.1**	**87.3**	82.6	**98.3**	**101.3**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 10. Repeatability and reproducibility data – di-n-hexyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	59.2	308.2	39.9	369.4	49.1	300.5
Analyst 1 replicate 2	68.6	286.0	57.4	269.4	51.6	289.2
Analyst 1 replicate 3	67.8	298.2	53.0	261.1	56.8	282.2
Analyst 1 replicate 4	50.6	301.0	56.5	291.9	52.5	294.5
Analyst 1 replicate 5	65.5	300.8	50.0	340.3	49.6	No data
Analyst 1 replicate 6	58.6	291.8	48.3	366.4	51.4	297.1
Analyst 1 replicate 7	62.3	333.9	48.2	343.6	48.2	293.4
Analyst 1 replicate 8	60.0	291.6	51.2	284.0	49.0	289.5
Average	**61.6**	**301.4**	**50.6**	**315.8**	**51.0**	**292.3**
RSD (%)	**9.5**	**4.9**	**10.9**	**13.9**	**5.4**	**2.1**
Analyst 2 replicate 1	45.5	286.1	53.1	259.8	47.8	273.9
Analyst 2 replicate 2	49.6	294.0	50.5	289.3	46.9	289.6
Analyst 2 replicate 3	53.3	No data	49.9	258.3	46.0	278.2
Average	**49.5**	**290.0**	**51.1**	**269.2**	**46.9**	**280.6**
Recovery (%)	**99.0**	**96.7**	**102.3**	**89.7**	**93.8**	**93.5**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 11. Repeatability and reproducibility data – benzyl butyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	42.8	273.6	41.8	245.7	46.5	293.2
Analyst 1 replicate 2	46.5	258.1	48.8	253.2	43.3	268.8
Analyst 1 replicate 3	39.7	262.1	42.7	252.5	45.6	268.9
Analyst 1 replicate 4	49.6	244.7	46.4	273.1	40.5	272.2
Analyst 1 replicate 5	46.1	256.6	40.6	283.9	42.6	No data
Analyst 1 replicate 6	46.0	250.6	45.6	248.8	41.9	234.6
Analyst 1 replicate 7	44.0	255.4	40.4	279.7	45.4	267.8
Analyst 1 replicate 8	48.5	254.3	42.5	280.5	44.2	232.0
Average	**45.4**	**256.9**	**43.6**	**264.7**	**43.8**	**262.5**
RSD (%)	**7.0**	**3.3**	**6.9**	**6.1**	**4.7**	**8.3**
Analyst 2 replicate 1	43.8	270.1	45.9	266.9	43.8	280.2
Analyst 2 replicate 2	47.1	273.2	49.4	299.1	46.5	292.9
Analyst 2 replicate 3	47.1	274.9	47.3	261.7	47.7	283.1
Average	**46.0**	**272.9**	**47.5**	**275.9**	**46.0**	**285.4**
Recovery (%)	**92.0**	**90.9**	**95.1**	**92.0**	**92.1**	**95.1**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 12. Repeatability and reproducibility data - dicyclohexyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	Cheese		Pork mince		Orange juice	
	50 µg/kg spike	300 µg/kg spike	50 µg/kg spike	300 µg/kg spike	50 µg/kg spike	300 µg/kg spike
Analyst 1 replicate 1	48.6	306.5	43.8	272.1	51.3	311.1
Analyst 1 replicate 2	51.0	292.6	51.7	281.9	52.2	302.4
Analyst 1 replicate 3	50.3	290.8	52.6	275.9	52.1	302.0
Analyst 1 replicate 4	50.2	296.5	50.8	295.6	52.1	305.3
Analyst 1 replicate 5	48.6	284.7	43.5	321.0	51.1	No data
Analyst 1 replicate 6	48.6	298.9	48.2	285.4	63.5	302.6
Analyst 1 replicate 7	49.5	301.7	48.5	340.4	50.4	301.5
Analyst 1 replicate 8	53.0	287.8	48.1	281.5	51.3	301.0
Average	**50.0**	**294.9**	**48.4**	**294.2**	**53.0**	**303.7**
RSD (%)	**3.1**	**2.5**	**7.0**	**8.2**	**8.1**	**1.2**
Analyst 2 replicate 1	46.1	274.8	41.6	254.0	45.0	281.3
Analyst 2 replicate 2	46.9	280.4	46.0	284.0	45.6	293.1
Analyst 2 replicate 3	48.9	273.0	45.7	246.5	46.4	278.9
Average	**47.3**	**276.1**	**44.4**	**261.5**	**45.7**	**284.5**
Recovery (%)	**94.7**	**92.0**	**88.9**	**87.2**	**91.4**	**94.8**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 13. Repeatability and reproducibility data – di-(2-ethylhexyl) phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	Cheese		Pork mince		Orange juice	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	351.2	628.9	125.0	329.4	56.2	309.7
Analyst 1 replicate 2	303.3	651.0	144.9	394.0	69.5	311.9
Analyst 1 replicate 3	343.4	690.7	134.6	348.9	56.3	300.5
Analyst 1 replicate 4	422.9	820.4	152.1	346.8	55.3	306.0
Analyst 1 replicate 5	465.4	577.7	121.8	398.3	53.5	No data
Analyst 1 replicate 6	374.9	622.6	140.0	407.9	55.2	301.9
Analyst 1 replicate 7	326.1	578.1	139.6	479.8	52.0	300.2
Analyst 1 replicate 8	450.1	765.9	171.2	365.6	54.0	294.9
Average	**379.7**	**666.9**	**141.1**	**383.8**	**56.5**	**303.6**
RSD (%)	**15.8**	**13.1**	**11.1**	**12.5**	**9.6**	**2.0**
Analyst 2 replicate 1	37.3	273.0	25.7	258.3	58.1	315.2
Analyst 2 replicate 2	50.1	226.5	53.3	280.7	57.5	322.6
Analyst 2 replicate 3	35.9	285.4	58.3	278.1	57.0	313.5
Average	**41.1**	**261.6**	**45.8**	**272.4**	**57.5**	**317.1**
Recovery (%)	**82.2**	**87.2**	**91.5**	**90.8**	**115.0**	**105.7**

Concentrations normalised to 50 and 300 µg/kg for clarity.
When determining the repeatability the phthalate concentrations in the unspiked foodstuffs were not determined and therefore the replicate results for Analyst 1 have not been corrected for any background levels present in the foodstuffs.

Table 14. Repeatability and reproducibility data - di-n-heptyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	63.9	311.2	43.8	277.4	52.7	297.8
Analyst 1 replicate 2	63.2	299.4	53.2	287.9	54.3	303.9
Analyst 1 replicate 3	63.1	278.5	55.1	277.9	52.9	296.7
Analyst 1 replicate 4	72.1	257.9	42.4	295.2	56.0	310.0
Analyst 1 replicate 5	60.2	301.6	40.8	326.8	49.9	No data
Analyst 1 replicate 6	57.4	231.9	48.7	316.4	52.9	300.2
Analyst 1 replicate 7	55.4	332.0	52.0	349.1	50.1	295.2
Analyst 1 replicate 8	67.3	303.4	49.8	283.9	53.1	297.6
Average	**62.8**	**289.5**	**48.2**	**301.8**	**52.7**	**300.2**
RSD (%)	**8.5**	**11.0**	**11.0**	**8.7**	**3.8**	**1.7**
Analyst 2 replicate 1	40.5	281.5	39.0	267.2	46.8	280.9
Analyst 2 replicate 2	46.4	278.8	60.3	286.4	49.8	295.2
Analyst 2 replicate 3	45.2	287.2	55.6	266.4	47.3	285.9
Average	**44.1**	**282.5**	**51.6**	**273.3**	**48.0**	**287.4**
Recovery (%)	**88.1**	**94.2**	**103.3**	**91.1**	**95.9**	**95.8**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 15. Repeatability and reproducibility data – di-n-octyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	82.5	309.3	34.6	247.2	48.4	299.7
Analyst 1 replicate 2	60.4	300.0	35.3	291.9	50.9	287.5
Analyst 1 replicate 3	57.0	291.1	48.5	261.4	50.2	286.9
Analyst 1 replicate 4	55.7	297.9	40.7	277.1	52.2	302.5
Analyst 1 replicate 5	56.6	294.3	37.1	299.1	51.0	No data
Analyst 1 replicate 6	60.0	302.5	45.9	309.6	47.6	296.6
Analyst 1 replicate 7	57.9	324.7	41.6	327.2	48.5	294.8
Analyst 1 replicate 8	50.8	289.1	42.7	273.0	50.2	298.8
Average	**60.1**	**301.1**	**40.8**	**285.8**	**49.9**	**295.2**
RSD (%)	**15.9**	**3.8**	**12.1**	**9.2**	**3.1**	**2.0**
Analyst 2 replicate 1	46.4	254.2	38.7	227.2	42.8	270.9
Analyst 2 replicate 2	49.8	265.5	42.0	270.1	43.8	282.1
Analyst 2 replicate 3	48.6	474.6	39.4	236.1	43.9	272.8
Average	**48.3**	**331.4**	**40.0**	**244.5**	**43.5**	**275.2**
Recovery (%)	**96.5**	**110.5**	**80.1**	**81.5**	**87.1**	**91.7**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 16. Repeatability and reproducibility data – n-octyl-n-decyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	50 µg/kg spike	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**	**50 µg/kg spike**	**300 µg/kg spike**
Analyst 1 replicate 1	51.8	281.1	48.5	262.1	51.8	309.0
Analyst 1 replicate 2	60.9	308.5	39.6	264.1	51.3	315.4
Analyst 1 replicate 3	57.7	301.1	42.0	269.7	53.7	320.2
Analyst 1 replicate 4	46.5	292.4	44.1	273.5	52.8	323.3
Analyst 1 replicate 5	58.9	296.3	50.5	282.1	52.8	No data
Analyst 1 replicate 6	48.0	299.1	42.4	252.0	52.7	322.3
Analyst 1 replicate 7	63.4	313.2	51.9	205.7	52.8	335.6
Analyst 1 replicate 8	55.4	332.2	35.9	305.1	52.0	331.1
Average	**55.3**	**303.0**	**44.4**	**264.3**	**52.5**	**322.4**
RSD (%)	**11.0**	**5.1**	**12.5**	**10.8**	**1.5**	**2.8**
Analyst 2 replicate 1	51.4	294.1	51.2	371.1	42.4	298.2
Analyst 2 replicate 2	45.2	301.0	48.5	337.8	44.4	338.5
Analyst 2 replicate 3	58.8	283.5	54.4	369.2	50.2	345.9
Average	**51.8**	**292.9**	**51.4**	**359.4**	**45.6**	**327.5**
Recovery (%)	**103.6**	**97.6**	**102.8**	**119.8**	**91.3**	**109.2**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 17. Repeatability and reproducibility data – diisononyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 1 and 5 mg/kg

Sample	Cheese		Pork mince		Orange juice	
	1 mg/kg spike	**5 mg/kg spike**	**1 mg/kg spike**	**5 mg/kg spike**	**1 mg/kg spike**	**5 mg/kg spike**
Analyst 1 replicate 1	1.1	5.2	1.5	4.6	0.8	4.0
Analyst 1 replicate 2	1.2	6.4	1.5	3.1	0.6	3.7
Analyst 1 replicate 3	1.3	5.9	1.6	4.0	0.7	4.8
Analyst 1 replicate 4	1.4	4.8	1.2	3.5	0.7	5.1
Analyst 1 replicate 5	1.4	3.6	1.2	5.0	0.8	3.7
Analyst 1 replicate 6	1.4	6.1	1.4	No data	0.8	4.9
Analyst 1 replicate 7	1.4	5.0	1.1	4.4	0.7	3.9
Analyst 1 replicate 8	1.6	4.5	1.1	4.9	0.8	4.2
Average	**1.3**	**5.2**	**1.3**	**4.2**	**0.8**	**4.3**
RSD (%)	**10.5**	**17.5**	**16.0**	**16.3**	**7.0**	**13.4**
Analyst 2 replicate 1	1.0	6.0	0.9	3.9	1.0	4.8
Analyst 2 replicate 2	1.1	5.2	0.6	5.5	1.1	7.0
Analyst 2 replicate 3	0.9	5.3	0.7	49.	1.0	No data
Average	**1.0**	**5.5**	**0.7**	**4.8**	**1.0**	**5.9**
Recovery (%)	**102.6**	**110.1**	**67.6**	**94.8**	**102.8**	**118.1**

Concentrations normalised to 1 and 5 mg/kg for clarity.

Table 18. Repeatability and reproducibility data – diisodecyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 1 and 5 mg/kg

Sample	**Cheese**		**Pork mince**		**Orange juice**	
	1 mg/kg spike	**5 mg/kg spike**	**1 mg/kg spike**	**5 mg/kg spike**	**1 mg/kg spike**	**5 mg/kg spike**
Analyst 1 replicate 1	0.9	4.1	1.0	3.6	0.7	3.5
Analyst 1 replicate 2	0.8	4.7	1.1	3.0	0.8	3.6
Analyst 1 replicate 3	0.9	5.1	1.2	3.2	0.7	4.2
Analyst 1 replicate 4	0.8	6.4	0.9	2.8	0.7	4.6
Analyst 1 replicate 5	0.8	3.9	0.9	3.9	0.7	3.4
Analyst 1 replicate 6	1.1	4.1	1.2	No data	0.7	4.3
Analyst 1 replicate 7	1.2	4.5	0.8	3.3	0.7	3.5
Analyst 1 replicate 8	1.2	4.3	0.8	4.5	0.8	3.7
Average	**1.0**	**4.6**	**1.0**	**3.5**	**0.7**	**3.8**
RSD (%)	**18.2**	**17.1**	**15.1**	**16.3**	**7.0**	**11.8**
Analyst 2 replicate 1	0.6	5.7	0.9	5.8	1.0	5.0
Analyst 2 replicate 2	1.0	6.3	0.6	4.0	1.1	6.2
Analyst 2 replicate 3	1.0	5.0	0.6	5.3	1.0	6.6
Average	**0.9**	**5.7**	**0.7**	**4.4**	**1.0**	**5.9**
Recovery (%)	**89.3**	**107.5**	**72.0**	**87.1**	**100.2**	**118.4**

Concentrations normalised to 1 and 5 mg/kg for clarity.

Table 19. Repeatability and reproducibility data – di-n-decyl phthalate: Determined concentration in the three selected foodstuffs spiked at nominal concentrations of 50 and 300 µg/kg

Sample	Cheese		Pork mince		Orange juice	
	50 µg/kg spike	300 µg/kg spike	50 µg/kg spike	300 µg/kg spike	50 µg/kg spike	300 µg/kg spike
Analyst 1 replicate 1	42.9	193.4	26.6	231.9	55.6	402.4
Analyst 1 replicate 2	65.5	203.7	31.7	267.6	48.1	405.7
Analyst 1 replicate 3	51.6	220.2	48.0	234.5	53.9	386.3
Analyst 1 replicate 4	64.6	195.1	34.4	271.0	65.9	393.5
Analyst 1 replicate 5	39.7	208.8	29.9	287.7	61.1	274.7
Analyst 1 replicate 6	47.2	207.9	41.0	294.5	51.6	405.0
Analyst 1 replicate 7	58.4	214.3	42.1	323.2	59.5	415.5
Analyst 1 replicate 8	54.9	204.4	36.8	268.4	57.1	419.9
Average	**53.1**	**206.0**	**36.3**	**272.4**	**56.6**	**387.9**
RSD (%)	**18.0**	**4.4**	**19.6**	**11.1**	**9.9**	**12.1**
Analyst 2 replicate 1	39.7	213.9	40.5	211.1	43.4	281.4
Analyst 2 replicate 2	43.2	247.2	42.0	216.7	40.1	289.7
Analyst 2 replicate 3	45.5	410.9	35.6	216.7	42.3	290.7
Average	**42.8**	**290.7**	**39.4**	**214.8**	**41.9**	**287.1**
Recovery (%)	**85.7**	**96.9**	**78.8**	**71.6**	**83.9**	**95.7**

Concentrations normalised to 50 and 300 µg/kg for clarity.

Table 20. Monophthalate esters - transitions and collision energies

Analyte	**Transitions**	**Collision energy (eV)**
Monoisopropyl phthalate	206.8 → 120.8 206.8 → 137.9	17 10
Total mono-n-butyl phthalate and monoisobutyl phthalate	220.9 → 177.0 220.9 → 133.9	10 13
Mono-n-butyl-phthalate ONLY	220.9 → 69.0	10
Monobenzyl phthalate	255.0 → 106.9 255.0 → 182.8	15 12
Monocyclohexyl phthalate	246.9 → 97.0 246.9 → 146.9	15 15
Mono-n-pentyl phthalate	234.9 → 85.0 234.9 → 191.1	15 10
Mono-2-ethylhexyl phthalate	277.1 → 133.9 277.1 → 127.0	15 15
Mono-n-octyl phthalate	277.1 → 127.0 277.1 → 233.0	18 15
Monoisononyl phthalate	291.0 → 140.9 291.0 → 247.0	18 15

Table 21. Limits of detection and quantification of the phthalate monoesters in the three food matrices tested

ANALYTE	LOD (µg/kg) in cheese	LOQ (µg/kg) in cheese	LOD (µg/kg) in pork mince	LOQ (µg/kg) in pork mince	LOD (µg/kg) in orange juice	LOQ (µg/kg) in orange juice
Monoisopropyl phthalate	1.8	6.0	1.9	6.3	1.7	5.6
Total monoisobutyl phthalate and mono-n-butyl phthalate	4.1	13.5	2.8	9.3	4.9	16.2
Mono-n-butyl phthalate	2.0	6.6	4.1	13.7	4.2	13.8
Monobenzyl phthalate	1.9	6.3	2.0	6.6	1.7	5.6
Monocyclohexyl phthalate	1.7	5.7	1.7	5.7	1.6	5.5
Mono-n-pentyl phthalate	1.8	5.9	1.8	5.9	1.7	5.6
Mono-2-ethylhexyl phthalate	2.0	6.7	6.1	20.4	1.7	5.6
Mono-n-octyl phthalate	1.7	5.7	1.7	5.6	1.7	5.6
Monoisononyl phthalate	3.4	11.4	2.6	8.6	1.7	5.7

Table 22. Repeatability and reproducibility data – monoisopropyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	**Cheese**	**Pork mince**	**Orange juice**
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	39.8	32.3	51.6
Analysis 1 replicate 2	38.5	32.0	48.6
Analysis 1 replicate 3	40.0	29.7	53.4
Analysis 1 replicate 4	43.0	28.2	54.0
Analysis 1 replicate 5	39.0	25.4	50.6
Analysis 1 replicate 6	40.5	35.7	54.3
Average	**40.1**	**30.6**	**52.1**
RSD (%)	**4.0**	**11.7**	**4.3**
Recovery (%)	**85.4**	**65.0**	**110.8**
Analysis 2 replicate 1	42.7	28.2	43.3
Analysis 2 replicate 2	40.6	28.1	45.5
Analysis 2 replicate 3	35.4	27.7	44.2
Average	**39.6**	**28.0**	**44.3**

Table 23. Repeatability and reproducibility data – monoisobutyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	**Cheese**	**Pork mince**	**Orange juice**
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	48.6	41.5	47.1
Analysis 1 replicate 2	57.0	43.0	40.0
Analysis 1 replicate 3	59.3	58.4	42.9
Analysis 1 replicate 4	41.7	48.2	52.6
Analysis 1 replicate 5	37.1	44.2	50.7
Analysis 1 replicate 6	58.2	60.4	30.7
Average	**50.3**	**49.3**	**44.0**
RSD (%)	**18.7**	**16.6**	**18.3**
Recovery (%)	**100.6**	**98.6**	**88.0**
Analysis 2 replicate 1	57.8	36.8	37.0
Analysis 2 replicate 2	43.5	46.8	43.3
Analysis 2 replicate 3	54.0	50.5	37.6
Average	**51.7**	**44.7**	**39.3**

Table 24. Repeatability and reproducibility data – mono-n-butyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	**Cheese**	**Pork mince**	**Orange juice**
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	37.6	41.5	54.5
Analysis 1 replicate 2	59.0	55.0	45.5
Analysis 1 replicate 3	47.7	48.4	57.0
Analysis 1 replicate 4	45.2	53.9	53.8
Analysis 1 replicate 5	55.8	59.0	49.8
Analysis 1 replicate 6	40.8	59.0	46.9
Average	**47.7**	**52.8**	**51.2**
RSD (%)	**17.5**	**12.9**	**8.9**
Recovery (%)	**95.3**	**105.6**	**102.5**
Analysis 2 replicate 1	52.3	51.1	48.3
Analysis 2 replicate 2	55.6	56.3	54.7
Analysis 2 replicate 3	34.0	63.3	53.3
Average	**47.3**	**56.9**	**52.1**

Table 25. Repeatability and reproducibility data – monobenzyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	**Cheese**	**Pork mince**	**Orange juice**
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	49.1	42.4	56.5
Analysis 1 replicate 2	47.8	42.9	55.4
Analysis 1 replicate 3	47.9	44.9	56.7
Analysis 1 replicate 4	52.1	47.9	57.3
Analysis 1 replicate 5	55.5	44.2	54.5
Analysis 1 replicate 6	49.6	48.0	60.6
Average	**50.3**	**45.0**	**56.8**
RSD (%)	**5.9**	**5.4**	**3.7**
Recovery (%)	**107.1**	**95.7**	**120.9**
Analysis 2 replicate 1	51.8	45.1	52.3
Analysis 2 replicate 2	54.5	53.2	55.3
Analysis 2 replicate 3	57.6	43.0	54.9
Average	**54.6**	**47.1**	**54.2**

Table 26. Repeatability and reproducibility data – monocyclohexyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	**Cheese**	**Pork mince**	**Orange juice**
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	49.8	56.3	56.9
Analysis 1 replicate 2	50.0	60.1	55.5
Analysis 1 replicate 3	50.1	59.8	57.5
Analysis 1 replicate 4	51.6	54.7	59.3
Analysis 1 replicate 5	46.7	55.0	57.6
Analysis 1 replicate 6	48.9	56.0	64.6
Average	**49.5**	**57.0**	**58.6**
RSD (%)	**3.3**	**4.2**	**5.4**
Recovery (%)	**105.3**	**121.3**	**124.7**
Analysis 2 replicate 1	54.9	61.4	49.0
Analysis 2 replicate 2	52.6	60.5	47.3
Analysis 2 replicate 3	48.8	56.7	46.6
Average	**52.1**	**59.5**	**47.6**

Table 27. Repeatability and reproducibility data – mono-n-pentyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	Cheese	Pork mince	Orange juice
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	54.2	51.7	48.7
Analysis 1 replicate 2	53.4	54.1	50.5
Analysis 1 replicate 3	54.7	54.5	53.3
Analysis 1 replicate 4	56.7	51.0	49.6
Analysis 1 replicate 5	51.8	50.8	50.2
Analysis 1 replicate 6	54.0	52.7	48.9
Average	**54.1**	**52.5**	**50.2**
RSD (%)	**3.0**	**3.0**	**3.4**
Recovery (%)	**115.1**	**111.7**	**106.8**
Analysis 2 replicate 1	63.7	59.9	55.5
Analysis 2 replicate 2	62.6	61.0	53.6
Analysis 2 replicate 3	56.5	56.6	54.4
Average	**60.9**	**59.2**	**54.5**

Table 28. Repeatability and reproducibility data – mono-2-ethylhexyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	Cheese	Pork mince	Orange juice
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	58.6	50.5	48.1
Analysis 1 replicate 2	52.4	47.8	48.1
Analysis 1 replicate 3	53.7	51.3	48.5
Analysis 1 replicate 4	51.8	53.7	47.0
Analysis 1 replicate 5	50.5	43.2	46.6
Analysis 1 replicate 6	54.3	51.5	49.2
Average	**53.5**	**49.7**	**47.9**
RSD (%)	**5.3**	**7.4**	**2.0**
Recovery (%)	**111.6**	**100.4**	**101.9**
Analysis 2 replicate 1	53.0	45.6	56.0
Analysis 2 replicate 2	55.2	43.8	57.0
Analysis 2 replicate 3	54.2	43.1	55.7
Average	**54.1**	**44.2**	**56.2**

Table 29. Repeatability and reproducibility data – mono-n-octyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	Cheese	Pork mince	Orange juice
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	47.7	42.3	48.2
Analysis 1 replicate 2	46.3	39.1	47.1
Analysis 1 replicate 3	46.3	42.3	49.1
Analysis 1 replicate 4	45.3	43.6	46.5
Analysis 1 replicate 5	43.6	35.5	46.9
Analysis 1 replicate 6	45.6	43.5	47.2
Average	**45.8**	**41.1**	**47.5**
RSD (%)	**3.0**	**7.7**	**2.0**
Recovery (%)	**94.3**	**85.0**	**101.1**
Analysis 2 replicate 1	44.5	39.2	44.6
Analysis 2 replicate 2	48.0	42.5	45.7
Analysis 2 replicate 3	45.2	38.3	46.0
Average	**48.9**	**40.0**	**45.4**

Table 30. Repeatability and reproducibility data – monoisononyl phthalate: Determined concentration in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	**Cheese**	**Pork mince**	**Orange juice**
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	44.3	38.6	46.7
Analysis 1 replicate 2	45.2	39.2	47.5
Analysis 1 replicate 3	45.9	38.4	48.9
Analysis 1 replicate 4	44.0	37.9	49.6
Analysis 1 replicate 5	42.5	35.3	46.7
Analysis 1 replicate 6	46.0	38.5	48.2
Average	**44.6**	**38.0**	**47.9**
RSD (%)	**3.0**	**3.6**	**2.5**
Recovery (%)	**95.0**	**80.8**	**102.0**
Analysis 2 replicate 1	60.9	49.7	52.2
Analysis 2 replicate 2	62.1	50.2	53.6
Analysis 2 replicate 3	57.4	51.6	53.5
Average	**60.1**	**50.5**	**53.1**

Table 31. Repeatability and reproducibility data – phthalic acid. Concentration determined in the three selected foodstuffs spiked at a nominal concentration of 50 µg/kg

Sample	**Cheese**	**Pork mince**	**Orange juice**
	50 µg/kg spike	**50 µg/kg spike**	**50 µg/kg spike**
Analysis 1 replicate 1	54.0	39.7	58.5
Analysis 1 replicate 2	44.4	34.8	58.9
Analysis 1 replicate 3	47.6	46.5	59.6
Analysis 1 replicate 4	46.0	46.7	58.8
Analysis 1 replicate 5	48.4	47.4	57.3
Analysis 1 replicate 6	43.6	46.1	56.4
Average	**47.3**	**43.5**	**58.2**
RSD (%)	**7.9**	**11.8**	**2.0**
Recovery (%)	**95**	**87**	**116**
Analysis 2 replicate 1	48.3	52.3	57.4
Analysis 2 replicate 2	45.4	57.4	50.2
Analysis 2 replicate 3	41.1	54.2	Lost
Average	**44.9**	**54.6**	**53.8**

Table 32. Limits of detection and quantification of the dimethyl phthalate in the three food matrices tested

LOD (µg/kg) in cheese	LOQ (µg/kg) in cheese	LOD (µg/kg) in pork mince	LOQ (µg/kg) in pork mince	LOD (µg/kg) in orange juice	LOQ (µg/kg) in orange juice
9.92	33.1	15.2	50.7	25.6	85.3

Table 33. Repeatability and reproducibility data – Determined concentration of dimethyl phthalate in the three selected foodstuffs spiked at nominal concentrations of 420 µg/kg

Sample	Cheese	Pork mince	Orange juice
	420 µg/kg spike*	**420 µg/kg spike***	**420 µg/kg spike***
Analysis 1 replicate 1	418	351	311
Analysis 1 replicate 2	359	411	428
Analysis 1 replicate 3	332	336	423
Analysis 1 replicate 4	390	442	424
Analysis 1 replicate 5	390	415	352
Analysis 1 replicate 6	No data	387	389
Average	**378**	**390**	**388**
RSD (%)	**8.8**	**10**	**12**
Analysis 2 replicate 1	377	370	422
Analysis 2 replicate 2	444	391	361
Analysis 2 replicate 3	397	350	474
Average	**406**	**370**	**419**
Recovery (%)	**97**	**88**	**100**

* Concentrations normalised to 420 µg/kg for clarity

Table 34. Sample details for the 20 TDS samples

Fera LIMS sample number	**TDS sample number**	**Food group**	**Food type**
S08-032193	I3003578	1	Bread
S08-032194	I3003592	15	Fresh fruit
S08-032195	I3003593	16	Fruit products
S08-032196	I3003596	19	Dairy products
S08-032197	I3003585	8	Oils and fats
S08-032198	I3003595	18	Milk
S08-032199	I3003597	20	Nuts
S08-032200	I3003594	17	Beverages
S08-032201	I3003582	5	Meat products
S08-032202	I3003581	4	Offal
S08-032203	I3003588	11	Green vegetables
S08-032204	I3003586	9	Eggs
S08-032205	I3003579	2	Miscellaneous cereals
S08-032206	I3003584	7	Fish
S08-032207	I3003587	10	Sugars and preserves
S08-032208	I3003591	14	Canned vegetables
S08-032209	I3003583	6	Poultry
S08-032210	I3003580	3	Carcass meat
S08-032211	I3003590	13	Other vegetables
S08-032212	I3003589	12	Potatoes

Table 35. Phthalate diester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Dimethyl phthalate	Diethyl phthalate	Diisopropyl phthalate
S08-032193	< LOD (LOD = 6.7 µg/kg)	< LOQ (LOQ = 9.4 µg/kg)	< LOD (LOD = 1.5 µg/kg)
S08-032194	< LOD (LOD = 0.9 µg/kg)	< LOQ (LOQ = 6.8 µg/kg)	< LOD (LOD = 0.7 µg/kg)
S08-032195	< LOD (LOD = 7.5 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 4.4 µg/kg)
S08-032196	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.2 µg/kg)
S08-032197	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 20.5 µg/kg)	< LOD (LOD = 6.0 µg/kg)
S08-032198	< LOD (LOD = 18.4 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 1.6 µg/kg)
S08-032199	< LOD (LOD = 4.7 µg/kg)	< LOQ (LOQ = 10.7 µg/kg)	< LOD (LOD = 3.8 µg/kg)
S08-032200	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 0.7 µg/kg)
S08-032201	< LOD (LOD = 2.8 µg/kg)	39.9 µg/kg – failed confirmation	< LOD (LOD = 3.3 µg/kg)
S08-032202	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 3.1 µg/kg)
S08-032203	< LOD (LOD = 13.5 µg/kg)	51.8 µg/kg – failed confirmation	< LOD (LOD = 19.3 µg/kg)
S08-032204	< LOD (LOD = 9.8 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 1.5 µg/kg)
S08-032205	< LOD (LOD = 4.9 µg/kg)	13.4 µg/kg	< LOD (LOD = 1.5 µg/kg)
S08-032206	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOQ (LOQ = 10.0 µg/kg)
S08-032207	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 12.0 µg/kg)	< LOD (LOD = 4.8 µg/kg)
S08-032208	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 2.6 µg/kg)
S08-032209	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 4.9 µg/kg)
S08-032210	< LOD (LOD = 19.0 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 6.4 µg/kg)
S08-032211	< LOD (LOD = 10.3 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 3.9 µg/kg)
S08-032212	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 3.8 µg/kg)

Table 35 continued. Phthalate diester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Dially phthalate	Diisobutyl phthalate	Di-n-butyl phthalate
S08-032193	< LOD (LOD = 7.1 µ/kg)	16.6 µg/kg	16.2 µg/kg
S08-032194	< LOD (LOD = 5.2 µg/kg)	< LOQ (LOQ = 16.5 µg/kg)	< LOQ (LOQ = 14.9 µg/kg)
S08-032195	< LOD (LOD = 21.0 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOQ (LOQ = 12.1 µg/kg)
S08-032196	< LOD (LOD = 6.0 µg/kg)	< LOQ (LOQ = 8.3 µg/kg)	< LOQ (LOQ = 7.5 µg/kg)
S08-032197	< LOD (LOD = 30.5 µg/kg)	41.3 µg/kg	16.0 µg/kg
S08-032198	< LOD (LOD = 5.2 µg/kg)	< LOQ (LOQ = 5.8 µg/kg)	< LOQ (LOQ = 4.6 µg/kg)
S08-032199	< LOD (LOD = 15.8 µg/kg)	49.0 µg/kg	28.3 µg/kg
S08-032200	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 12.4 µg/kg)	2.4 µg/kg – failed confirmation
S08-032201	< LOD (LOD = 12.4 µg/kg)	18.0 µg/kg – failed confirmation	15.4 µg/kg
S08-032202	< LOD (LOD = 11.7 µg/kg)	6.4 µg/kg – failed confirmation	< LOD (LOD = 8.4 µg/kg)
S08-032203	< LOD (LOD = 34.9 µg/kg)	< LOD (LOD = 11.9 µg/kg)	< LOQ (LOQ = 15.2 µg/kg)
S08-032204	< LOD (LOD = 7.7 µg/kg)	< LOQ (LOQ = 5.9 µg/kg)	< LOQ (LOQ = 4.9 µg/kg)
S08-032205	< LOD (LOD = 5.3 µg/kg)	80.8 µg/kg	13.8 µg/kg
S08-032206	< LOD (LOD = 13.1 µg/kg)	17.6 µg/kg – failed confirmation	9.3 µg/kg
S08-032207	< LOD (LOD = 13.2 µg/kg)	< LOQ (LOQ = 13.5 µg/kg)	6.7 µg/kg – failed confirmation
S08-032208	< LOD (LOD = 7.9 µg/kg)	< LOQ (LOQ = 5.6 µg/kg)	< LOQ (LOQ = 4.6 µg/kg)
S08-032209	< LOD (LOD = 18.8 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 6.5 µg/kg)
S08-032210	< LOD (LOD = 15.7 µg/kg)	5.3 µg/kg – failed confirmation	6.3 µg/kg
S08-032211	< LOD (LOD = 10.7 µg/kg)	8.9 µg/kg – failed confirmation	3.5 µg/kg – failed confirmation
S08-032212	< LOD (LOD = 10.1 µg/kg)	< LOQ (LOQ = 11.8 µg/kg)	< LOQ (LOQ = 9.6 µg/kg)

Table 35 continued. Phthalate diester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Di-n-pentyl phthalate	Di-n-hexyl phthalate	Benzyl butyl phthalate
S08-032193	< LOQ (LOQ = 11.3 µg/kg)	< LOQ (LOQ = 16.0 µg/kg)	8.1 µg/kg
S08-032194	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOQ (LOQ = 39.7 µg/kg)
S08-032195	< LOD (LOD = 10.2 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 16.5 µg/kg)
S08-032196	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 8.9 µg/kg)
S08-032197	< LOQ (LOQ = 4.8 µg/kg)	< LOD (LOD = 23.3 µg/kg)	< LOD (LOD = 30.5 µg/kg)
S08-032198	< LOQ (LOQ = 4.9 µg/kg)	< LOD (LOD = 2.4 µg/kg)	< LOD (LOD = 6.2 µg/kg)
S08-032199	< LOD (LOD = 27.6 µg/kg)	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 10.7 µg/kg)
S08-032200	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.8 µg/kg)	< LOD (LOD = 2.0 µg/kg)
S08-032201	< LOD (LOD = 13.3 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 12.8 µg/kg)
S08-032202	< LOD (LOD = 30.4 µg/kg)	< LOD (LOD = 14.6 µg/kg)	< LOD (LOD = 15.6 µg/kg)
S08-032203	< LOD (LOD = 10.9 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 5.8 µg/kg)
S08-032204	< LOQ (LOQ = 9.8 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 9.1 µg/kg)
S08-032205	< LOD (LOD = 25.3 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOQ (LOQ = 15.5 µg/kg)
S08-032206	< LOD (LOD = 32.0 µg/kg)	< LOD (LOD = 8.3 µg/kg)	8.8 µg/kg – failed confirmation
S08-032207	< LOD (LOD = 18.5 µg/kg)	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 10.5 µg/kg)
S08-032208	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 4.7 µg/kg)
S08-032209	< LOD (LOD = 20.5 µg/kg)	< LOD (LOD = 9.0 µg/kg)	< LOD (LOD = 14.0 µg/kg)
S08-032210	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 11.2 µg/kg)
S08-032211	< LOD (LOD = 22.0 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 7.4 µg/kg)
S08-032212	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 7.5 µg/kg)

Table 35 continued. Phthalate diester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Dicyclohexyl phthalate	Di-(2-ethylhexyl) phthalate	Diheptyl phthalate
S08-032193	< LOD (LOD = 3.6 µg/kg)	124.6 µg/kg	< LOD (LOD = 9.3 µg/kg)
S08-032194	< LOD (LOD = 4.6 µg/kg)	< LOQ (LOQ = 16.6 µg/kg)	< LOD (LOD = 6.2 µg/kg)
S08-032195	< LOD (LOD = 9.3 µg/kg)	< LOQ (LOQ = 29.0 µg/kg)	< LOD (LOD = 27.6 µg/kg)
S08-032196	< LOD (LOD = 12.8 µg/kg)	140.7 µg/kg	< LOD (LOD = 21.6 µg/kg)
S08-032197	< LOD (LOD = 31.3 µg/kg)	105.5 µg/kg	< LOD (LOD = 39.6 µg/kg)
S08-032198	< LOD (LOD = 3.1 µg/kg)	< LOQ (LOQ = 29.9 µg/kg)	< LOD (LOD = 13.5 µg/kg)
S08-032199	< LOD (LOD = 12.0 µg/kg)	111.8 µg/kg	< LOD (LOD = 27.0 µg/kg)
S08-032200	< LOD (LOD = 0.8 µg/kg)	< LOQ (LOQ = 10.4 µg/kg)	< LOD (LOD = 4.9 µg/kg)
S08-032201	< LOD (LOD = 8.3 µg/kg)	329.1 µg/kg	< LOD (LOD = 36.2 µg/kg)
S08-032202	98.1 µg/kg – failed confirmation	< LOQ (LOQ = 32.4 µg/kg)	< LOD (LOD = 25.8 µg/kg)
S08-032203	< LOD (LOD = 8.1 µg/kg)	< LOQ (LOQ = 13.6 µg/kg)	< LOD (LOD = 11.5 µg/kg)
S08-032204	< LOD (LOD = 9.8 µg/kg)	< LOQ (LOQ = 42.7 µg/kg)	< LOD (LOD = 24.7 µg/kg)
S08-032205	< LOD (LOD = 3.9 µg/kg)	104.5 µg/kg	< LOD (LOD = 16.1 µg/kg)
S08-032206	7.1 µg/kg – failed confirmation	789.1 µg/kg	< LOD (LOD = 16.3 µg/kg)
S08-032207	< LOD (LOD = 6.6 µg/kg)	115.6 µg/kg	< LOD (LOD = 22.3 µg/kg)
S08-032208	< LOD (LOD = 3.7 µg/kg)	< LOQ (LOQ = 21.7 µg/kg)	< LOD (LOD = 16.6 µg/kg)
S08-032209	< LOD (LOD = 13.2 µg/kg)	322.1 µg/kg	< LOD (LOD = 37.7 µg/kg)
S08-032210	< LOD (LOD = 7.0 µg/kg)	89.7 µg/kg	< LOD (LOD = 20.1 µg/kg)
S08-032211	< LOD (LOD = 4.0 µg/kg)	34.8 µg/kg	< LOD (LOD = 26.1 µg/kg)
S08-032212	< LOD (LOD = 4.5 µg/kg)	< LOQ (LOQ = 33.2 µg/kg)	< LOD (LOD = 33.2 µg/kg)

Table 35 continued. Phthalate diester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Dioctyl phthalate	n-Octyl-n-decyl phthalate	Diisononyl phthalate
S08-032193	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 12.2 µg/kg)	< LOD (LOD = 71.9 µg/kg)
S08-032194	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 190.0 µg/kg)
S08-032195	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 142.5 µg/kg)
S08-032196	< LOD (LOD = 14.0 µg/kg)	< LOD (LOD = 19.9 µg/kg)	< LOD (LOD = 508.3 µg/kg)
S08-032197	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 649.0 µg/kg)
S08-032198	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 271.9 µg/kg)
S08-032199	< LOD (LOD = 16.8 µg/kg)	< LOD (LOD = 34.5 µg/kg)	< LOD (LOD = 309.9 µg/kg)
S08-032200	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 34.9 µg/kg)	< LOD (LOD = 42.6 µg/kg)
S08-032201	< LOD (LOD = 14.8 µg/kg)	< LOD (LOD = 22.3 µg/kg)	< LOD (LOD = 316.2 µg/kg)
S08-032202	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 128.6 µg/kg)
S08-032203	< LOQ (LOQ = 10.1 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 71.9 µg/kg)
S08-032204	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 43.8 µg/kg)	< LOD (LOD = 185.0 µg/kg)
S08-032205	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 16.9 µg/kg)	< LOD (LOD = 107.4 µg/kg)
S08-032206	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 19.2 µg/kg)	< LOD (LOD = 116.2 µg/kg)
S08-032207	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 15.7 µg/kg)	< LOD (LOD = 212.0 µg/kg)
S08-032208	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 10.6 µg/kg)	< LOD (LOD = 57.8 µg/kg)
S08-032209	< LOD (LOD = 12.8 µg/kg)	< LOD (LOD = 12.0 µg/kg)	< LOD (LOD = 259.5 µg/kg)
S08-032210	< LOD (LOD = 7.5 µg/kg)	< LOD (LOD = 13.3 µg/kg)	< LOD (LOD = 165.1 µg/kg)
S08-032211	< LOD (LOD = 8.2 µg/kg)	< LOD (LOD = 13.9 µg/kg)	< LOD (LOD = 126.5 µg/kg)
S08-032212	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 175.8 µg/kg)

Table 35 continued. Phthalate diester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Diisodecyl phthalate	Di-n-decyl phthalate
S08-032193	< LOD (LOD = 383.6 µg/kg)	< LOQ (LOQ = 19.1 µg/kg)
S08-032194	< LOD (LOD = 129.4 µg/kg)	< LOD (LOD = 5.0 µg/kg)
S08-032195	< LOD (LOD = 158.3 µg/kg)	< LOD (LOD = 8.3 µg/kg)
S08-032196	< LOD (LOD = 708.7 µg/kg)	< LOD (LOD = 33.3 µg/kg)
S08-032197	< LOD (LOD = 495.7 µg/kg)	< LOD (LOD = 24.6 µg/kg)
S08-032198	< LOD (LOD = 728.5 µg/kg)	< LOD (LOD = 11.4 µg/kg)
S08-032199	< LOD (LOD = 826.8 µg/kg)	< LOD (LOD = 38.1 µg/kg)
S08-032200	< LOD (LOD = 84.5 µg/kg)	< LOD (LOD = 1.6 µg/kg)
S08-032201	< LOD (LOD = 498.5 µg/kg)	< LOD (LOD = 22.1 µg/kg)
S08-032202	< LOD (LOD = 259.2 µg/kg)	< LOD (LOD = 8.5 µg/kg)
S08-032203	< LOD (LOD = 139.6 µg/kg)	< LOD (LOD = 2.6 µg/kg)
S08-032204	< LOD (LOD = 378.5 µg/kg)	< LOD (LOD = 18.4 µg/kg)
S08-032205	< LOD (LOD = 498.2 µg/kg)	< LOD (LOD = 9.2 µg/kg)
S08-032206	< LOD (LOD = 240.1 µg/kg)	< LOD (LOD = 7.3 µg/kg)
S08-032207	< LOD (LOD = 836.0 µg/kg)	< LOD (LOD = 17.0 µg/kg)
S08-032208	< LOD (LOD = 233.8 µg/kg)	< LOD (LOD = 6.4 µg/kg)
S08-032209	< LOD (LOD = 464.3 µg/kg)	< LOD (LOD = 19.4 µg/kg)
S08-032210	< LOD (LOD = 401.9 µg/kg)	< LOD (LOD = 11.5 µg/kg)
S08-032211	< LOD (LOD = 282.3 µg/kg)	< LOD (LOD = 9.9 µg/kg)
S08-032212	< LOD (LOD = 575.4 µg/kg)	< LOD (LOD = 14.2 µg/kg)

Table 36. Phthalate monoester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Monoisopropyl phthalate	Monoisobutyl phthalate	Mono-n-butyl phthalate
S08-032193	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032194	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032195	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032196	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032197	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032198	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)
S08-032199	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032200	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)
S08-032201	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	32 µg/kg
S08-032202	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)
S08-032203	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032204	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032205	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032206	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	52 µg/kg
S08-032207	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)
S08-032208	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032209	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 27 µg/kg)
S08-032210	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	29 µg/kg
S08-032211	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)
S08-032212	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 8.0 µg/kg)

Table 36 continued. Phthalate monoester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Mono-n-pentyl phthalate	Monocyclohexyl phthalate	Monobenzyl phthalate
S08-032193	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032194	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032195	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032196	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032197	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032198	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032199	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032200	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032201	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032202	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032203	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032204	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032205	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032206	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032207	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032208	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032209	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032210	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032211	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)
S08-032212	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 7.0 µg/kg)

Table 36 continued. Phthalate monoester concentrations measured in the 20 TDS samples

Fera LIMS sample number	Mono-(2-ethylhexyl) phthalate	Mono-n-octyl phthalate	Monoisononyl phthalate
S08-032193	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032194	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032195	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032196	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032197	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032198	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032199	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032200	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032201	< LOQ (LOQ = 50 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032202	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032203	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032204	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032205	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032206	< LOQ (LOQ = 50 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032207	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032208	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032209	54 µg/kg	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032210	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032211	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)
S08-032212	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)	< LOD (LOD = 15 µg/kg)

Table 37. Sample details and phthalic acid concentrations measured in the 20 TDS samples

Fera LIMS sample number	Phthalic acid concentration (µg/kg)
S08-032193	27
S08-032194	150 #
S08-032195	190 #
S08-032196	360 #
S08-032197	36
S08-032198	66
S08-032199	270 #
S08-032200	100
S08-032201	< LOQ (LOQ = 24 µg/kg)
S08-032202	< LOQ (LOQ = 340 µg/kg)
S08-032203	89
S08-032204	35
S08-032205	140 #
S08-032206	< LOD (LOD = 23 µg/kg)
S08-032207	110
S08-032208	84
S08-032209	130 #
S08-032210	36
S08-032211	99
S08-032212	84

Outside calibration range. Concentration indicative.

Table 38. Sample details and phthalic acid concentrations measured in the 20 TDS samples

Fera LIMS sample number	**Measured total phthalate concentration (µg/kg) #**
S08-032193	187
S08-032194	90
S08-032195	361
S08-032196	157
S08-032197	90
S08-032198	74
S08-032199	479
S08-032200	53
S08-032201	163
S08-032202	144
S08-032203	202
S08-032204	102
S08-032205	340
S08-032206	206
S08-032207	67
S08-032208	162
S08-032209	177
S08-032210	116
S08-032211	206
S08-032212	163

Calculated as dimethyl phthalate equivalents

Table 39. Sample details – phthalate source studies

LIMS number	Foodstuff	Description of packaging	Packaging size	Mass / volume of food
S09-003788	Damson jam	Glass jar, metal closure with a PVC gasket	0.946 g	340 g
S09-003789	Tomato pesto	Glass jar, metal closure with a PVC gasket	0.353 g	190 g
S09-003790	Pickled red cabbage	Glass jar, metal closure with a PVC gasket	0.432 g	710 g
S09-003791	Pudliszki goulash	Glass jar, metal closure with a PVC gasket	0.365 g	520 g
S09-003792	Bolognese sauce	Glass jar, metal closure with a PVC gasket	0.327 g	320 g
S09-003793	Tomato relish	Glass jar, metal closure with a PVC gasket	0.301 g	310 g
S09-003794	Strawberry yoghurt	Plastic container with foiled lidding	4.06 dm^2	500 g
S09-003795	Date and walnut loaf	Cartonboard tray, paper case in a plastic bag	4.12 dm^2	205 g
S09-003796	Chocolate	Paper/foil/plastic laminate	0.34 dm^2	25 g
S09-003797	Cashew nuts	Plastic bag	5.60 dm^2	300 g
S09-003798	Apricots	Plastic bag	5.60 dm^2	250 g
S09-003799	Mint sauce	Glass jar, metal closure with a PVC gasket	0.298 g	175 g
S09-003800	Green pesto	Glass jar, metal closure with a PVC gasket	0.347 g	120 g
S09-003801	Peach yoghurt	Plastic container and lid	2.30 dm^2	150 g
S09-003802	Fruit drink	Paper/foil/plastic laminate	3.00 dm^2	288 ml
S09-003803	Southern fried chicken breast	Plastic bag	11.0 dm^2	380 g
S09-003804	Ham and cheese wrap	Plastic bag in a cartonboard box	4.37 dm^2	100 g
S09-003805	Battered fish fingers	Cartonboard box	10.8 dm^2	284 g
S09-003806	Pork pie	Paper wrap	3.96 dm^2	140 g
S09-003807	Crispbreads	Cartonboard box with plastic bag liner	5.85 dm^2	150 g
S09-003808	Lasagne sheets	Cartonboard box with plastic window	6.75 dm^2	500 g
S09-003809	Tofu	Foil lined pouch	3.15 dm^2	349 g

Table 39 continued. Sample details – phthalate source studies

LIMS number	Foodstuff	Description of packaging	Packaging size	Mass / volume of food
S09-003810	Sage and onion stuffing	Cartonboard box with plastic bag liner	6.41 dm^2	100 g
S09-003811	Mulligatawny soup	Paper/plastic laminate	4.34 dm^2	600 g
S09-003812	Porridge	Cartonboard box with paper/plastic laminate bag liner	2.50 dm^2	30 g
S09-003813	Cookies	Cartonboard box with plastic window	4.66 dm^2	225 g
S09-003814	Butter	Plastic wrap	3.30 dm^2	500 g
S09-003815	Hot cross buns	Cartonbaordtray in plastic bag	3.72 dm^2	300 g
S09-003816	Raisins	Cartonboard box	1.35 dm^2	43 g

Table 40. Concentrations of the phthalates detected in the gaskets and packaging materials

LIMS code	Foodstuff	Dimethyl phthalate	Diethyl phthalate	Diisobutyl phthalate	Di-n-butyl phthalate	Di-(2-ethylhexyl) phthalate	Diisononyl phthalate	Diisodecyl phthalate
S09-003793	Tomato relish	< LOQ	< LOQ	5.2 mg/kg	< LOQ	2.8 mg/kg	10.9 mg/kg	3.9 mg/kg
S09-003794	Strawberry yoghurt	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	146.5 $\mu g/dm^2$	< LOQ
S09-003802	Fruit drink	< LOQ	< LOQ	10.5 $\mu g/dm^2$	1.9 $\mu g/dm^2$	4.0 $\mu g/dm^2$	< LOQ	< LOQ
S09-003803	Southern fried chicken breast	< LOQ	< LOQ	4.0 $\mu g/dm^2$	< LOQ	10.5 $\mu g/dm^2$	< LOQ	< LOQ
S09-003804	Ham and cheese wrap	< LOQ	< LOQ	< LOQ	< LOQ	6.8 $\mu g/dm^2$	< LOQ	< LOQ
S09-003807	Crispbreads	1.1 $\mu g/dm^2$	1.6 $\mu g/dm^2$	80.3 $\mu g/dm^2$	14.4 $\mu g/dm^2$	51.5 $\mu g/dm^2$	13.1 $\mu g/dm^2$	< LOQ
S09-003808	Lasagne sheets	1.2 $\mu g/dm^2$	< LOQ	147 $\mu g/dm^2$	3.9 $\mu g/dm^2$	18.3 $\mu g/dm^2$	< LOQ	< LOQ
S09-003809	Tofu	< LOQ	< LOQ	8.4 $\mu g/dm^2$	1.6 $\mu g/dm^2$	2.2 $\mu g/dm^2$	< LOQ	< LOQ
S09-003810	Sage and onion stuffing	< LOQ	< LOQ	15.1 $\mu g/dm^2$	2.5 $\mu g/dm^2$	14.4 $\mu g/dm^2$	< LOQ	< LOQ

Table 41. Calculated concentrations in the foods assuming 100% transfer from the gaskets and packaging materials

LIMS code	Foodstuff	Dimethyl phthalate	Diethyl phthalate	Diisobutyl phthalate	Di-n-butyl phthalate	Di-(2-ethylhexyl) phthalate	Diisononyl phthalate	Diisodecyl phthalate
S09-003793	Tomato relish	< LOQ	< LOQ	5 µg/kg	< LOQ	3 µg/kg	11 µg/kg	4 µg/kg
S09-003794	Strawberry yoghurt	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	1200 µg/kg	< LOQ
S09-003802	Fruit drink	< LOQ	< LOQ	109 µg/kg	20 µg/kg	40 µg/kg	< LOQ	< LOQ
S09-003803	Southern fried chicken breast	< LOQ	< LOQ	116 µg/kg	< LOQ	304 µg/kg	< LOQ	< LOQ
S09-003804	Ham and cheese wrap	< LOQ	< LOQ	< LOQ	< LOQ	212 µg/kg	< LOQ	< LOQ
S09-003807	Crispbreads	43 µg/kg	63 µg/kg	3139 µg/kg	563 µg/kg	2013 µg/kg	512 µg/kg	< LOQ
S09-003808	Lasagne sheets	16 µg/kg	< LOQ	1961 µg/kg	52 µg/kg	244 µg/kg	< LOQ	< LOQ
S09-003809	Tofu	< LOQ	< LOQ	76 µg/kg	14 µg/kg	20 µg/kg	< LOQ	< LOQ
S09-003810	Sage and onion stuffing	< LOQ	< LOQ	967 µg/kg	160 µg/kg	922 µg/kg	< LOQ	< LOQ

Table 42. Measured diisobutyl phthalate concentrations in the lasagne sheets taken at different distances from the packaging

Sample	Layer 1 0 – 0.3 cm from the packaging	Layer 2 0.3 – 0.6 cm from the packaging	Layer 3 2.0 – 2.3 cm from the packaging
Sample 1	223	105	86
Sample 2	259	111	35
Sample 3	206	104	63

Table 43. Retail food sample details

Sample code	Product description
S09-017239	Scottish mussels in shell
S09-017240	Ox liver
S09-017241	Breast of lamb
S09-017242	Granary loaf
S09-017243	Sea bass fillets
S09-017244	Salmon fillets
S09-017245	Jersey milk
S09-017246	British double cream
S09-017247	Lard
S09-017248	British Wiltshire cured thick cut gammon steak
S09-017249	Lasagne
S09-017250	Blue Stilton
S09-017251	British lean beef steak mince (12% fat)
S09-017252	Chocolate sponge sandwich
S09-017253	Large free range eggs
S09-017254	Fisherman's bake
S09-017255	Cornflakes
S09-017256	Infant milk - ready to use
S09-017257	Apple, peach & mango fruit pots
S09-017258	Bourbon creams
S09-017259	White bread rolls
S09-017260	Conger eel
S09-017261	Shin of beef
S09-017262	Pigs liver
S09-017263	Diced lamb
S09-017264	Boneless chicken breasts
S09-017265	Calves liver - sliced
S09-017266	Red mullet
S09-017267	Lemon sole
S09-017268	Sprats
S09-017269	Dairy butter
S09-017270	Cornish clotted cream
S09-017271	Traditional Normandy camembert
S09-017272	Goose fat
S09-017273	Ripple roll
S09-017274	Rice pudding
S09-017275	Ginger biscuits
S09-017276	Organic extra virgin olive oil

Table 43 continued. Retail food sample details

Sample code	Product description
S09-017277	Fish pie
S09-017278	Lasagne
S09-017279	Fresh filtered whole milk
S09-017280	Greek style natural probiotic yoghurt
S09-017281	Jumbo salted cod cutlets
S09-017282	Matzos
S09-017283	Egg pasta lasagne
S09-017284	Mackerel fillets in oil
S09-017285	Infant milk - ready to use
S09-017286	Olive pomace oil blend
S09-017287	Lasagne
S09-017288	Organic cod fish fingers
S09-017289	Rosemary & garlic lamb chipolatas
S09-017290	Unsalted butter
S09-017291	Large roast chicken - whole
S09-017292	Belgian biscuit collection
S09-017293	Wholemeal pitta
S09-017294	Wheat cereal
S09-017295	Grapeseed oil
S09-017296	Organic follow-on milk – ready to use
S09-017297	Lasagne
S09-017298	Vegetable fat spread
S09-017299	New Zealand lamb offal - liver
S09-017300	Neck cutlets
S09-017301	Chicken thighs - bone-in/ skin on
S09-017302	Beef topside
S09-017303	Pork chops - bone-in/ skin on
S09-017304	Welsh lambs liver
S09-017305	Welsh breakfast sausage
S09-017306	Gingerbread ghosties
S09-017307	Brown bread
S09-017308	Farmhouse fruit cake
S09-017309	Fresh eggs - 12 Class A mixed weight
S09-017310	Wild local caught trout fillets
S09-017311	Lamb mince
S09-017312	Turkey breast roast
S09-017313	Swordfish
S09-017314	Fresh baby hake

Table 43 continued. Retail food sample details

Sample code	Product description
S09-017315	Pure vegetable oil
S09-017316	Infant milk - ready to use
S09-017317	Diced chicken
S09-017318	Lemon curd yogurt
S09-017319	Duck eggs
S09-017320	Free range chicken
S09-017321	Rolled pork shoulder joint
S09-017322	Beer & treacle back bacon
S09-017323	Pigs liver
S09-017324	Dressed crab
S09-017325	Sprats (large)
S09-017326	Bloater
S09-017327	Whole smoked eel
S09-017328	Lorne sausage
S09-017329	Smoked ham shank
S09-017330	Refined vegetable fat
S09-017331	Infant milk - formula
S09-017332	Half spring chicken
S09-017333	Tilapia
S09-017334	Grated Emmental
S09-017335	Fresh tuna
S09-017336	Salmon fillets
S09-017337	Halal chicken liver
S09-017338	Halal lamb carcase pieces
S09-017339	Fresh British pork leg joint
S09-017340	Salmon in dill sauce - frozen steam meal
S09-017341	British chicken livers
S09-017342	Whitebait IQF
S09-017343	Beef dripping
S09-017344	Duck eggs
S09-017345	Microwaveable porage oats - syrup swirl
S09-017346	Rye wholemeal bread
S09-017347	Milk brioche rolls
S09-017348	Brie
S09-017349	Sirloin steaks
S09-017350	Pork sausage
S09-017351	Lamb loin chops
S09-017352	Pecan & maple crisp

Table 43 continued. Retail food sample details

Sample code	Product description
S09-017353	De-luxe muesli
S09-017354	Craster kipper fillets
S09-017355	Salmon
S09-017356	Cream crackers
S09-017357	Sunflower oil
S09-017358	Six fresh barn eggs - medium, class A
S09-017359	Turkey breast steaks
S09-017360	Wild Alaskan salmon fillets
S09-017361	Fillets of anchovies
S09-017362	Lamb hotpot
S09-017363	Rice cereal
S09-017364	Angel slices
S09-017365	Growing up milk for toddlers – ready to use
S09-017366	Cookies
S09-017367	Cauliflower & broccoli cheese
S09-017368	Free range eggs - large
S09-017369	Wild fallow venison liver
S09-017370	Belly of pork (rind on)
S09-017371	Iced fairy cakes
S09-017372	Wild pink salmon
S09-017373	Scottish smoked peppered mackerel
S09-017374	Sausages
S09-017375	Prime gammon joint - unsmoked
S09-017376	Naturally smoked haddock fillets
S09-017377	Spare rib of pork joint
S09-017378	Smoked salmon slices
S09-017379	Extra thick real cream - UHT
S09-017380	Cereal
S09-017381	Infant milk - formula
S09-017382	Organic wholemeal bread
S09-017383	Goat milk nutrition
S09-017384	Wild Alaskan salmon fillets
S09-017385	Soya infant formula
S09-017386	Turkey
S09-017387	Turkey breast butter basted
S09-017388	Organic free range British eggs - mixed weight
S09-017389	Sliced white plain bread
S09-017390	Beef sausages

Table 43 continued. Retail food sample details

Sample code	Product description
S09-017391	2 River cobbler fillets
S09-017392	Garnished turkey drumsticks
S09-017393	Eye roast joint
S09-017394	Frying steak
S09-017395	Herring fillets
S09-017396	Boneless pork chops
S09-017397	Boneless stuffed chicken
S09-017398	Trout fillet
S09-017399	Streaky bacon
S09-017400	Organic Irish oak smoked salmon
S09-017401	Traditionally smoked mackerel fillets
S09-017402	Raspberry and coconut cakes
S09-017403	Soya infant formula
S09-017404	Toasted sesame oil
S09-017405	South Pacific sardines in tomato sauce
S09-017406	Infant milk - formula
S09-017407	Honey cereal
S09-017408	Halal bone-in chicken thigh pieces
S09-017409	British pork mince
S09-017410	Organic bone-in lamb shoulder roast
S09-017411	Organic beef stew with potatoes
S09-017412	Strawberry dairy dessert
S09-017413	Groundnut oil
S09-017414	Infant milk - formula
S09-017415	Rye crispbread
S09-017416	Organic gingerbread men
S09-017417	Iced cake
S09-017418	Organic infant milk - formula
S09-017419	Cottage pie
S09-017420	Halibut
S09-017421	Trout
S09-017422	Whitebait
S09-017423	Trout
S09-017424	Fruity malt loaf - sliced
S09-017425	Follow-on milk - ready to use
S09-017426	Breaded chicken pieces
S09-017427	Salmon en croute
S09-017428	Wątróbki rybne - cod liver in brine

Table 43 continued. Retail food sample details

Sample code	Product description
S09-017429	Follow-on milk - ready to use
S09-017430	Infant milk - formula
S09-017431	Biscotti
S09-017432	Wild fallow venison liver
S09-017433	Free range belly pork
S09-017434	Yellowfin tuna
S09-017435	Mackerel fillets
S09-017436	Mackerel
S09-017437	Organic whole milk
S09-017438	Sardines
S09-017439	British turkey mince
S09-017440	Foie de lotte (monkfish liver)
S09-017441	Dressed crab
S09-017442	Kipper fillets
S09-017443	Sea bream
S09-017444	Herrings
S09-017445	Red sea bream
S09-017446	Farmed sea bass
S09-017447	Mackerel
S09-017448	Lard
S09-017449	Pork sausages
S09-017450	English minced lamb
S09-017451	Turkey leg
S09-017452	Boneless leg of pork
S09-017453	Whole herring
S09-017454	Organic whole milk
S09-017455	Sardines
S09-017456	Whole kippers
S09-017457	Organic British free range chicken thigh fillets
S09-017458	British turkey lean mince
S09-017459	Extra virgin olive oil
S09-017460	Classic pork sausage - 16
S09-017461	New Zealand farm assured lamb mince
S09-017462	Extra virgin olive oil
S09-017463	Lard
S09-017464	Free range turkey breast mince
S09-017465	Welsh farm assured whole milk
S09-017466	Dressed crab

Table 43 continued. Retail food sample details

Sample code	Product description
S09-017467	Mackerel
S09-017468	IQF whitebait
S09-017469	Whitebait
S09-017470	Minced lamb
S09-017471	Minced beef
S09-017472	Boneless leg of pork
S09-017473	Minced beef
S09-017474	Salted butter
S09-017475	Newmarket sausages
S09-017476	Farmed sea bass fillets
S09-017477	Lard
S09-017478	Free range chicken legs
S09-017479	Turkey mince
S09-017480	Boneless leg of pork
S09-017481	Farmhouse butter
S09-017482	Whole milk
S09-017483	Fresh sardines
S09-017484	Blended olive pomace oil
S09-017485	Salmon steaks
S09-017486	Sardines – fresh
S09-017487	Smoked mackerel
S09-017488	Sardines
S09-017489	Olive pomace oil
S09-017490	Smoked salmon
S09-017491	Hebridean white crab meat
S09-017492	Salmon
S09-017493	Smoked mackerel
S09-017494	Minced beef
S09-017495	Smoked salmon
S09-017496	British turkey drumsticks
S09-017497	Butter ghee
S09-017498	Wild salmon steaks
S09-017499	Wild Solway salmon

Table 44. Retail food sample results

	S09-017239	S09-017240	S09-017241	S09-017242	S09-017243
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 6.0 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 1.6 µg/kg)
DiPP	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 8.9 µg/kg)	< LOQ (LOQ = 3.3 µg/kg)	< LOD (LOD = 1.2 µg/kg)
DAP	< LOD (LOD = 14.6 µg/kg)	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 40.3 µg/kg)	< LOD (LOD = 13.4 µg/kg)	< LOD (LOD = 4.1 µg/kg)
DiBP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 33.2 µg/kg)	< LOD (LOD = 33.2 µg/kg)	67.4 µg/kg	< LOD (LOD = 33.2 µg/kg)
DBP	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 24.6 µg/kg)	< LOD (LOD = 24.6 µg/kg)	< LOQ (LOQ = 26.6 µg/kg)	< LOD (LOD = 24.6 µg/kg)
DPP	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 4.6 µg/kg)	6.0 µg/kg (not confirmed)	< LOD (LOD = 0.8 µg/kg)
DHP	56.4 µg/kg (not confirmed)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 6.9 µg/kg)	11.2 µg/kg (not confirmed)	< LOD (LOD = 2.8 µg/kg)
BBP	< LOD (LOD = 27.9 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 15.1 µg/kg)	< LOD (LOD = 8.6 µg/kg)	< LOD (LOD = 5.2 µg/kg)
DCHP	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 4.4 µg/kg)
DEHP	< LOQ (LOQ = 27.2 µg/kg)	< LOD (LOD = 26.3 µg/kg)	< LOQ (LOQ = 87.6 µg/kg)	< LOQ (LOQ = 92.1 µg/kg)	< LOQ (LOQ = 87.6 µg/kg)
DOP	< LOD (LOD = 12.2 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 30.1 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 6.0 µg/kg)
DiNP	< LOD (LOD = 285.8 µg/kg)	< LOD (LOD = 157.1 µg/kg)	< LOD (LOD = 358.2 µg/kg)	< LOD (LOD = 164.2 µg/kg)	< LOD (LOD = 449.7 µg/kg)
DiDP	< LOD (LOD = 98.9 µg/kg)	< LOD (LOD = 76.5 µg/kg)	< LOD (LOD = 257.3 µg/kg)	< LOD (LOD = 101.0 µg/kg)	< LOD (LOD = 273.6 µg/kg)
DDP	< LOD (LOD = 12.5 µg/kg)	< LOD (LOD = 15.4 µg/kg)	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 15.1 µg/kg)	< LOD (LOD = 13.0 µg/kg)

Table 44 continued. Retail food sample results

	S09-017244	S09-017245	S09-017246	S09-017247	S09-017248
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 2.3 µg/kg)
DEP	< LOD (LOD =2.9 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOQ (LOQ = 15.2 µg/kg)	< LOD (LOD = 1.9 µg/kg)
DiPP	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 1.4 µg/kg)
DAP	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD =12.9 µg/kg)	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 5.4 µg/kg)
DiBP	< LOD (LOD = 33.2 µg/kg)	< LOD (LOD = 33.2 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOQ (LOQ = 40.4 µg/kg)	< LOD (LOD = 12.1 µg/kg)
DBP	< LOD (LOD = 24.6 µg/kg)	< LOD (LOD = 24.6 µg/kg)	< LOQ (LOQ = 50.4 µg/kg)	141.0 µg/kg	< LOD (LOD = 15.1 µg/kg)
DPP	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 16.9 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 2.0 µg/kg)
DHP	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 20.8 µg/kg)	< LOD (LOD = 13.9 µg/kg)	< LOD (LOD = 2.7 µg/kg)
BBP	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 28.7 µg/kg)	< LOD (LOD = 17.3 µg/kg)	< LOD (LOD = 7.9 µg/kg)
DCHP	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 42.6 µg/kg)	< LOD (LOD = 21.3 µg/kg)	< LOD (LOD = 5.9 µg/kg)
DEHP	< LOD (LOD = 26.3 µg/kg)	108.6 µg/kg	327.9 µg/kg	118.9 µg/kg	< LOQ (LOQ = 93.3 µg/kg)
DOP	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 37.8 µg/kg)	< LOD (LOD = 40.2 µg/kg)	< LOD (LOD = 15.0 µg/kg)
DiNP	< LOD (LOD = 372.0 µg/kg)	< LOD (LOD = 360.6 µg/kg)	< LOD (LOD = 631.7 µg/kg)	< LOD (LOD = 4222.6 µg/kg)	< LOD (LOD = 770.9 µg/kg)
DiDP	< LOD (LOD = 387.7 µg/kg)	< LOD (LOD = 156.2 µg/kg)	< LOD (LOD = 483.8 µg/kg)	< LOD (LOD = 4293.9 µg/kg)	< LOD (LOD = 533.3 µg/kg)
DDP	< LOD (LOD = 13.3 µg/kg)	< LOD (LOD = 51.8 µg/kg)	< LOD (LOD = 67.7 µg/kg)	< LOD (LOD = 175.7 µg/kg)	< LOD (LOD = 10.0 µg/kg)

Table 44 continued. Retail food sample results

	S09-017249	**S09-017250**	**S09-017251**	**S09-017252**	**S09-017253**
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 12.0 µg/kg)
DEP	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 15.7 µg/kg)
DiPP	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 6.5 µg/kg)
DAP	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 8.5 µg/kg)	34.3 µg/kg (not confirmed)	< LOD (LOD = 37.0 µg/kg)
DiBP	< LOD (LOD = 19.0 µg/kg)	< LOQ (LOQ = 40.4 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 19.0 µg/kg)	< LOD (LOD = 19.0 µg/kg)
DBP	< LOD (LOD = 19.5 µg/kg)	< LOD (LOD = 15.1 µg/kg)	< LOQ (LOQ = 50.4 µg/kg)	< LOD (LOD = 19.5 µg/kg)	< LOD (LOD = 19.5 µg/kg)
DPP	< LOD (LOD = 1.78 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 4.0 µg/kg)
DHP	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 15.2 µg/kg)	< LOD (LOD = 6.6 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 4.1 µg/kg)
BBP	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 32.8 µg/kg)	< LOD (LOD = 12.7 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 10.8 µg/kg)
DCHP	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 24.5	< LOD (LOD = 10.3 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 5.5 µg/kg)
DEHP	< LOQ (LOQ = 81.7 µg/kg)	496.3 µg/kg	255.8 µg/kg	< LOQ (LOQ = 81.7 µg/kg)	< LOD (LOD = 24.5 µg/kg)
DOP	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 15.3 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 4.3 µg/kg)
DiNP	< LOD (LOD = 121.9 µg/kg)	3055.4 µg/kg same profile	< LOD (LOD = 877.7 µg/kg)	< LOD (LOD = 204.4 µg/kg)	< LOD (LOD = 357.7 µg/kg)
DiDP	< LOD (LOD = 88.0 µg/kg)	< LOD (LOD = 277.8 µg/kg)	< LOD (LOD = 722.6 µg/kg)	< LOD (LOD = 83.3 µg/kg)	< LOD (LOD = 253.0 µg/kg)
DDP	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 34.5 µg/kg)	< LOD (LOD = 31.1 µg/kg)	< LOD (LOD = 11.8 µg/kg)	< LOD (LOD = 11.0 µg/kg)

Table 44 continued. Retail food sample results

	S09-017254	S09-017255	S09-017256	S09-017257	S09-017258
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 15.6 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 24.5 µg/kg)
DEP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 13.7 µg/kg)	< LOD (LOD = 34.7 µg/kg)	< LOD (LOD = 13.8 µg/kg)	< LOD (LOD = 17.5 µg/kg)
DiPP	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 30.5 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 13.4 µg/kg)
DAP	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 43.9 µg/kg)	< LOD (LOD = 107.3 µg/kg)	< LOD (LOD = 35.7 µg/kg)	< LOD (LOD = 50.3 µg/kg)
DiBP	< LOQ (LOQ = 10.2 µg/kg)	< LOQ (LOQ = 63.5 µg/kg)	< LOD (LOD = 19.0 µg/kg)	< LOD (LOD = 19.0 µg/kg)	< LOQ (LOQ = 43.1 µg/kg)
DBP	< LOQ (LOQ = 17.3 µg/kg)	< LOD (LOD = 19.5 µg/kg)	< LOD (LOD = 19.5 µg/kg)	< LOD (LOD = 19.5 µg/kg)	59.7 µg/kg
DPP	No data	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 22.0 µg/kg)	< LOD (LOD = 5.6 µg/kg)	No data
DHP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 23.5 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 24.8 µg/kg)
BBP	< LOD (LOD = 5.9 µg/kg)	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 36.3 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 51.1 µg/kg)
DCHP	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 21.1 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 35.1 µg/kg)
DEHP	< LOQ (LOQ = 42.3 µg/kg)	< LOD (LOD = 24.5 µg/kg)	< LOQ (LOQ = 81.7 µg/kg)	< LOD (LOD = 24.5 µg/kg)	< LOQ (LOQ = 138.4 µg/kg)
DOP	< LOD (LOD = 8.1 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 77.2 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 86.8 µg/kg)
DiNP	< LOD (LOD = 155.9 µg/kg)	< LOD (LOD = 198.9 µg/kg)	< LOD (LOD = 806.6 µg/kg)	< LOD (LOD = 102.3 µg/kg)	< LOQ (LOQ = 7042.6 µg/kg)
DiDP	< LOD (LOD = 136.7 µg/kg)	< LOD (LOD = 89.9 µg/kg)	< LOD (LOD = 1687.3 µg/kg)	< LOD (LOD = 110.1 µg/kg)	< LOD (LOD = 1639.2 µg/kg)
DDP	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 58.6 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 115.5 µg/kg)

Table 44 continued. Retail food sample results

	S09-017259	S09-017260	S09-017261	S09-017262	S09-017263
DMP	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 1.5 µg/kg)
DEP	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 7.2 µg/kg)
DiPP	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 3.8 µg/kg)
DAP	< LOD (LOD = 23.0 µg/kg)	< LOD (LOD =10.4 µg/kg)	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD =14.5 µg/kg)	< LOD (LOD = 16.1 µg/kg)
DiBP	16.7 µg/kg	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 2.7 µg/kg)
DBP	< LOQ (LOQ = 12.0 µg/kg)	< LOQ (LOQ = 23.2 µg/kg)	< LOQ (LOQ = 6.7 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 2.5 µg/kg)
DPP	No data	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 1.9 µg/kg)
DHP	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 2.3 µg/kg)
BBP	< LOD (LOD = 22.7 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 8.6 µg/kg)
DCHP	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 5.0 µg/kg)
DEHP	92.3 µg/kg	< LOQ (LOQ = 94.9 µg/kg)	< LOQ (LOQ = 27.2 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOQ (LOQ = 27.2 µg/kg)
DOP	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 13.4 µg/kg)
DiNP	< LOD (LOD = 186.4 µg/kg)	< LOD (LOD = 104.0 µg/kg)	< LOD (LOD = 170.1 µg/kg)	< LOD (LOD = 76.9 µg/kg)	< LOD (LOD = 108.3 µg/kg)
DiDP	< LOD (LOD = 62.2 µg/kg)	< LOD (LOD = 58.6 µg/kg)	< LOD (LOD = 58.2 µg/kg)	< LOD (LOD = 42.8 µg/kg)	< LOD (LOD = 43.9 µg/kg)
DDP	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 5.1 µg/kg)

Table 44 continued. Retail food sample results

	S09-017264	S09-017265	S09-017266	S09-017267	S09-017268
DMP	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.53 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 3.8 µg/kg)
DEP	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOQ (LOQ = 17.5 µg/kg)	< LOD (LOD = 8.6 µg/kg)
DiPP	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOQ (LOQ = 11.0 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 5.9 µg/kg)
DAP	< LOD (LOD =11.9 µg/kg)	< LOD (LOD =18.9 µg/kg)	< LOD (LOD = 14.3 µg/kg)	< LOD (LOD = 16.7 µg/kg)	< LOD (LOD =18.8 µg/kg)
DiBP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD =2.8 µg/kg)	< LOQ (LOQ = 7.5 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 3.3 µg/kg)
DBP	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 7.0 µg/kg)
DPP	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 2.4 µg/kg)	< LOD (LOD = 4.3 µg/kg)
DHP	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 4.2 µg/kg)
BBP	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 6.7 µg/kg)
DCHP	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 8.8 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 5.4 µg/kg)
DEHP	< LOD (LOD = 28.5 µg/kg)	< LOD (LOD = 19.3 µg/kg)	< LOQ (LOQ = 64.24 µg/kg)	< LOD (LOD = 19.3 µg/kg)	< LOD (LOD = 28.5 µg/kg)
DOP	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 10.3 µg/kg)	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 2.0 µg/kg)
DiNP	< LOD (LOD = 126.5 µg/kg)	< LOD (LOD = 86.9 µg/kg)	< LOD (LOD = 495.4 µg/kg)	< LOD (LOD =175.7 µg/kg)	< LOD (LOD = 86.9 µg/kg)
DiDP	< LOD (LOD = 50.4 µg/kg)	< LOD (LOD = 72.4 µg/kg)	< LOD (LOD = 319.7 µg/kg)	< LOD (LOD = 209.1 µg/kg)	< LOD (LOD = 72.4 µg/kg)
DDP	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 18.4 µg/kg)	< LOD (LOD = 9.9 µg/kg)	< LOD (LOD = 1.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017269	**S09-017270**	**S09-017271**	**S09-017272**	**S09-017273**
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 12.7 µg/kg)	< LOD (LOD = 2.4 µg/kg)
DiPP	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 11.4 µg/kg)	< LOD (LOD = 1.2 µg/kg)
DAP	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 21.6 µg/kg)	< LOD (LOD =8.8 µg/kg)	< LOD (LOD = 30.9 µg/kg)	< LOD (LOD = 4.2 µg/kg)
DiBP	23.6 µg/kg (not confirmed)	186.0 µg/kg	< LOD (LOD = 3.3 µg/kg)	29.8 µg/kg (not confirmed)	< LOQ (LOQ = 10.2 µg/kg)
DBP	< LOQ (LOQ = 8.2 µg/kg)	< LOQ (LOQ = 27.6 µg/kg)	< LOD (LOD = 7.0 µg/kg)	18.1 µg/kg (not confirmed)	< LOD (LOD = 5.2 µg/kg)
DPP	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 3.1 µg/kg)	No data	No data
DHP	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 50.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)
BBP	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 8.8 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 36.6 µg/kg)	< LOD (LOD = 6.8 µg/kg)
DCHP	< LOD (LOD = 11.4 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 59.1 µg/kg)	< LOD (LOD = 11.7 µg/kg)
DEHP	346.6 µg/kg	690.0 µg/kg	< LOQ (LOQ = 94.9 µg/kg)	128.7 µg/kg (not confirmed)	133.8 µg/kg (not confirmed)
DOP	< LOD (LOD = 50.2 µg/kg)	< LOD (LOD = 27.4 µg/kg)	< LOD (LOD = 12.7 µg/kg)	< LOD (LOD = 321.1 µg/kg)	< LOD (LOD = 86.1 µg/kg)
DiNP	1499.6 µg/kg (same profile)	< LOD (LOD = 1097.7 µg/kg)	< LOD (LOD = 642.1 µg/kg)	< LOD (LOD = 6216.3 µg/kg)	< LOQ (LOQ = 707.5 µg/kg)
DiDP	< LOD (LOD = 914.5 µg/kg)	< LOD (LOD = 517.2 µg/kg)	No data	< LOD (LOD = 4783.6 µg/kg)	< LOD (LOD = 323.5 µg/kg)
DDP	< LOD (LOD = 91.5 µg/kg)	< LOD (LOD = 132.3 µg/kg)	No data	< LOD (LOD = 515.1 µg/kg)	< LOD (LOD = 12.6 µg/kg)

Table 44 continued. Retail food sample results

	S09-017274	S09-017275	S09-017276	S09-017277	S09-017278
DMP	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 5.3 µg/kg)	5.5 µg/kg (not confirmed)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.4 µg/kg)
DEP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 4.2 µg/kg)
DiPP	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 2.8 µg/kg)
DAP	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 19.3 µg/kg)	< LOD (LOD = 7.7 µg/kg)	< LOD (LOD =10.29 µg/kg)	< LOD (LOD = 11.4 µg/kg)
DiBP	< LOQ (LOQ = 10.2 µg/kg)	15.8 µg/kg (not confirmed)	49.4 µg/kg	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 2.8 µg/kg)
DBP	< LOQ (LOQ = 17.3 µg/kg)	< LOQ (LOQ = 19.3 µg/kg)	56.8 µg/kg	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 8.3 µg/kg)
DPP	< LOD (LOD = 10.1 µg/kg)	No data	< LOD (LOD = 13.1 µg/kg)	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 1.8 µg/kg)
DHP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 13.9 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 1.9 µg/kg)
BBP	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 23.6 µg/kg)	2083.8 µg/kg	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 4.1 µg/kg)
DCHP	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 18.4 µg/kg)	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 2.4 µg/kg)
DEHP	57.4 µg/kg	109.8 µg/kg	6446.8 µg/kg	< LOD (LOD = 28.5 µg/kg)	< LOD (LOD = 19.3 µg/kg)
DOP	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 14.6 µg/kg)	< LOD (LOD = 26.8 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 1.9 µg/kg)
DiNP	< LOD (LOD = 354.9 µg/kg)	< LOD (LOD = 491.0 µg/kg)	< LOD (LOD = 4752.3 µg/kg)	< LOD (LOD = 93.7 µg/kg)	< LOD (LOD = 81.2 µg/kg)
DiDP	< LOD (LOD = 199.0 µg/kg)	< LOD (LOD = 461.5 µg/kg)	< LOD (LOD = 1967.0 µg/kg)	< LOD (LOD = 67.4 µg/kg)	< LOD (LOD = 51.7 µg/kg)
DDP	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 27.8 µg/kg)	< LOD (LOD = 17.5 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 1.9 µg/kg)

Table 44 continued. Retail food sample results

	S09-017279	S09-017280	S09-017281	S09-017282	S09-017283
DMP	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 1.5 µg/kg)
DEP	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 4.7 µg/kg)
DiPP	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 3.3 µg/kg)
DAP	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 8.4 µg/kg)
DiBP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 3.0 µg/kg)	29.9 µg/kg
DBP	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOQ (LOQ = 17.3 µg/kg)
DPP	< LOD (LOD = 2.7 µg/kg)	No data	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 1.4 µg/kg)
DHP	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 4.8 µg/kg)
BBP	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 19.1 µg/kg)	< LOD (LOD = 13.0 µg/kg)	< LOD (LOD = 9.3 µg/kg)
DCHP	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 6.2 µg/kg)
DEHP	< LOQ (LOQ = 51.4 µg/kg)	78.1 µg/kg	< LOQ (LOQ = 42.3 µg/kg)	< LOQ (LOQ = 42.3 µg/kg)	< LOQ (LOQ = 42.3 µg/kg)
DOP	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 28.2 µg/kg)	< LOD (LOD = 16.1 µg/kg)	< LOD (LOD = 23.5 µg/kg)	< LOD (LOD = 8.6 µg/kg)
DiNP	< LOD (LOD = 291.1 µg/kg)	< LOD (LOD = 368.2 µg/kg)	< LOD (LOD = 194.4 µg/kg)	< LOD (LOD = 229.6 µg/kg)	< LOQ (LOQ = 775.2 µg/kg)
DiDP	< LOD (LOD = 210.5 µg/kg)	< LOD (LOD = 478.4 µg/kg)	< LOD (LOD = 145.8 µg/kg)	< LOD (LOD = 136.4 µg/kg)	< LOD (LOD = 80.4 µg/kg)
DDP	< LOD (LOD = 24.2 µg/kg)	< LOD (LOD = 48.4 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 19.9 µg/kg)	< LOD (LOD = 45.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017284	S09-017285	S09-017286	S09-017287	S09-017288
DMP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 3.0 µg/kg)
DEP	< LOQ (LOQ = 11.5 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 5.6 µg/kg)
DiPP	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 4.0 µg/kg)
DAP	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 14.6 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 12.0 µg/kg)
DiBP	< LOQ (LOQ = 7.4 µg/kg)	< LOQ (LOQ = 7.4 µg/kg)	15.3 µg/kg (not confirmed)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 5.8 µg/kg)
DBP	< LOQ (LOQ = 12.4 µg/kg)	< LOQ (LOQ = 12.4 µg/kg)	18.0 µg/kg (not confirmed)	< LOD (LOD = 3.7 µg/kg)	< LOQ (LOQ = 18.0 µg/kg)
DPP	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 4.0 µg/kg)	< LOQ (LOQ = 48.1 µg/kg)	< LOD (LOD = 0.7 µg/kg)	No data
DHP	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 54.0 µg/kg)	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 10.5 µg/kg)
BBP	< LOD (LOD = 13.4 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 43.5 µg/kg)	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 16.3 µg/kg)
DCHP	No data	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 17.8 µg/kg)	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 12.5 µg/kg)
DEHP	84.8 µg/kg	< LOQ (LOQ = 49.9 µg/kg)	433.6 µg/kg	< LOD (LOD = 15.0 µg/kg)	57.2 µg/kg (not confirmed)
DOP	< LOD (LOD = 80.0 µg/kg)	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 78.0 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 23.4 µg/kg)
DiNP	< LOD (LOD = 478.8 µg/kg)	< LOD (LOD = 354.2 µg/kg)	< LOD (LOD = 1432.8 µg/kg)	< LOD (LOD = 85.7 µg/kg)	< LOD (LOD = 698.0 µg/kg)
DiDP	< LOD (LOD = 629.8 µg/kg)	< LOD (LOD = 346.1 µg/kg)	< LOD (LOD = 1391.6 µg/kg)	< LOD (LOD = 43.1 µg/kg)	< LOD (LOD = 355.9 µg/kg)
DDP	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 13.9 µg/kg)	< LOD (LOD = 82.3 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 31.6 µg/kg)

Table 44 continued. Retail food sample results

	S09-017289	**S09-017290**	**S09-017291**	**S09-017292**	**S09-017293**
DMP	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 3.8 µg/kg)
DEP	< LOQ (LOQ = 18.7 µg/kg)	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 6.5 µg/kg)
DiPP	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 4.9 µg/kg)
DAP	< LOD (LOD = 14.7 µg/kg)	< LOD (LOD = 22.9 µg/kg)	< LOD (LOD = 16.9 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 15.3 µg/kg)
DiBP	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOQ (LOQ = 19.3 µg/kg)	< LOD (LOD = 5.8 µg/kg)
DBP	< LOQ (LOQ = 11.4 µg/kg)	< LOQ (LOQ = 24.9 µg/kg)	< LOQ (LOQ = 11.4 µg/kg)	< LOQ (LOQ = 15.5 µg/kg)	< LOQ (LOQ = 12.9 µg/kg)
DPP	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 4.4 µg/kg)	No data	No data
DHP	< LOQ (LOQ = 30.9 µg/kg)	< LOD (LOD = 18.2 µg/kg)	< LOD (LOD = 9.1 µg/kg)	< LOD (LOD = 14.9 µg/kg)	< LOD (LOD = 11.7 µg/kg)
BBP	< LOD (LOD = 16.6 µg/kg)	< LOD (LOD = 20.8 µg/kg)	< LOD (LOD = 15.5 µg/kg)	< LOD (LOD = 18.2 µg/kg)	< LOD (LOD = 22.7 µg/kg)
DCHP	< LOD (LOD = 12.8 µg/kg)	< LOD (LOD = 34.6 µg/kg)	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 18.3 µg/kg)	< LOD (LOD = 18.8 µg/kg)
DEHP	163.1 µg/kg	2591.5 µg/kg	196.5 µg/kg	218.5 µg/kg	93.7 µg/kg
DOP	< LOD (LOD = 17.9 µg/kg)	< LOD (LOD = 44.4 µg/kg)	< LOD (LOD = 49.2 µg/kg)	< LOD (LOD = 25.8 µg/kg)	< LOD (LOD = 12.9 µg/kg)
DiNP	635.7 µg/kg	< LOD (LOD = 2742.7 µg/kg)	< LOD (LOD = 450.6 µg/kg)	< LOD (LOD = 710.5 µg/kg)	< LOD (LOD = 474.9 µg/kg)
DiDP	< LOD (LOD = 441.6 µg/kg)	< LOD (LOD = 2714.3 µg/kg)	< LOD (LOD = 359.4 µg/kg)	< LOD (LOD = 832.0 µg/kg)	< LOD (LOD = 434.2 µg/kg)
DDP	< LOD (LOD = 23.2 µg/kg)	< LOD (LOD = 93.6 µg/kg)	< LOD (LOD = 72.3 µg/kg)	< LOD (LOD = 94.5 µg/kg)	< LOD (LOD = 32.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017294	S09-017295	S09-017296	S09-017297	S09-017298
DMP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)
DEP	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 11.5 µg/kg)
DiPP	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 9.7 µg/kg)
DAP	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 7.5 µg/kg)	< LOD (LOD = 29.3 µg/kg)	< LOD (LOD = 8.2 µg/kg)	< LOD (LOD = 27.7 µg/kg)
DiBP	< LOQ (LOQ = 10.2 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 10.4 µg/kg)
DBP	< LOQ (LOQ = 17.3 µg/kg)	< LOQ (LOQ = 12.4 µg/kg)	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOQ (LOQ = 24.4 µg/kg)
DPP	< LOD (LOD = 2.2 µg/kg)	No data	< LOD (LOD = 14.2 µg/kg)	< LOD (LOD = 2.3 µg/kg)	No data
DHP	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 14.3 µg/kg)	< LOD (LOD = 3.1 µg/kg)	69.9 µg/kg (not confirmed)
BBP	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 34.0 µg/kg)	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 33.3 µg/kg)
DCHP	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 14.3 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 31.9 µg/kg)
DEHP	49.1 µg/kg	< LOQ (LOQ = 49.9 µg/kg)	< LOQ (LOQ = 39.5 µg/kg)	< LOQ (LOQ = 36.8 µg/kg)	159.8 µg/kg
DOP	< LOD (LOD = 8.9 µg/kg)	< LOD (LOD = 25.9 µg/kg)	< LOD (LOD = 25.0 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 71.3 µg/kg)
DiNP	< LOD (LOD = 308.2 µg/kg)	< LOD (LOD = 361.4 µg/kg)	< LOD (LOD = 1170.9 µg/kg)	< LOD (LOD = 391.94 µg/kg)	< LOD (LOD = 615.7 µg/kg)
DiDP	< LOD (LOD = 186.5 µg/kg)	< LOD (LOD = 333.1 µg/kg)	< LOD (LOD = 581.6 µg/kg)	< LOD (LOD = 161.8 µg/kg)	< LOD (LOD = 969.86 µg/kg)
DDP	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 16.3 µg/kg)	< LOD (LOD = 33.9 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 125.3 µg/kg)

Table 44 continued. Retail food sample results

	S09-017299	S09-017300	S09-017301	S09-017302	S09-017303
DMP	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 3.8 µg/kg)
DEP	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 3.6 µg/kg)
DiPP	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 2.5 µg/kg)
DAP	< LOD (LOD = 21.4 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 12.3 µg/kg)	< LOD (LOD = 9.5 µg/kg)
DiBP	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOQ (LOQ = 10.8 µg/kg)	< LOD (LOD = 3.2 µg/kg)
DBP	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 9.8 µg/kg)	< LOD (LOD = 7.3 µg/kg)	35.8 µg/kg (not confirmed)	< LOD (LOD = 7.3 µg/kg)
DPP	36.3 µg/kg (not confirmed)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 9.1 µg/kg)	< LOD (LOD = 7.9 µg/kg)
DHP	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 12.7 µg/kg)	< LOD (LOD = 16.8 µg/kg)	< LOD (LOD = 20.2 µg/kg)
BBP	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 20.2 µg/kg)	< LOD (LOD = 21.6 µg/kg)	< LOD (LOD = 28.2 µg/kg)	< LOD (LOD = 41.6 µg/kg)
DCHP	< LOD (LOD = 11.4 µg/kg)	< LOD (LOD = 13.7 µg/kg)	< LOD (LOD = 16.0 µg/kg)	< LOD (LOD = 23.4 µg/kg)	< LOD (LOD = 26.2 µg/kg)
DEHP	< LOD (LOD = 10.4 µg/kg)	< LOD (LOD = 18.1 µg/kg)	65.4 µg/kg	< LOQ (LOQ = 76.7 µg/kg)	< LOD (LOD = 31.2 µg/kg)
DOP	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 19.6 µg/kg)	< LOD (LOD = 26.8 µg/kg)	< LOD (LOD = 8.6 µg/kg)	< LOD (LOD = 29.6 µg/kg)
DiNP	< LOD (LOD = 491.0 µg/kg)	< LOD (LOD = 336.8 µg/kg)	1819.6 µg/kg (same profile)	< LOD (LOD = 541.2 µg/kg)	< LOD (LOD = 421.7 µg/kg)
DiDP	< LOD (LOD = 332.2 µg/kg)	< LOD (LOD = 321.9 µg/kg)	< LOQ (LOQ = 1945.8 µg/kg)	< LOD (LOD = 57.9 µg/kg)	< LOD (LOD = 212.0 µg/kg)
DDP	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 28.7 µg/kg)	< LOD (LOD = 84.7 µg/kg)	< LOD (LOD = 64.8 µg/kg)	< LOQ (LOQ = 169.2 µg/kg)

Table 44 continued. Retail food sample results

	S09-017304	**S09-017305**	**S09-017306**	**S09-017307**	**S09-017308**
DMP	< LOD (LOD = 10.6 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 8.6 µg/kg)	< LOD (LOD = 6.4 µg/kg)	< LOQ (LOQ = 22.6 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 3.0 µg/kg)
DiPP	< LOD (LOD = 9.7 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 6.6 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 2.3 µg/kg)
DAP	< LOD (LOD = 68.7 µg/kg)	< LOD (LOD = 18.9 µg/kg)	< LOD (LOD = 20.1 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 6.5 µg/kg)
DiBP	< LOD (LOD = 12.2 µg/kg)	< LOD (LOD = 4.1 µg/kg)	142.3 µg/kg (not confirmed)	21.5 µg/kg (not confirmed)	46.1 µg/kg
DBP	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 7.3 µg/kg)	39.8 µg/kg (not confirmed)	< LOQ (LOQ = 17.8 µg/kg)	13.7 µg/kg (not confirmed)
DPP	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 6.6 µg/kg)	< LOD (LOD = 2.5 µg/kg)
DHP	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 13.9 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 3.8 µg/kg)
BBP	< LOD (LOD = 14.2 µg/kg)	< LOD (LOD = 18.5 µg/kg)	< LOD (LOD = 20.2 µg/kg)	< LOD (LOD = 17.0 µg/kg)	< LOD (LOD = 6.6 µg/kg)
DCHP	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 11.9 µg/kg)	< LOD (LOD = 20.0 µg/kg)	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 3.9 µg/kg)
DEHP	83.7 µg/kg (not confirmed)	166.1 µg/kg	91.3 µg/kg	< LOQ (LOQ = 55.4 µg/kg)	245.0 µg/kg
DOP	< LOD (LOD = 26.1 µg/kg)	< LOD (LOD = 22.9 µg/kg)	< LOD (LOD = 31.1 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 6.7 µg/kg)
DiNP	< LOD (LOD = 576.8 µg/kg)	< LOD (LOD = 345.9 µg/kg)	< LOD (LOD = 1317.3 µg/kg)	< LOD (LOD = 410.8 µg/kg)	< LOD (LOD = 108.4 µg/kg)
DiDP	< LOD (LOD = 616.0 µg/kg)	< LOD (LOD = 258.9 µg/kg)	< LOD (LOD = 876.3 µg/kg)	< LOD (LOD = 279.8 µg/kg)	< LOD (LOD = 276.5 µg/kg)
DDP	< LOD (LOD = 21.5 µg/kg)	< LOD (LOD = 41.1 µg/kg)	< LOD (LOD = 59.8 µg/kg)	< LOD (LOD = 12.5 µg/kg)	< LOD (LOD = 15.7 µg/kg)

Table 44 continued. Retail food sample results

	S09-017309	S09-017310	S09-017311	S09-017312	S09-017313
DMP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 3.0 µg/kg)
DEP	< LOD (LOD = 16.7 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOQ (LOQ = 10.0 µg/kg)
DiPP	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 2.4 µg/kg)
DAP	< LOD (LOD = 40.3 µg/kg)	< LOD (LOD = 14.1 µg/kg)	< LOD (LOD = 21.8 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 8.5 µg/kg)
DiBP	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 8.2 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOQ (LOQ = 15.4 µg/kg)
DBP	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 12.0 µg/kg)	< LOD (LOD = 7.5 µg/kg)	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 12.6 µg/kg)
DPP	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 7.2 µg/kg)	32.3 µg/kg (not confirmed)	< LOD (LOD = 12.0 µg/kg)
DHP	< LOD (LOD = 13.6 µg/kg)	< LOD (LOD = 19.8 µg/kg)	< LOD (LOD = 8.2 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 17.7 µg/kg)
BBP	< LOD (LOD = 23.9 µg/kg)	< LOD (LOD = 31.3 µg/kg)	< LOD (LOD = 15.1 µg/kg)	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 26.9 µg/kg)
DCHP	< LOD (LOD = 17.4 µg/kg)	< LOD (LOD = 29.2 µg/kg)	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 11.7 µg/kg)
DEHP	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 36.9 µg/kg)	189.7 µg/kg	< LOD (LOD = 18.1 µg/kg)	< LOQ (LOQ = 61.8 µg/kg)
DOP	< LOD (LOD = 32.8 µg/kg)	< LOD (LOD = 46.5 µg/kg)	< LOD (LOD = 29.1 µg/kg)	< LOD (LOD = 15.5 µg/kg)	< LOD (LOD = 41.1 µg/kg)
DiNP	< LOD (LOD = 434.6 µg/kg)	< LOD (LOD = 2193.8 µg/kg)	< LOD (LOD = 143.6 µg/kg)	< LOD (LOD = 236.6 µg/kg)	< LOD (LOD = 757.2 µg/kg)
DiDP	< LOD (LOD = 461.8 µg/kg)	< LOD (LOD = 546.8 µg/kg)	< LOD (LOD = 142.8 µg/kg)	< LOD (LOD = 178.3 µg/kg)	< LOD (LOD = 684.5 µg/kg)
DDP	< LOD (LOD = 38.5 µg/kg)	< LOD (LOD = 78.2 µg/kg)	< LOD (LOD = 11.7 µg/kg)	< LOD (LOD = 17.7 µg/kg)	< LOD (LOD = 81.9 µg/kg)

Table 44 continued. Retail food sample results

	S09-017314	S09-017315	S09-017316	S09-017317	S09-017318
DMP	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 6.0 µg/kg)
DEP	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 9.1 µg/kg)	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 2.7 µg/kg)
DiPP	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 2.0 µg/kg)
DAP	< LOD (LOD = 14.0 µg/kg)	< LOD (LOD = 22.8 µg/kg)	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 6.4 µg/kg)
DiBP	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOQ (LOQ = 15.4 µg/kg)
DBP	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 7.1 µg/kg)
DPP	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOQ (LOQ = 22.5 µg/kg)
DHP	< LOD (LOD = 13.5 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 17.0 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 10.5 µg/kg)
BBP	< LOD (LOD = 22.2 µg/kg)	< LOD (LOD = 15.0 µg/kg)	< LOD (LOD = 14.5 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 15.9 µg/kg)
DCHP	< LOD (LOD = 13.3 µg/kg)	< LOD (LOD = 10.6 µg/kg)	< LOD (LOD = 22.9 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 26.1 µg/kg)
DEHP	< LOQ (LOQ = 61.8 µg/kg)	< LOD (LOD = 14.6 µg/kg)	< LOQ (LOQ = 67.4 µg/kg)	< LOD (LOD = 18.1 µg/kg)	102.2 µg/kg
DOP	< LOD (LOD = 18.0 µg/kg)	< LOD (LOD = 21.3 µg/kg)	< LOD (LOD = 60.4 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 32.8 µg/kg)
DiNP	< LOD (LOD = 1247.0 µg/kg)	< LOD (LOD = 575.8 µg/kg)	< LOD (LOD = 506.8 µg/kg)	< LOQ (LOQ = 722.7 µg/kg)	< LOD (LOD = 883.6 µg/kg)
DiDP	< LOD (LOD = 415.2 µg/kg)	< LOD (LOD = 482.0 µg/kg)	< LOD (LOD = 416.6 µg/kg)	< LOD (LOD = 144.5 µg/kg)	< LOD (LOD = 349.4 µg/kg)
DDP	< LOD (LOD = 25.4 µg/kg)	< LOD (LOD = 24.0 µg/kg)	< LOD (LOD = 77.0 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 54.3 µg/kg)

Table 44 continued. Retail food sample results

	S09-017319	**S09-017320**	**S09-017321**	**S09-017322**	**S09-017323**
DMP	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 3.1 µg/kg)	44.7 µg/kg (not confirmed)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 7.7 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 1.4 µg/kg)
DiPP	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 0.9 µg/kg)
DAP	< LOD (LOD = 27.3 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 5.9 µg/kg)	< LOD (LOD = 15.9 µg/kg)	< LOD (LOD = 4.9 µg/kg)
DiBP	< LOD (LOD = 9.8 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOQ (LOQ = 10.8 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 2.8 µg/kg)
DBP	< LOD (LOD = 13.3 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 4.9 µg/kg)
DPP	< LOD (LOD = 25.9 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 2.9 µg/kg)	No data	No data
DHP	< LOD (LOD = 28.8 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 6.0 µg/kg)	< LOD (LOD = 20.6 µg/kg)	< LOD (LOD = 2.1 µg/kg)
BBP	< LOD (LOD = 45.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 42.3 µg/kg)	< LOD (LOD = 5.5 µg/kg)
DCHP	< LOD (LOD = 36.0 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 28.0 µg/kg)	< LOD (LOD = 3.0 µg/kg)
DEHP	< LOD (LOD = 39.3 0 µg/kg)	31.7 µg/kg	< LOQ (LOQ = 62.2 µg/kg)	< LOQ (LOQ = 29.6 µg/kg)	< LOD (LOD = 8.9 µg/kg)
DOP	< LOQ (LOQ = 188.4 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 28.8 µg/kg)	< LOD (LOD = 33.2 µg/kg)	< LOD (LOD = 2.6 µg/kg)
DiNP	< LOD (LOD = 1360.5 µg/kg)	< LOD (LOD = 176.8 µg/kg)	< LOD (LOD = 712.5 µg/kg)	< LOD (LOD = 1096.7 µg/kg)	< LOD (LOD = 108.4 µg/kg)
DiDP	< LOD (LOD = 886.1 µg/kg)	< LOD (LOD = 97.7 µg/kg)	< LOD (LOD = 526.2 µg/kg)	< LOD (LOD = 847.0 µg/kg)	< LOD (LOD = 99.6 µg/kg)
DDP	< LOD (LOD = 184.4 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 101.3 µg/kg)	< LOD (LOD = 34.1 µg/kg)	< LOD (LOD = 4.2 µg/kg)

Table 44 continued. Retail food sample results

	S09-017324	**S09-017325**	**S09-017326**	**S09-017327**	**S09-017328**
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 1.9 µg/kg)	16.8 µg/kg (not confirmed)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 11.1 µg/kg)	2.9 µg/kg (not confirmed)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 3.0 µg/kg)
DiPP	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 8.0 µg/kg)	4.3 µg/kg (not confirmed)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 1.9 µg/kg)
DAP	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 38.4 µg/kg)	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 9.2 µg/kg)
DiBP	61.9 µg/kg	< LOD (LOD = 11.7 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 16.5 µg/kg)
DBP	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 13.6 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 12.1 µg/kg)
DPP	< LOD (LOD = 2.3 µg/kg)	No data	No data	< LOD (LOD = 44.7 µg/kg)	No data
DHP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 24.3 µg/kg)	< LOD (LOD = 9.0 µg/kg)	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 1.4 µg/kg)
BBP	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 26.8 µg/kg)	< LOD (LOD = 13.0 µg/kg)	< LOD (LOD = 10.4 µg/kg)	< LOD (LOD = 2.7 µg/kg)
DCHP	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 99.3 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 14.7 µg/kg)	< LOD (LOD = 2.4 µg/kg)
DEHP	< LOQ (LOQ = 29.6 µg/kg)	96.2 µg/kg	< LOD (LOD = 8.9 µg/kg)	< LOD (LOD = 32.0 µg/kg)	70.8 µg/kg
DOP	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 35.0 µg/kg)	< LOD (LOD = 14.1 µg/kg)	< LOD (LOD = 33.5 µg/kg)	< LOD (LOD = 3.9 µg/kg)
DiNP	< LOD (LOD = 477.6 µg/kg)	< LOD (LOD = 1178.8 µg/kg)	< LOD (LOD = 601.7 µg/kg)	< LOD (LOD = 1958.1 µg/kg)	< LOD (LOD = 225.5 µg/kg)
DiDP	< LOD (LOD = 242.9 µg/kg)	< LOD (LOD = 418.0 µg/kg)	< LOD (LOD = 481.6 µg/kg)	< LOD (LOD = 1324.3 µg/kg)	< LOD (LOD = 359.7 µg/kg)
DDP	< LOD (LOD = 33.1 µg/kg)	< LOD (LOD = 26.4 µg/kg)	< LOD (LOD = 26.3 µg/kg)	< LOD (LOD = 118.2 µg/kg)	< LOD (LOD = 138.1 µg/kg)

Table 44 continued. Retail food sample results

	S09-017329	**S09-017330**	**S09-017331**	**S09-017332**	**S09-017333**
DMP	9.8 µg/kg (not confirmed)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 7.5 µ g/kg)
DEP	0.8 µg/kg (not confirmed)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 2.4 µg/kg)	< LOD (LOD = 6.7 µg/kg)
DiPP	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 4.4 µg/kg)
DAP	< LOD (LOD = 7.5 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 32.2 µg/kg)
DiBP	< LOD (LOD = 16.5 µg/kg)	18.0 µg/kg (not confirmed)	13.1 µg/kg	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 8.0 µg/kg)
DBP	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 15.0 µg/kg)
DPP	No data	91.5 µg/kg (not confirmed)	< LOD (LOD = 1.1 µg/kg)	No data	No data
DHP	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 9.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 11.7 µg/kg)
BBP	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 13.8 µg/kg)	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 18.9 µg/kg)
DCHP	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 5.9 µg/kg)	No data
DEHP	113.9 µg/kg	< LOQ (LOQ = 61.1 µg/kg)	< LOQ (LOQ = 62.0 µg/kg)	< LOQ (LOQ = 62.0 µg/kg)	< LOQ (LOQ = 61.1 µg/kg)
DOP	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 18.5 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 13.1 µg/kg)	< LOD (LOD = 12.4 µg/kg)
DiNP	< LOD (LOD = 290.0 µg/kg)	< LOD (LOD = 360.0 µg/kg)	< LOD (LOD = 230.5 µg/kg)	< LOD (LOD = 1044.9 µg/kg)	< LOD (LOD = 515.7 µg/kg)
DiDP	< LOD (LOD = 285.8 µg/kg)	< LOD (LOD = 518.1 µg/kg)	< LOD (LOD = 140.4 µg/kg)	< LOD (LOD = 624.9 µg/kg)	< LOD (LOD = 311.9 µg/kg)
DDP	< LOD (LOD = 25.2 µg/kg)	< LOD (LOD = 43.4 µg/kg)	< LOQ (LOQ = 25.6 µg/kg)	< LOD (LOD = 12.6 µg/kg)	< LOD (LOD = 30.1 µg/kg)

Table 44 continued. Retail food sample results

	S09-017334	**S09-017335**	**S09-017336**	**S09-017337**	**S09-017338**
DMP	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 3.6 µg/kg)
DEP	271.9 µg/kg	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 1.0 µg/kg)	< LOD (LOD = 3.6 µg/kg)
DiPP	< LOD (LOD = 1.6 µg/kg)	8.8 µg/kg (not confirmed)	< LOQ (LOQ = 10.9 µg/kg)	< LOD (LOD = 0.8 µg/kg)	< LOD (LOD = 1.7 µg/kg)
DAP	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 11.7 µg/kg)
DiBP	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 16.5 µg/kg)
DBP	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 12.1 µg/kg)
DPP	No data	No data	< LOD (LOD = 6.1 µg/kg)	No data	No data
DHP	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 8.2 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 4.4 µg/kg)
BBP	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 12.2 µg/kg)	< LOD (LOD = 18.9 µg/kg)	< LOD (LOD = 10.9 µg/kg)
DCHP	< LOD (LOD = 11.3 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 6.9 µg/kg)
DEHP	366.3 µg/kg	< LOQ (LOQ = 63.5 µg/kg)	< LOQ (LOQ = 61.1 µg/kg)	< LOD (LOD = 18.6 µg/kg)	< LOQ (LOQ = 61.4 µg/kg)
DOP	< LOD (LOD = 23.0 µg/kg)	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 13.6 µg/kg)	< LOD (LOD = 29.2 µg/kg)	< LOD (LOD = 10.3 µg/kg)
DiNP	< LOD (LOD = 671.6 µg/kg)	644.3 µg/kg (same profile)	< LOD (LOD = 731.8 µg/kg)	< LOD (LOD = 1385.2 µg/kg)	< LOD (LOD = 788.6 µg/kg)
DiDP	< LOD (LOD = 737.3 µg/kg)	< LOD (LOD = 292.2 µg/kg)	< LOD (LOD = 398.2 µg/kg)	< LOD (LOD = 780.7 µg/kg)	< LOD (LOD = 454.5 µg/kg)
DDP	< LOD (LOD = 77.0 µg/kg)	< LOD (LOD = 17.6 µg/kg)	< LOD (LOD = 52.9 µg/kg)	< LOD (LOD = 51.0 µg/kg)	< LOD (LOD = 35.6 µg/kg)

Table 44 continued. Retail food sample results

	S09-017339	S09-017340	S09-017341	S09-017342	S09-017343
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 15.7 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 1.5 µg/kg)
DEP	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 13.9 µg/kg)	< LOD (LOD = 17.3 µg/kg)	< LOD (LOD = 71.0 µg/kg)	< LOD (LOD = 2.4 µg/kg)
DiPP	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 8.6 µg/kg)	< LOD (LOD = 9.7 µg/kg)	23.3 µg/kg (not confirmed)	< LOD (LOD = 2.0 µg/kg)
DAP	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 35.8 µg/kg)	< LOD (LOD = 57.5 µg/kg)	< LOD (LOD = 174.9 µg/kg)	< LOD (LOD = 8.0 µg/kg)
DiBP	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 43.8 µg/kg)	15.6 µg/kg (not confirmed)
DBP	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 5.9 µg/kg)	< LOD (LOD = 76.3 µg/kg)	< LOD (LOD = 63.2 µg/kg)	< LOQ (LOQ = 18.4 µg/kg)
DPP	No data	< LOD (LOD = 14.2 µg/kg)	< LOD (LOD = 31.7 µg/kg)	< LOD (LOD = 50.7 µg/kg)	No data
DHP	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 13.5 µg/kg)	< LOD (LOD = 65.7 µg/kg)	< LOD (LOD = 54.4 µg/kg)	< LOD (LOD = 10.0 µg/kg)
BBP	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 23.1 µg/kg)	< LOD (LOD = 90.8 µg/kg)	< LOD (LOD = 127.1 µg/kg)	< LOD (LOD = 18.6 µg/kg)
DCHP	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 15.5 µg/kg)	< LOD (LOD = 38.0 µg/kg)	< LOD (LOD = 54.8 µg/kg)	< LOD (LOD = 15.8 µg/kg)
DEHP	< LOQ (LOQ = 61.4 µg/kg)	< LOQ (LOQ = 61.1 µg/kg)	94.3 µg/kg (not confirmed)	< LOD (LOD = 18.3 µg/kg)	< LOQ (LOQ = 62.0 µg/kg)
DOP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 32.1 µg/kg)	< LOD (LOD = 48.1 µg/kg)	< LOD (LOD = 26.2 µg/kg)
DiNP	< LOD (LOD = 226.4 µg/kg)	< LOD (LOD = 341.0 µg/kg)	< LOD (LOD = 422.6 µg/kg)	< LOD (LOD = 951.4 µg/kg)	< LOD (LOD = 941.7 µg/kg)
DiDP	< LOD (LOD = 171.3 µg/kg)	< LOD (LOD = 183.3 µg/kg)	< LOD (LOD = 211.2 µg/kg)	< LOD (LOD = 434.7 µg/kg)	< LOD (LOD = 1704.9 µg/kg)
DDP	< LOD (LOD = 11.2 µg/kg)	< LOD (LOD = 18.8 µg/kg)	< LOD (LOD = 16.2 µg/kg)	< LOD (LOD = 16.4 µg/kg)	< LOD (LOD = 26.9 µg/kg)

Table 44 continued. Retail food sample results

	S09-017344	S09-017345	S09-017346	S09-017347	S09-017348
DMP	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 0.8 µg/kg)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.4 µg/kg)
DiPP	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 1.4 µg/kg)	< LOQ (LOQ = 5.5 µg/kg)	< LOD (LOD = 1.7 µg/kg)
DAP	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 8.1 µg/kg)	< LOD (LOD = 8.9 µg/kg)	< LOD (LOD = 8.1 µg/kg)	< LOD (LOD = 9.2 µg/kg)
DiBP	< LOD (LOD = 3.2 µg/kg)	< LOQ (LOQ = 10.5 µg/kg)	108.2 µg/kg	< LOQ (LOQ = 10.5 µg/kg)	< LOD (LOD = 4.3 µg/kg)
DBP	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 5.5 µg/kg)	28.1 µg/kg (not confirmed)	< LOQ (LOQ = 18.4 µg/kg)	< LOD (LOD = 7.6 µg/kg)
DPP	< LOD (LOD = 1.0 µg/kg)	< LOD (LOD = 17.1 µg/kg)	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 14.0 µg/kg)	No data
DHP	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 7.6 µg/kg)
BBP	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 11.3 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 11.4 µg/kg)	< LOD (LOD = 13.6 µg/kg)
DCHP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 9.5 µg/kg)
DEHP	< LOQ (LOQ = 62.0 µg/kg)	< LOQ (LOQ = 62.0 µg/kg)	< LOQ (LOQ = 62.0 µg/kg)	< LOD (LOD = 153.4 µg/kg)	< LOQ (LOQ = 92.2 µg/kg)
DOP	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 11.9 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 8.5 µg/kg)
DiNP	< LOD (LOD = 463.5 µg/kg)	< LOD (LOD = 1303.4 µg/kg)	< LOD (LOD = 567.9 µg/kg)	< LOD (LOD = 2158.8 µg/kg)	< LOD (LOD = 713.9 µg/kg)
DiDP	< LOD (LOD = 642.8 µg/kg)	< LOD (LOD = 1113.2 µg/kg)	< LOD (LOD = 486.3 µg/kg)	< LOD (LOD = 6243.9 µg/kg)	< LOD (LOD = 2776.4 µg/kg)
DDP	< LOD (LOD = 51.4 µg/kg)	< LOD (LOD = 23.3 µg/kg)	< LOQ (LOQ = 22.7 µg/kg)	< LOD (LOD = 42.5 µg/kg)	< LOD (LOD = 57.6 µg/kg)

Table 44 continued. Retail food sample results

	S09-017349	S09-017350	S09-017351	S09-017352	S09-017353
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 5.2 µg/kg)
DEP	< LOD (LOD = 1.4 µg/kg)	< LOQ (LOQ = 4.5 µg/kg)	< LOD (LOD = 1.9 µg/kg)	8.3 µg/kg (not confirmed)	< LOQ (LOQ = 23.1 µg/kg)
DiPP	< LOD (LOD = 0.8 µg/kg)	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 16.8 µg/kg)	< LOD (LOD = 5.4 µg/kg)
DAP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 7.7 µg/kg)	27.8 µg/kg (not confirmed)	< LOD (LOD = 23.0 µg/kg)
DiBP	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 4.3 µg/kg)	24.2 µg/kg (not confirmed)	82.6 µg/kg
DBP	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 7.6 µg/kg)	33.0 µg/kg	24.4 µg/kg (not confirmed)
DPP	< LOD (LOD = 0.9 µg/kg)	No data	No data	No data	< LOD (LOD = 15.6 µg/kg)
DHP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 83.0 µg/kg)	< LOD (LOD = 10.6 µg/kg)
BBP	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 21.1 µg/kg)	< LOQ (LOQ = 52.1 µg/kg)
DCHP	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 18.8 µg/kg)	< LOD (LOD = 15.7 µg/kg)
DEHP	< LOD (LOD = 27.7 µg/kg)	< LOQ (LOQ = 92.2 µg/kg)	< LOD (LOD = 27.7 µg/kg)	< LOQ (LOQ = 75.5 µg/kg)	228.2 µg/kg
DOP	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 8.9 µg/kg)	< LOD (LOD = 60.6 µg/kg)	< LOD (LOD = 28.1 µg/kg)
DiNP	< LOD (LOD = 121.4 µg/kg)	< LOD (LOD = 201.4 µg/kg)	< LOD (LOD = 490.3 µg/kg)	596.7 µg/kg (not confirmed)	< LOQ (LOQ = 1609.3 µg/kg)
DiDP	< LOD (LOD = 105.2 µg/kg)	< LOD (LOD = 764.3 µg/kg)	< LOD (LOD = 336.6 µg/kg)	< LOD (LOD = 882.3 µg/kg)	< LOD (LOD = 763.6 µg/kg)
DDP	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 57.1 µg/kg)	< LOD (LOD = 25.4 µg/kg)	< LOD (LOD = 112.9 µg/kg)	< LOD (LOD = 26.3 µg/kg)

Table 44 continued. Retail food sample results

	S09-017354	S09-017355	S09-017356	S09-017357	S09-017358
DMP	< LOD (LOD = 13.0 µg/kg)	< LOD (LOD = 1.1 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 14.8 µg/kg)	< LOD (LOD = 7.4 µg/kg)
DEP	340.3 µg/kg	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 12.5 µg/kg)	< LOD (LOD = 7.3 µg/kg)
DiPP	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 4.2 µg/kg)
DAP	< LOD (LOD = 25.1 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 18.9 µg/kg)	< LOD (LOD = 31.6 µg/kg)	< LOD (LOD = 20.6 µg/kg)
DiBP	< LOQ (LOQ = 14.3 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 4.8 µg/kg)
DBP	< LOQ (LOQ = 25.4 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 4.3 µg/kg)
DPP	< LOD (LOD = 69.1 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD =16.9 µg/kg)	< LOD (LOD = 19.2 µg/kg)	< LOD (LOD = 3.9 µg/kg)
DHP	< LOD (LOD = 20.3 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 43.5 µg/kg)	< LOD (LOD = 95.3 µg/kg)	< LOD (LOD = 32.7 µg/kg)
BBP	< LOD (LOD = 25.9 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 22.3 µg/kg)	< LOD (LOD = 25.8 µg/kg)	< LOD (LOD = 16.6 µg/kg)
DCHP	< LOD (LOD = 53.8 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 14.5 µg/kg)	< LOD (LOD = 33.3 µg/kg)	< LOD (LOD = 5.3 µg/kg)
DEHP	271.0 µg/kg	< LOQ (LOQ = 92.2 µg/kg)	< LOQ (LOQ = 72.1 µg/kg)	< LOQ (LOQ = 75.5 µg/kg)	< LOQ (LOQ = 75.5 µg/kg)
DOP	< LOD (LOD = 136.9 µg/kg)	< LOD (LOD = 18.6 µg/kg)	< LOD (LOD = 30.1 µg/kg)	< LOD (LOD = 156.7 µg/kg)	< LOD (LOD = 6.8 µg/kg)
DiNP	11576.0 µg/kg (same profile)	< LOD (LOD = 1788.0 µg/kg)	< LOD (LOD = 850.9 µg/kg)	< LOD (LOD = 3822.2 µg/kg)	< LOD (LOD = 919.9 µg/kg)
DiDP	2164.9 µg/kg (not confirmed)	< LOD (LOD = 1407.9 µg/kg)	< LOD (LOD = 684.1 µg/kg)	< LOD (LOD = 2656.0 µg/kg)	< LOD (LOD = 443.3 µg/kg)
DDP	< LOD (LOD = 48.4 µg/kg)	< LOD (LOD = 112.1 µg/kg)	< LOD (LOD = 20.9 µg/kg)	< LOD (LOD = 88.0 µg/kg)	< LOD (LOD = 57.7 µg/kg)

Table 44 continued. Retail food sample results

	S09-017359	S09-017360	S09-017361	S09-017362	S09-017363
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 7.4 µg/kg)
DEP	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 15.5 µg/kg)
DiPP	< LOD (LOD = 0.8 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 9.5 µg/kg)
DAP	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 19.6 µg/kg)	72.9 µg/kg (not confirmed)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 40.2 µg/kg)
DiBP	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 2.3 µg/kg)	40.0 µg/kg (not confirmed)
DBP	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 3.6 µg/kg)	15.3 µg/kg (not confirmed)
DPP	No data	No data	< LOD (LOD = 12.0 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 4.5 µg/kg)
DHP	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 16.8 µg/kg)	< LOD (LOD = 68.2 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 10.5 µg/kg)
BBP	< LOD (LOD = 17.5 µg/kg)	< LOD (LOD = 26.3 µg/kg)	< LOD (LOD = 25.9 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 8.2 µg/kg)
DCHP	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 17.8 µg/kg)	< LOD (LOD = 44.4 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 9.4 µg/kg)
DEHP	< LOQ (LOQ = 92.2 µg/kg)	314.3 µg/kg	140.9 µg/kg (not confirmed)	< LOQ (LOQ = 72.1 µg/kg)	< LOQ (LOQ = 75.5 µg/kg)
DOP	< LOD (LOD = 8.6 µg/kg)	< LOD (LOD = 19.8 µg/kg)	< LOD (LOD = 103.6 µg/kg)	< LOD (LOD = 2.4 µg/kg)	< LOD (LOD = 17.7 µg/kg)
DiNP	< LOD (LOD = 525.8 µg/kg)	< LOD (LOD = 1596.5 µg/kg)	< LOD (LOD = 690.9 µg/kg)	< LOD (LOD = 190.5 µg/kg)	< LOD (LOD = 345.7 µg/kg)
DiDP	< LOD (LOD = 722.0 µg/kg)	< LOD (LOD = 331.6 µg/kg)	< LOD (LOD = 569.6 µg/kg)	< LOD (LOD = 86.1 µg/kg)	< LOD (LOD = 140.8 µg/kg)
DDP	< LOD (LOD = 24.1 µg/kg)	< LOD (LOD = 17.3 µg/kg)	< LOD (LOD = 32.4 µg/kg)	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 3.5 µg/kg)

Table 44 continued. Retail food sample results

	S09-017364	S09-017365	S09-017366	S09-017367	S09-017368
DMP	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 37.8 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 12.6 µg/kg)	< LOD (LOD = 15.2 µg/kg)
DEP	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 33.3 µg/kg)	< LOD (LOD = 11.7 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 15.8 µg/kg)
DiPP	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 18.1 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 9.8 µg/kg)	< LOD (LOD = 10.5 µg/kg)
DAP	< LOD (LOD = 17.9 µg/kg)	< LOD (LOD = 73.2 µg/kg)	< LOD (LOD = 26.8 µg/kg)	< LOD (LOD = 40.2 µg/kg)	< LOD (LOD = 53.7 µg/kg)
DiBP	11.1 µg/kg (not confirmed)	< LOQ (LOQ = 52.3 µg/kg)	< LOQ (LOQ = 21.7 µg/kg)	18.2 µg/kg (not confirmed)	< LOD (LOD = 12.1 µg/kg)
DBP	< LOD (LOD = 4.3 µg/kg)	53.3 µg/kg (not confirmed)	< LOD (LOD = 5.7 µg/kg)	< LOQ (LOQ = 17.8 µg/kg)	< LOD (LOD = 11.3 µg/kg)
DPP	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 14.3 µg/kg)	No data	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 8.5 µg/kg)
DHP	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 20.1 µg/kg)	< LOD (LOD = 25.0 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 55.0 µg/kg)
BBP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 42.7 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 16.0 µg/kg)	< LOD (LOD = 25.3 µg/kg)
DCHP	< LOD (LOD = 6.6 µg/kg)	< LOD (LOD = 26.4 µg/kg)	< LOD (LOD = 15.7 µg/kg)	< LOD (LOD = 21.1 µg/kg)	< LOD (LOD = 7.9 µg/kg)
DEHP	< LOQ (LOQ = 75.5 µg/kg)	155.2 µg/kg (not confirmed)	83.4 µg/kg	58.6 µg/kg	< LOD (LOD = 29.8 µg/kg)
DOP	< LOD (LOD = 38.9 µg/kg)	< LOD (LOD = 25.1 µg/kg)	< LOD (LOD = 60.6 µg/kg)	< LOD (LOD = 14.6 µg/kg)	< LOD (LOD = 22.6 µg/kg)
DiNP	< LOD (LOD = 568.8 µg/kg)	< LOD (LOD = 1307.9 µg/kg)	< LOD (LOD = 733.8 µg/kg)	< LOD (LOD = 1159.3 µg/kg)	< LOD (LOD = 1536.8 µg/kg)
DiDP	< LOD (LOD = 409.8 µg/kg)	< LOD (LOD = 1492.4 µg/kg)	< LOD (LOD = 725.7 µg/kg)	< LOD (LOD = 877.5 µg/kg)	< LOD (LOD = 645.9 µg/kg)
DDP	< LOD (LOD = 47.5 µg/kg)	< LOD (LOD = 33.9 µg/kg)	< LOD (LOD = 50.4 µg/kg)	< LOD (LOD = 37.6 µg/kg)	< LOD (LOD = 54.4 µg/kg)

Table 44 continued. Retail food sample results

	S09-017369	S09-017370	S09-017371	S09-017372	S09-017373
DMP	< LOD (LOD = 18.4 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 3.6 µg/kg)	48.7 µg/kg (not confirmed)
DEP	< LOD (LOD = 25.9 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 26.0 µg/kg)
DiPP	< LOD (LOD = 15.7 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 20.4 µg/kg)
DAP	< LOD (LOD = 63.1 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 16.4 µg/kg)	< LOD (LOD = 21.8 µg/kg)	< LOD (LOD = 67.1 µg/kg)
DiBP	< LOD (LOD = 13.6 µg/kg)	< LOD (LOD = 5.7 µg/kg)	19.3 µg/kg (not confirmed)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 24.0 µg/kg)
DBP	< LOD (LOD = 19.3 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 16.3 µg/kg)
DPP	< LOD (LOD = 21.7 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 16.0 µg/kg)
DHP	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 75.5 µg/kg)	< LOD (LOD = 21.4 µg/kg)	< LOD (LOD = 115.9 µg/kg)
BBP	< LOD (LOD = 11.3 µg/kg)	< LOD (LOD = 18.2 µg/kg)	< LOD (LOD = 29.8 µg/kg)	< LOD (LOD = 21.7 µg/kg)	< LOD (LOD = 56.7 µg/kg)
DCHP	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 24.5 µg/kg)	< LOD (LOD = 10.6 µg/kg)	< LOD (LOD = 73.7 µg/kg)
DEHP	< LOQ (LOQ = 67.4 µg/kg)	< LOQ (LOQ = 99.8 µg/kg)	< LOQ (LOQ = 99.4 µg/kg)	< LOD (LOD = 29.8 µg/kg)	402.9 µg/kg (not confirmed)
DOP	< LOD (LOD = 82.9 µg/kg)	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 46.3 µg/kg)	< LOD (LOD = 14.1 µg/kg)	< LOD (LOD = 103.6 µg/kg)
DiNP	< LOD (LOD = 579.8 µg/kg)	< LOD (LOD = 263.5 µg/kg)	< LOD (LOD = 2232.5 µg/kg)	< LOD (LOD = 490.7 µg/kg)	< LOQ (LOQ = 9489.5 µg/kg)
DiDP	< LOD (LOD = 300.0 µg/kg)	< LOD (LOD = 200.9 µg/kg)	< LOD (LOD = 1932.8 µg/kg)	< LOD (LOD = 210.3 µg/kg)	< LOD (LOD = 1924.9 µg/kg)
DDP	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 12.3 µg/kg)	< LOD (LOD = 168.0 µg/kg)	< LOD (LOD = 23.6 µg/kg)	< LOD (LOD = 160.0 µg/kg)

Table 44 continued. Retail food sample results

	S09-017374	S09-017375	S09-017376	S09-017377	S09-017378
DMP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 1.5 µg/kg)	22.0 µg/kg (not confirmed)	< LOD (LOD = 0.7 µg/kg)	8.2 µg/kg (not confirmed)
DEP	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 12.2 µg/kg)	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 7.7 µg/kg)
DiPP	< LOD (LOD = 7.5 µg/kg)	< LOD (LOD = 3.4 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 5.9 µg/kg)
DAP	< LOD (LOD = 35.0 µg/kg)	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 32.2 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 20.6 µg/kg)
DiBP	< LOD (LOD = 8.1 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 6.1 µg/kg)
DBP	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 3.4 µg/kg)	18.2 µg/kg (not confirmed)	< LOD (LOD = 6.2 µg/kg)	35.7 µg/kg (not confirmed)
DPP	< LOD (LOD = 21.7 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 0.8 µg/kg)	< LOD (LOD = 17.5 µg/kg)
DHP	< LOD (LOD = 32.7 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 22.4 µg/kg)
BBP	< LOD (LOD = 17.5 µg/kg)	< LOD (LOD = 9.0 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 12.1 µg/kg)
DCHP	< LOD (LOD = 19.7 µg/kg)	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 7.7 µg/kg)	< LOD (LOD = 27.5 µg/kg)
DEHP	< LOQ (LOQ = 99.4 µg/kg)	< LOD (LOD = 29.9 µg/kg)	< LOD (LOD = 29.8 µg/kg)	< LOD (LOD = 29.9 µg/kg)	2176.2 µg/kg
DOP	< LOD (LOD = 24.3 µg/kg)	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 8.9 µg/kg)	< LOD (LOD = 64.8 µg/kg)
DiNP	< LOD (LOD = 812.9 µg/kg)	< LOQ (LOQ = 796.1 µg/kg)	< LOD (LOD = 352.2 µg/kg)	< LOD (LOD = 226.4 µg/kg)	< LOD (LOD = 1250.2 µg/kg)
DiDP	< LOD (LOD = 582.5 µg/kg)	< LOD (LOD = 221.7 µg/kg)	< LOD (LOD = 138.9 µg/kg)	< LOD (LOD = 178.2 µg/kg)	< LOD (LOD = 1182.1 µg/kg)
DDP	< LOD (LOD = 69.1 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 14.4 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 141.1 µg/kg)

Table 44 continued. Retail food sample results

	S09-017379	**S09-017380**	**S09-017381**	**S09-017382**	**S09-017383**
DMP	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 8.4 µg/kg)
DEP	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 6.9 µg/kg)
DiPP	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 4.2 µg/kg)
DAP	< LOD (LOD = 19.2 µg/kg)	< LOD (LOD = 21.9 µg/kg)	< LOD (LOD = 21.8 µg/kg)	< LOD (LOD = 16.7 µg/kg)	< LOD (LOD = 16.9 µg/kg)
DiBP	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOQ (LOQ = 16.0 µg/kg)	< LOQ (LOQ = 12.7 µg/kg)	< LOD (LOD = 3.7 µg/kg)
DBP	< LOQ (LOQ = 15.8 µg/kg)	< LOQ (LOQ = 14.0 µg/kg)	< LOQ (LOQ = 13.9 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 4.2 µg/kg)
DPP	< LOD (LOD = 21.0 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 7.5 µg/kg)	< LOD (LOD = 4.0 µg/kg)
DHP	< LOD (LOD = 36.8 µg/kg)	< LOD (LOD = 9.0 µg/kg)	< LOD (LOD = 21.4 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 4.4 µg/kg)
BBP	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 17.7 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 7.0 µg/kg)
DCHP	< LOD (LOD = 26.4 µg/kg)	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 11.5 µg/kg)
DEHP	221.5 µg/kg	< LOQ (LOQ = 51.4 µg/kg)	< LOQ (LOQ = 99.4 µg/kg)	58.9 µg/kg	95.6 µg/kg
DOP	< LOD (LOD = 58.4 µg/kg)	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 13.6 µg/kg)	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 11.7 µg/kg)
DiNP	< LOD (LOD = 1363.8 µg/kg)	< LOD (LOD = 387.2 µg/kg)	< LOD (LOD = 490.7 µg/kg)	< LOD (LOD = 517.3 µg/kg)	< LOD (LOD = 358.3 µg/kg)
DiDP	< LOD (LOD = 1406.1 µg/kg)	< LOD (LOD = 236.8 µg/kg)	< LOD (LOD = 426.5 µg/kg)	< LOQ (LOQ = 484.6 µg/kg)	< LOD (LOD = 263.1 µg/kg)
DDP	< LOD (LOD = 27.8 µg/kg)	< LOD (LOD = 27.5 µg/kg)	< LOD (LOD = 25.7 µg/kg)	< LOD (LOD = 16.6 µg/kg)	< LOD (LOD = 27.9 µg/kg)

Table 44 continued. Retail food sample results

	S09-017384	S09-017385	S09-017386	S09-017387	S09-017388
DMP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 71.8 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 13.3 µg/kg)	< LOD (LOD = 53.7 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 1.5 µg/kg)
DiPP	< LOD (LOD = 9.0 µg/kg)	< LOD (LOD = 28.9 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 1.1 µg/kg)
DAP	< LOD (LOD = 33.5 µg/kg)	< LOD (LOD = 187.8 µg/kg)	< LOD (LOD = 26.8 µg/kg)	< LOD (LOD = 13.4 µg/kg)	< LOD (LOD = 7.79 µg/kg)
DiBP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 30.0 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 7.9 µg/kg)
DBP	< LOD (LOD = 5.9 µg/kg)	< LOD (LOD = 31.3 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 9.0 µg/kg)
DPP	< LOD (LOD = 13.2 µg/kg)	< LOD (LOD = 29.2 µg/kg)	< LOD (LOD = 5.7 µg/kg)	41.9 µg/kg (not confirmed)	< LOD (LOD = 1.8 µg/kg)
DHP	< LOD (LOD = 12.2 µg/kg)	< LOD (LOD = 69.9 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 2.1 µg/kg)
BBP	< LOD (LOD = 20.2 µg/kg)	< LOD (LOD = 149.0 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 3.9 µg/kg)
DCHP	< LOD (LOD = 16.3 µg/kg)	< LOD (LOD = 35.2 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 8.4 µg/kg)
DEHP	68.6 µg/kg (not confirmed)	< LOQ (LOQ = 72.1 µg/kg)	55.7 µg/kg	59.5 µg/kg	< LOD (LOD = 31.2 µg/kg)
DOP	< LOD (LOD = 21.1 µg/kg)	< LOD (LOD = 38.3 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 11.8 µg/kg)
DiNP	< LOD (LOD = 1400.2 µg/kg)	< LOD (LOD = 3111.6 µg/kg)	< LOD (LOD = 55.8 µg/kg)	< LOD (LOD = 99.1 µg/kg)	< LOD (LOD = 322.0 µg/kg)
DiDP	< LOD (LOD = 428.5 µg/kg)	< LOD (LOD = 1920.5 µg/kg)	< LOD (LOD = 28.2 µg/kg)	< LOD (LOD = 75.0 µg/kg)	< LOD (LOD = 292.7 µg/kg)
DDP	< LOD (LOD = 32.6 µg/kg)	< LOD (LOD = 69.6 µg/kg)	< LOD (LOD = 21.2 µg/kg)	< LOD (LOD = 16.9 µg/kg)	< LOD (LOD = 3.6 µg/kg)

Table 44 continued. Retail food sample results

	S09-017389	S09-017390	S09-017391	S09-017392	S09-017393
DMP	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 1.5 µg/kg)
DEP	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 3.3 µg/kg)
DiPP	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 1.3 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 1.3 µg/kg)
DAP	< LOD (LOD = 21.2 µg/kg)	< LOD (LOD = 17.5 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 5.4 µg/kg)
DiBP	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 5.0 µg/kg)
DBP	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 6.8 µg/kg)	33.4 µg/kg	< LOD (LOD = 6.8 µg/kg)
DPP	< LOD (LOD = 18.2 µg/kg)	< LOD (LOD = 6.6 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 18.2 µg/kg)	< LOD (LOD = 3.1 µg/kg)
DHP	< LOD (LOD = 12.3 µg/kg)	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 2.6 µg/kg)
BBP	< LOD (LOD = 34.4 µg/kg)	< LOD (LOD = 20.2 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOQ (LOQ = 24.5 µg/kg)	< LOD (LOD = 6.7 µg/kg)
DCHP	< LOD (LOD = 18.1 µg/kg)	< LOD (LOD = 17.0 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 2.7 µg/kg)
DEHP	115.0 µg/kg	< LOD (LOD = 20.8 µg/kg)	48.5 µg/kg	204.3 µg/kg	< LOD (LOD = 13.9 µg/kg)
DOP	< LOD (LOD = 47.3 µg/kg)	< LOD (LOD = 35.7 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 5.5 µg/kg)
DiNP	< LOD (LOD = 834.9 µg/kg)	< LOD (LOD = 1599.2 µg/kg)	212.7 µg/kg (not confirmed)	677.2 µg/kg (not confirmed)	107.4 µg/kg (not confirmed)
DiDP	< LOD (LOD = 1187.4 µg/kg)	< LOD (LOD = 2513.3 µg/kg)	< LOD (LOD = 69.8 µg/kg)	< LOQ (LOQ = 288.4 µg/kg)	< LOQ (LOQ = 238.0 µg/kg)
DDP	< LOD (LOD = 9.4 µg/kg)	< LOD (LOD = 58.4 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 12.0 µg/kg)	< LOD (LOD = 5.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017394	S09-017395	S09-017396	S09-017397	S09-017398
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 3.6 µg/kg)
DiPP	< LOD (LOD = 1.7 µg/kg)	17.0 µg/kg (not confirmed)	< LOD (LOD = 1.3 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOQ (LOQ = 6.2 µg/kg)
DAP	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 14.6 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 17.9 µg/kg)
DiBP	< LOD (LOD = 5.0 µg/kg)	< LOQ (LOQ = 16.6 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 5.0 µg/kg)
DBP	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 56.1 µg/kg)
DPP	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 29.7 µg/kg)	< LOD (LOD = 44.0 µg/kg)
DHP	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 25.8 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 23.1 µg/kg)
BBP	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 48.6 µg/kg)	< LOD (LOD = 6.6 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 43.2 µg/kg)
DCHP	< LOD (LOD = 5.9 µg/kg)	< LOD (LOD = 32.2 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 25.8 µg/kg)
DEHP	< LOQ (LOQ = 46.3 µg/kg)	68.7 µg/kg	< LOQ (LOQ = 46.3 µg/kg)	< LOQ (LOQ = 46.3 µg/kg)	< LOD (LOD = 52.6 µg/kg)
DOP	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 49.4 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 68.4 µg/kg)
DiNP	< LOD (LOD = 73.2 µg/kg)	< LOD (LOD = 455.4 µg/kg)	< LOD (LOD = 98.1 µg/kg)	< LOD (LOD = 163.4 µg/kg)	< LOD (LOD = 416.7 µg/kg)
DiDP	< LOD (LOD = 45.7 µg/kg)	< LOD (LOD = 240.7 µg/kg)	< LOD (LOD = 142.8 µg/kg)	< LOD (LOD = 102.4 µg/kg)	< LOD (LOD = 408.1 µg/kg)
DDP	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 18.5 µg/kg)	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 37.6 µg/kg)

Table 44 continued. Retail food sample results

	S09-017399	S09-017400	S09-017401	S09-017402	S09-017403
DMP	< LOD (LOD = 11.0 µg/kg)	3.4 µg/kg (not confirmed)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 13.0 µg/kg)
DEP	< LOD (LOD = 16.2 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 19.3 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 26.0 µg/kg)
DiPP	< LOD (LOD = 15.9 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 20.9 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 7.8 µg/kg)
DAP	< LOD (LOD = 60.4 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 25.4 µg/kg)	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 34.8 µg/kg)
DiBP	< LOD (LOD = 20.9 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 9.7 µg/kg)	< LOD (LOD = 4.9 µg/kg)	5.3 µg/kg (not confirmed)
DBP	< LOD (LOD = 13.6 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 6.1 µg/kg)	19.4 µg/kg (not confirmed)
DPP	< LOD (LOD = 12.5 µg/kg)	< LOD (LOD = 8.4 µg/kg)	< LOD (LOD = 32.9 µg/kg)	< LOD (LOD = 5.8 µg/kg)	< LOD (LOD = 6.8 µg/kg)
DHP	< LOD (LOD = 24.5 µg/kg)	3.4 µg/kg (not confirmed)	< LOD (LOD = 32.7 µg/kg)	< LOD (LOD = 9.9 µg/kg)	< LOD (LOD = 14.9 µg/kg)
BBP	< LOD (LOD = 42.1 µg/kg)	18.2 µg/kg (not confirmed)	< LOD (LOD = 10.9 µg/kg)	< LOD (LOD = 10.3 µg/kg)	< LOD (LOD = 30.7 µg/kg)
DCHP	< LOD (LOD = 34.9 µg/kg)	< LOD (LOD = 32.8 µg/kg)	< LOD (LOD = 17.6 µg/kg)	42.7 µg/kg (not confirmed)	< LOD (LOD = 19.8 µg/kg)
DEHP	< LOD (LOD = 30.9 µg/kg)	849.7 µg/kg	< LOQ (LOQ =64.7 µg/kg)	364.9 µg/kg	37.5 µg/kg (not confirmed)
DOP	< LOD (LOD = 43.0 µg/kg)	< LOD (LOD = 29.1 µg/kg)	< LOD (LOD = 39.6 µg/kg)	< LOD (LOD = 31.7 µg/kg)	< LOD (LOD = 23.1 µg/kg)
DiNP	279.5 µg/kg (not confirmed)	< LOQ (LOQ = 885.5 µg/kg)	5302.5 µg/kg (same profile)	2773.7 µg/kg (not confirmed)	< LOD (LOD = 3750.6 µg/kg)
DiDP	< LOD (LOD = 57.1 µg/kg)	< LOD (LOD = 164.2 µg/kg)	< LOD (LOD = 494.5 µg/kg)	< LOD (LOD = 1009.9 µg/kg)	< LOD (LOD = 3549.3 µg/kg)
DDP	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 14.8 µg/kg)	< LOD (LOD = 76.0 µg/kg)	< LOD (LOD = 77.0 µg/kg)	< LOD (LOD = 115.2 µg/kg)

Table 44 continued. Retail food sample results

	S09-017404	S09-017405	S09-017406	S09-017407	S09-017408
DMP	< LOQ (LOQ = 16.4 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 12.5 µg/kg)	< LOD (LOD = 5.2 µg/kg)
DEP	< LOD (LOD = 12.6 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 9.6 µg/kg)
DiPP	< LOD (LOD = 20.0 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 6.0 µg/kg)
DAP	< LOD (LOD = 35.8 µg/kg)	< LOD (LOD = 15.2 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 63.0 µg/kg)	< LOD (LOD = 16.9 µg/kg)
DiBP	73.5 µg/kg (not confirmed)	7.4 µg/kg (not confirmed)	< LOQ (LOQ = 6.5 µg/kg)	58.5 µg/kg (not confirmed)	< LOD (LOD = 8.8 µg/kg)
DBP	< LOQ (LOQ = 22.5 µg/kg)	< LOD (LOD = 8.3 µg/kg)	< LOQ (LOQ = 5.5 µg/kg)	< LOQ (LOQ = 53.6 µg/kg)	< LOD (LOD = 4.3 µg/kg)
DPP	< LOD (LOD = 42.2 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 22.7 µg/kg)	< LOD (LOD = 11.9 µg/kg)
DHP	< LOD (LOD = 86.8 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 26.3 µg/kg)	< LOD (LOD = 11.0 µg/kg)
BBP	< LOD (LOD = 78.5 µg/kg)	< LOD (LOD = 13.0 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 63.5 µg/kg)	< LOD (LOD = 23.7 µg/kg)
DCHP	< LOD (LOD = 105.7 µg/kg)	< LOD (LOD = 16.0 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 38.3 µg/kg)	< LOD (LOD = 21.3 µg/kg)
DEHP	309.6 µg/kg (not confirmed)	81.0 µg/kg	< LOQ (LOQ = 12.1 µg/kg)	< LOD (LOD = 26.3 µg/kg)	< LOQ (LOQ = 49.8 µg/kg)
DOP	< LOD (LOD = 123.6 µg/kg)	< LOD (LOD = 19.1 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 39.7 µg/kg)	< LOD (LOD = 33.5 µg/kg)
DiNP	< LOQ (LOQ = 5555.8 µg/kg)	< LOD (LOD = 1691.4 µg/kg)	< LOD (LOD = 382.4 µg/kg)	< LOD (LOD = 3200.5 µg/kg)	< LOD (LOD = 204.9 µg/kg)
DiDP	< LOD (LOD = 1755.1 µg/kg)	< LOD (LOD = 1994.9 µg/kg)	< LOD (LOD = 436.8 µg/kg)	< LOD (LOD = 3255.5 µg/kg)	< LOD (LOD = 135.0 µg/kg)
DDP	< LOD (LOD = 133.7 µg/kg)	< LOD (LOD = 136.6 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 117.4 µg/kg)	< LOD (LOD = 8.3 µg/kg)

Table 44 continued. Retail food sample results

	S09-017409	S09-017410	S09-017411	S09-017412	S09-017413
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 73.4 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 4.5 µg/kg)
DEP	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 18.4 µg/kg)	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 3.8 µg/kg)
DiPP	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 3.2 µg/kg)
DAP	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 37.6 µg/kg)	< LOD (LOD = 16.1 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 10.1 µg/kg)
DiBP	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 8.6 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOQ (LOQ = 10.5 µg/kg)
DBP	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 7.6 µg/kg)	17.0 µg/kg (not confirmed)
DPP	< LOD (LOD = 16.9 µg/kg)	< LOD (LOD = 16.1 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 7.1 µg/kg)
DHP	15.8 µg/kg (not confirmed)	< LOD (LOD = 13.9 µg/kg)	< LOD (LOD = 6.0 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 10.7 µg/kg)
BBP	141.2 µg/kg (not confirmed)	< LOD (LOD = 30.6 µg/kg)	< LOD (LOD = 15.1 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 10.7 µg/kg)
DCHP	< LOQ (LOQ = 40.6 µg/kg)	< LOD (LOD = 22.3 µg/kg)	< LOD (LOD = 13.1 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 21.0 µg/kg)
DEHP	47.4 µg/kg	238.1 µg/kg	< LOD (LOD = 11.8 µg/kg)	< LOQ (LOQ = 65.9 µg/kg)	240.4 µg/kg
DOP	< LOD (LOD = 15.4 µg/kg)	< LOD (LOD = 57.1 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 58.5 µg/kg)
DiNP	< LOQ (LOQ = 528.9 µg/kg)	< LOD (LOD = 156.4 µg/kg)	< LOD (LOD = 727.4 µg/kg)	< LOD (LOD = 261.8 µg/kg)	< LOD (LOD = 3636.9 µg/kg)
DiDP	< LOD (LOD = 101.1 µg/kg)	< LOD (LOD = 115.9 µg/kg)	< LOD (LOD = 639.5 µg/kg)	< LOD (LOD = 79.5 µg/kg)	< LOD (LOD = 4042.1 µg/kg)
DDP	< LOD (LOD = 8.0 µg/kg)	< LOD (LOD = 7.8 µg/kg)	< LOD (LOD = 24.8 µg/kg)	< LOD (LOD = 8.1 µg/kg)	< LOD (LOD = 165.7 µg/kg)

Table 44 continued. Retail food sample results

	S09-017414	S09-017415	S09-017416	S09-017417	S09-017418
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 4.5 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 5.4 µg/kg)
DEP	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 9.4 µg/kg)	< LOD (LOD = 7.5 µg/kg)	43.5 µg/kg	< LOQ (LOQ = 19.9 µg/kg)
DiPP	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 4.5 µg/kg)
DAP	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 14.6 µg/kg)	< LOD (LOD = 26.8 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 17.9 µg/kg)
DiBP	< LOD (LOD = 7.9 µg/kg)	38.0 µg/kg	66.0 µg/kg (not confirmed)	13.4 µg/kg (not confirmed)	< LOQ (LOQ = 18.0 µg/kg)
DBP	< LOD (LOD = 9.0 µg/kg)	< LOQ (LOQ = 25.3 µg/kg)	35.7 µg/kg (not confirmed)	30.3 µg/kg (not confirmed)	< LOQ (LOQ = 25.3 µg/kg)
DPP	< LOD (LOD = 11.4 µg/kg)	< LOD (LOD = 17.9 µg/kg)	< LOD (LOD = 28.0 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 11.9 µg/kg)
DHP	< LOQ (LOQ = 15.1 µg/kg)	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 10.5 µg/kg)
BBP	< LOD (LOD = 10.9 µg/kg)	< LOD (LOD = 22.7 µg/kg)	< LOD (LOD = 20.2 µg/kg)	< LOD (LOD = 8.1 µg/kg)	< LOD (LOD = 20.2 µg/kg)
DCHP	< LOD (LOD =6.6 µg/kg)	< LOD (LOD = 14.2 µg/kg)	< LOD (LOD = 14.2 µg/kg)	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 17.3 µg/kg)
DEHP	< LOQ (LOQ = 67.4 µg/kg)	< LOQ (LOQ = 65.9 µg/kg)	108.4 µg/kg (not confirmed)	97.8 µg/kg	124.7 µg/kg
DOP	< LOD (LOD = 24.3 g/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 16.1 µg/kg)	< LOD (LOD = 56.6 µg/kg)	< LOD (LOD = 15.7 µg/kg)
DiNP	< LOD (LOD = 742.7 µg/kg)	< LOD (LOD = 315.8 µg/kg)	< LOD (LOD = 1769.5 µg/kg)	3374.7 µg/kg (not confirmed)	< LOD (LOD = 816.1 µg/kg)
DiDP	< LOD (LOD = 369.4 µg/kg)	< LOD (LOD = 241.9 µg/kg)	< LOD (LOD = 815.9 µg/kg)	< LOD (LOD = 777.7 µg/kg)	< LOD (LOD = 755.0 µg/kg)
DDP	< LOD (LOD = 16.6 µg/kg)	< LOD (LOD = 9.0 µg/kg)	< LOD (LOD = 33.0 µg/kg)	< LOD (LOD = 20.6 µg/kg)	< LOD (LOD = 38.2 µg/kg)

Table 44 continued. Retail food sample results

	S09-017419	S09-017420	S09-017421	S09-017422	S09-017423
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 13.0 µg/kg)	< LOD (LOD = 1.6 µg/kg)
DEP	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 17.6 µg/kg)	< LOD (LOD = 4.5 µg/kg)
DiPP	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 2.2 µg/kg)
DAP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 15.7 µg/kg)	< LOD (LOD = 13.6 µg/kg)	< LOD (LOD = 9.5 µg/kg)
DiBP	< LOD (LOD = 4.3 µg/kg)	< LOQ (LOQ = 14.3 µg/kg)	< LOD (LOD = 10.0 µg/kg)	< LOD (LOD = 17.2 µg/kg)	< LOD (LOD = 4.3 µg/kg)
DBP	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 25.4 µg/kg)	< LOD (LOD = 5.5 µg/kg)
DPP	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 22.5 µg/kg)	< LOD (LOD = 10.8 µg/kg)
DHP	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 12.6 µg/kg)	< LOD (LOD = 14.3 µg/kg)	< LOD (LOD = 9.8 µg/kg)
BBP	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 14.3 µg/kg)	< LOD (LOD = 15.8 µg/kg)	< LOD (LOD = 26.2 µg/kg)	< LOD (LOD = 15.8 µg/kg)
DCHP	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 9.4 µg/kg)	< LOD (LOD = 16.2 µg/kg)	< LOD (LOD = 58.4 µg/kg)	< LOD (LOD = 8.1 µg/kg)
DEHP	< LOQ (LOQ = 56.9 µg/kg)	< LOQ (LOQ = 56.9 µg/kg)	< LOD (LOD = 17.1 µg/kg)	< LOD (LOD = 20.2 µg/kg)	< LOQ (LOQ = 56.9 µg/kg)
DOP	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 23.7 µg/kg)	< LOD (LOD = 26.3 µg/kg)	< LOD (LOD = 14.6 µg/kg)
DiNP	< LOD (LOD = 368.2 µg/kg)	< LOD (LOD = 543.9 µg/kg)	< LOD (LOD = 1045.3 µg/kg)	< LOD (LOD = 222.8 µg/kg)	< LOD (LOD = 775.1 µg/kg)
DiDP	< LOD (LOD = 260.1 µg/kg)	< LOD (LOD = 475.4 µg/kg)	< LOD (LOD = 818.1 µg/kg)	< LOD (LOD = 147.0 µg/kg)	< LOD (LOD = 566.0 µg/kg)
DDP	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 14.1 µg/kg)	< LOD (LOD = 43.3 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 35.3 µg/kg)

Table 44 continued. Retail food sample results

	S09-017424	S09-017425	S09-017426	S09-017427	S09-017428
DMP	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 0.7 µg/kg)	0.9 µg/kg (not confirmed)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 6.9 µg/kg)
DEP	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 19.9 µg/kg)
DiPP	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 1.5 µg/kg)	46.4 µg/kg (not confirmed)
DAP	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 26.1 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 19.9 µg/kg)
DiBP	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 25.5 µg/kg)
DBP	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 6.8 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOQ (LOQ = 25.3 µg/kg)	< LOD (LOD = 23.0 µg/kg)
DPP	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 0.8 µg/kg)	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 51.9 µg/kg)	< LOD (LOD = 26.2 µg/kg)
DHP	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 1.0 µg/kg)	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 41.2 µg/kg)
BBP	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 1.9 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 9.8 µg/kg)	< LOD (LOD = 35.6 µg/kg)
DCHP	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 11.9 µg/kg)	< LOD (LOD = 28.2 µg/kg)	< LOD (LOD = 60.5 µg/kg)
DEHP	< LOD (LOD = 17.1 µg/kg)	< LOQ (LOQ = 46.3 µg/kg)	76.1 µg/kg	204.3 µg/kg	46.8 µg/kg (not confirmed)
DOP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 29.7 µg/kg)	< LOD (LOD = 22.5 µg/kg)	< LOD (LOD = 60.6 µg/kg)
DiNP	< LOQ (LOQ = 609.2 µg/kg)	< LOD (LOD = 101.0 µg/kg)	< LOD (LOD = 665.1 µg/kg)	1374.8 µg/kg (not confirmed)	< LOD (LOD = 3900.6 µg/kg)
DiDP	< LOD (LOD = 135.7 µg/kg)	< LOD (LOD = 114.8 µg/kg)	< LOD (LOD = 2373.2 µg/kg)	< LOD (LOD = 323.5 µg/kg)	< LOD (LOD = 3877.2 µg/kg)
DDP	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 11.7 µg/kg)	< LOD (LOD = 99.2 µg/kg)	< LOD (LOD = 45.4 µg/kg)	< LOD (LOD = 132.3 µg/kg)

Table 44 continued. Retail food sample results

	S09-017429	S09-017430	S09-017431	S09-017432	S09-017433
DMP	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 20.4 µg/kg)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOQ (LOQ = 14.6 µg/kg)	< LOD (LOD = 16.2 µg/kg)	< LOD (LOD = 2.6 µg/kg)
DiPP	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 1.1 µg/kg)
DAP	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 7.0 µg/kg)	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 48.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)
DiBP	< LOQ (LOQ = 15.2 µg/kg)	< LOQ (LOQ = 15.2 µg/kg)	21.6 µg/kg (not confirmed)	< LOD (LOD = 20.9 µg/kg)	< LOD (LOD = 4.6 µg/kg)
DBP	< LOQ (LOQ = 24.0 µg/kg)	< LOQ (LOQ = 24.0 µg/kg)	< LOQ (LOQ = 24.0 µg/kg)	< LOD (LOD = 80.3 µg/kg)	< LOD (LOD = 7.2 µg/kg)
DPP	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 4.4 µg/kg)	< LOD (LOD = 19.0 µg/kg)	23.4 µg/kg (not confirmed)
DHP	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 8.8 µg/kg)	< LOD (LOD = 17.5 µg/kg)	< LOD (LOD = 11.1 µg/kg)
BBP	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 8.9 µg/kg)	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 40.0 µg/kg)	< LOD (LOD = 12.3 µg/kg)
DCHP	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 10.5 µg/kg)	72.5 µg/kg (not confirmed)	< LOD (LOD = 13.6 µg/kg)
DEHP	< LOQ (LOQ = 43.5 µg/kg)	< LOQ (LOQ = 43.5 µg/kg)	< LOQ (LOQ = 43.5 µg/kg)	< LOD (LOD = 18.6 µg/kg)	101.4 µg/kg (not confirmed)
DOP	< LOD (LOD = 7.7 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 11.1 µg/kg)	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 21.4 µg/kg)
DiNP	< LOD (LOD = 283.4 µg/kg)	< LOD (LOD = 645.9 µg/kg)	< LOD (LOD = 877.7 µg/kg)	< LOD (LOD = 290.9 µg/kg)	< LOD (LOD = 760.4 µg/kg)
DiDP	< LOD (LOD = 366.2 µg/kg)	< LOD (LOD = 656.7 µg/kg)	< LOD (LOD = 831.0 µg/kg)	< LOD (LOD = 215.4 µg/kg)	< LOD (LOD = 944.4 µg/kg)
DDP	< LOD (LOD = 16.9 µg/kg)	< LOD (LOD = 47.6 µg/kg)	< LOD (LOD = 32.3 µg/kg)	< LOD (LOD = 17.2 µg/kg)	< LOD (LOD = 70.6 µg/kg)

Table 44 continued. Retail food sample results

	S09-017434	**S09-017435**	**S09-017436**	**S09-017437**	**S09-017438**
DMP	< LOD (LOD = 2.3 µg/kg)	< LOD (LOD = 7.4 µg/kg)	< LOD (LOD = 5.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 14.7 µg/kg)
DEP	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 36.1 µg/kg)	< LOD (LOD = 27.2 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 39.7 µg/kg)
DiPP	< LOD (LOD = 2.4 µg/kg)	< LOD (LOD = 51.4 µg/kg)	< LOD (LOD = 24.3 µg/kg)	< LOD (LOD = 0.8 µg/kg)	48.5 µg/kg (not confirmed)
DAP	< LOD (LOD = 23.7 µg/kg)	< LOD (LOD = 94.7 µg/kg)	< LOD (LOD = 53.7 µg/kg)	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 165.1 µg/kg)
DiBP	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 45.0 µg/kg)	< LOD (LOD = 24.2 µg/kg)	< LOQ (LOQ = 15.2 µg/kg)	< LOD (LOD = 30.6 µg/kg)
DBP	< LOD (LOD = 6.1 µg/kg)	< LOD (LOD = 51.6 µg/kg)	< LOD (LOD = 37.6 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 27.9 µg/kg)
DPP	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 29.5 µg/kg)	< LOD (LOD = 15.3 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 33.9 µg/kg)
DHP	< LOD (LOD = 6.0 µg/kg)	< LOD (LOD = 46.8 µg/kg)	< LOD (LOD = 27.2 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 22.8 µg/kg)
BBP	< LOD (LOD = 16.1 µg/kg)	< LOD (LOD = 40.5 µg/kg)	< LOD (LOD = 28.4 µg/kg)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 32.9 µg/kg)
DCHP	< LOD (LOD = 8.3 µg/kg)	< LOD (LOD = 26.6 µg/kg)	< LOD (LOD = 25.5 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 46.4 µg/kg)
DEHP	< LOQ (LOQ = 39.5 µg/kg)	< LOD (LOD = 94.2 µg/kg)	64.9 µg/kg (not confirmed)	< LOQ (LOQ = 43.5 µg/kg)	< LOD (LOD = 84.4 µg/kg)
DOP	< LOD (LOD = 10.3 µg/kg)	< LOD (LOD = 86.5 µg/kg)	< LOD (LOD = 52.7 µg/kg)	< LOD (LOD = 8.8 µg/kg)	< LOD (LOD = 30.4 µg/kg)
DiNP	< LOD (LOD = 518.1 µg/kg)	< LOD (LOD = 3946.7 µg/kg)	< LOD (LOD = 2769.6 µg/kg)	< LOD (LOD = 565.3 µg/kg)	< LOD (LOD = 729.8 µg/kg)
DiDP	< LOD (LOD = 269.6 µg/kg)	< LOD (LOD = 3142.5 µg/kg)	< LOD (LOD = 2152.0 µg/kg)	< LOD (LOD = 384.6 µg/kg)	< LOD (LOD = 1046.4 µg/kg)
DDP	< LOD (LOD = 13.0 µg/kg)	< LOD (LOD = 152.0 µg/kg)	< LOD (LOD = 191.2 µg/kg)	< LOD (LOD = 31.4 µg/kg)	< LOD (LOD = 65.1 µg/kg)

Table 44 continued. Retail food sample results

	S09-017439	S09-017440	S09-017441	S09-017442	S09-017443
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 16.4 µg/kg)	< LOD (LOD = 11.9 µg/kg)
DEP	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 10.8 µg/kg)	< LOD (LOD = 10.5 µg/kg)	< LOQ (LOQ = 47.5 µg/kg)	16.4 µg/kg (not confirmed)
DiPP	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 6.1 µg/kg)	39.4 µg/kg (not confirmed)	38.1 µg/kg (not confirmed)
DAP	< LOD (LOD = 19.2 µg/kg)	< LOD (LOD = 24.8 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 26.8 4 µg/kg)	< LOD (LOD = 109.7 µg/kg)
DiBP	< LOD (LOD = 6.5 µg/kg)	46.1 µg/kg (not confirmed)	< LOQ (LOQ = 5.0 µg/kg)	6.7 µg/kg (not confirmed)	< LOD (LOD = 24.7 µg/kg)
DBP	< LOD (LOD = 6.5 µg/kg)	< LOD (LOD = 106.6 µg/kg)	< LOQ (LOQ = 7.3 µg/kg)	< LOD (LOD = 56.7 µg/kg)	< LOQ (LOQ = 7.3 µg/kg)
DPP	271.5 µg/kg (not confirmed)	< LOD (LOD = 88.3 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 20.5 µg/kg)	< LOD (LOD = 31.0 µg/kg)
DHP	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 108.9 µg/kg)	< LOD (LOD = 5.3 µg/kg)	509.8 µg/kg (not confirmed)	< LOD (LOD = 27.5 µg/kg)
BBP	< LOD (LOD = 17.0 µg/kg)	No data	< LOD (LOD = 14.0 µg/kg)	101.6 µg/kg (not confirmed)	< LOD (LOD = 69.8 µg/kg)
DCHP	< LOD (LOD = 13.5 µg/kg)	< LOD (LOD = 53.3 µg/kg)	< LOD (LOD = 13.0 µg/kg)	< LOD (LOD = 53.3 µg/kg)	< LOQ (LOQ = 128.3 µg/kg)
DEHP	125.0 µg/kg (not confirmed)	196.1 µg/kg (not confirmed)	94.1 µg/kg (not confirmed)	199.5 µg/kg (not confirmed)	24.7 µg/kg (not confirmed)
DOP	< LOD (LOD = 21.8 µg/kg)	< LOD (LOD = 85.2 µg/kg)	< LOD (LOD = 23.0 µg/kg)	< LOD (LOD = 37.1 µg/kg)	< LOD (LOD = 49.8 µg/kg)
DiNP	919.0 µg/kg (not confirmed)	< LOD (LOD = 1630.7 µg/kg)	< LOD (LOD = 272.8 µg/kg)	< LOD (LOD = 1636.6 µg/kg)	< LOD (LOD = 975.8 µg/kg)
DiDP	< LOD (LOD = 383.7 µg/kg)	< LOD (LOD = 1922.9 µg/kg)	< LOD (LOD = 181.8 µg/kg)	< LOD (LOD = 510.6 µg/kg)	< LOD (LOD = 777.7 µg/kg)
DDP	< LOD (LOD = 50.8 µg/kg)	< LOD (LOD = 100.8 µg/kg)	< LOD (LOD = 22.0 µg/kg)	< LOD (LOD = 43.7 µg/kg)	< LOD (LOD = 37.3 µg/kg)

Table 44 continued. Retail food sample results

	S09-017444	S09-017445	S09-017446	S09-017447	S09-017448
DMP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 37.8 µg/kg)	< LOD (LOD = 68.4 µg/kg)	No data	< LOD (LOD = 53.6 µg/kg)
DEP	< LOD (LOD = 9.6 µg/kg)	< LOD (LOD = 43.3 µg/kg)	< LOD (LOD = 78.2 µg/kg)	No data	< LOD (LOD = 59.4 µg/kg)
DiPP	9.4 µg/kg (not confirmed)	< LOD (LOD = 33.7 µg/kg)	< LOD (LOD = 60.0 µg/kg)	No data	< LOD (LOD = 47.5 µg/kg)
DAP	< LOD (LOD = 13.4 µg/kg)	< LOD (LOD = 129.8 µg/kg)	< LOD (LOD = 256.1 µg/kg)	No data	< LOD (LOD = 119.1 µg/kg)
DiBP	< LOQ (LOQ = 5.0 µg/kg)	< LOD (LOD = 27.9 µg/kg)	< LOQ (LOQ = 222.7 µg/kg)	No data	< LOD (LOD = 33.5 µg/kg)
DBP	< LOD (LOD = 4.1 µg/kg)	< LOQ (LOQ = 81.3 µg/kg)	< LOQ (LOQ = 213.3 µg/kg)	No data	< LOD (LOD = 38.4 µg/kg)
DPP	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 29.4 µg/kg)	< LOD (LOD = 60.6 µg/kg)	No data	No data
DHP	< LOD (LOD = 10.7 µg/kg)	< LOQ (LOQ = 81.1 µg/kg)	< LOD (LOD = 50.1 µg/kg)	No data	< LOD (LOD = 68.8 µg/kg)
BBP	< LOD (LOD = 15.1 µg/kg)	< LOD (LOD = 72.6 µg/kg)	< LOD (LOD = 121.0 µg/kg)	No data	< LOD (LOD = 132.8 µg/kg)
DCHP	< LOD (LOD = 41.5 µg/kg)	< LOD (LOD = 25.4 µg/kg)	< LOD (LOD = 62.4 µg/kg)	No data	< LOD (LOD = 116.9 µg/kg)
DEHP	104.6 µg/kg (not confirmed)	No data	No data	No data	No data
DOP	< LOD (LOD = 17.1 µg/kg)	< LOD (LOD = 26.1 µg/kg)	< LOD (LOD = 79.3 µg/kg)	No data	< LOD (LOD = 115.7 µg/kg)
DiNP	< LOD (LOD = 268.7 µg/kg)	< LOD (LOD = 1527.2 µg/kg)	No data	No data	No data
DiDP	< LOD (LOD = 120.7 µg/kg)	< LOD (LOD = 1243.8 µg/kg)	No data	No data	No data
DDP	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 39.5 µg/kg)	< LOD (LOD = 150.1 µg/kg)	No data	No data

Table 44 continued. Retail food sample results

	S09-017449	**S09-017450**	**S09-017451**	**S09-017452**	**S09-017453**
DMP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 104.5 µg/kg)	< LOD (LOD = 83.8 µg/kg)	< LOD (LOD = 40.5 µg/kg)	< LOD (LOD = 14.6 µg/kg)
DEP	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 126.5 µg/kg)	< LOD (LOD = 95.2 µg/kg)	< LOD (LOD = 36.3 µg/kg)	< LOD (LOD = 40.6 µg/kg)
DiPP	< LOD (LOD = 4.7 µg/kg)	< LOD (LOD = 78.3 µg/kg)	< LOD (LOD = 84.0 µg/kg)	< LOD (LOD = 4.7 µg/kg)	18.9 µg/kg (not confirmed)
DAP	< LOD (LOD = 18.9 µg/kg)	No data	< LOD (LOD = 277.5 µg/kg)	< LOD (LOD = 96.6 µg/kg)	< LOD (LOD = 56.1 µg/kg)
DiBP	< LOD (LOD = 4.2 µg/kg)	< LOQ (LOQ = 173.1 µg/kg)	< LOD (LOD = 66.7 µg/kg)	< LOD (LOD = 25.7 µg/kg)	9.0 µg/kg (not confirmed)
DBP	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 54.7 µg/kg)	< LOD (LOD = 49.2 µg/kg)	< LOD (LOD = 28.8 µg/kg)	< LOD (LOD = 49.1 µg/kg)
DPP	< LOD (LOD = 52.0 µg/kg)	No data	< LOD (LOD = 76.0 µg/kg)	< LOD (LOD = 65.8 µg/kg)	< LOD (LOD = 34.1 µg/kg)
DHP	< LOD (LOD = 8.1 µg/kg)	< LOD (LOD = 48.7 µg/kg)	< LOD (LOD = 85.9 µg/kg)	< LOD (LOD = 24.1 µg/kg)	< LOD (LOD = 9.9 µg/kg)
BBP	< LOD (LOD = 18.6 µg/kg)	< LOD (LOD = 124.8 µg/kg)	< LOD (LOD = 193.6 µg/kg)	< LOD (LOD = 66.4 µg/kg)	20.0 µg/kg (not confirmed)
DCHP	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 73.2 µg/kg)	< LOD (LOD = 116.0 µg/kg)	< LOD (LOD = 30.9 µg/kg)	< LOD (LOD = 16.0 µg/kg)
DEHP	< LOQ (LOQ = 36.6 µg/kg)	No data	105.9 µg/kg (not confirmed)	< LOQ (LOQ = 102.5 µg/kg)	50.5 µg/kg (not confirmed)
DOP	< LOD (LOD = 18.7 µg/kg)	< LOD (LOD = 76.5 µg/kg)	< LOD (LOD = 99.0 µg/kg)	< LOD (LOD = 74.7 µg/kg)	< LOD (LOD = 69.8 µg/kg)
DiNP	< LOD (LOD = 405.9 µg/kg)	No data	No data	< LOD (LOD = 3065.0 µg/kg)	< LOD (LOD = 1084.5 µg/kg)
DiDP	< LOD (LOD = 574.0 µg/kg)	No data	No data	< LOD (LOD = 1724.8 µg/kg)	< LOD (LOD = 181.8 µg/kg)
DDP	< LOD (LOD = 19.1 µg/kg)	< LOD (LOD = 108.9 µg/kg)	< LOD (LOD = 110.7 µg/kg)	< LOD (LOD = 36.4 µg/kg)	< LOD (LOD = 29.7 µg/kg)

Table 44 continued. Retail food sample results

	S09-017454	S09-017455	S09-017456	S09-017457	S09-017458
DMP	< LOD (LOD = 17.9 µg/kg)	< LOD (LOD = 106.2 µg/kg)	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 61.1 µg/kg)	< LOD (LOD = 3.8 9 µg/kg)
DEP	< LOD (LOD = 21.9 µg/kg)	< LOD (LOD = 148.9 µg/kg)	< LOD (LOD = 48.1 µg/kg)	< LOD (LOD = 49.1 µg/kg)	< LOD (LOD = 14.8 µg/kg)
DiPP	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 102.4 µg/kg)	68.6 µg/kg (not confirmed)	< LOD (LOD = 34.8 µg/kg)	< LOD (LOD = 10.9 µg/kg)
DAP	< LOD (LOD = 45.1 µg/kg)	No data	< LOD (LOD = 119.2 µg/kg)	< LOD (LOD = 123.8 µg/kg)	< LOD (LOD = 35.8 µg/kg)
DiBP	< LOD (LOD = 11.3 µg/kg)	< LOD (LOD = 143.0 µg/kg)	< LOD (LOD = 27.0 µg/kg)	< LOD (LOD = 27.5 µg/kg)	< LOD (LOD = 11.1 µg/kg)
DBP	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 127.1 µg/kg)	50.9 µg/kg (not confirmed)	< LOD (LOD = 21.8 µg/kg)	< LOD (LOD = 9.1 µg/kg)
DPP	< LOD (LOD = 30.4 µg/kg)	< LOD (LOD = 179.5 µg/kg)	< LOD (LOD = 21.7 µg/kg)	< LOD (LOD = 50.7 µg/kg)	< LOD (LOD = 28.1 µg/kg)
DHP	< LOD (LOD = 12.8 µg/kg)	< LOD (LOD = 84.7 µg/kg)	< LOD (LOD = 18.3 µg/kg)	< LOD (LOD = 18.1 µg/kg)	< LOD (LOD = 11.5 µg/kg)
BBP	< LOD (LOD = 33.0 µg/kg)	< LOD (LOD = 245.3 µg/kg)	35.8 µg/kg (not confirmed)	< LOD (LOD = 50.4 µg/kg)	< LOD (LOD = 16.6 µg/kg)
DCHP	< LOD (LOD = 23.2 µg/kg)	< LOD (LOD = 168.2 µg/kg)	< LOD (LOD = 49.9 µg/kg)	< LOD (LOD = 36.3 µg/kg)	< LOD (LOD = 15.5 µg/kg)
DEHP	< LOQ (LOQ = 44.1 µg/kg)	< LOD (LOD = 182.0 µg/kg)	97.4 µg/kg	< LOQ (LOQ = 109.2 µg/kg)	76.9 µg/kg (not confirmed)
DOP	< LOD (LOD = 27.3 µg/kg)	< LOD (LOD = 203.9 µg/kg)	< LOD (LOD = 55.2 µg/kg)	< LOD (LOD = 77.1 µg/kg)	< LOD (LOD = 49.3 µg/kg)
DiNP	< LOD (LOD = 887.2 µg/kg)	No data	< LOD (LOD = 1511.0 µg/kg)	< LOD (LOD = 1903.8 µg/kg)	< LOD (LOD = 611.6 µg/kg)
DiDP	< LOD (LOD = 882.3 µg/kg)	No data	< LOD (LOD = 236.8 µg/kg)	< LOD (LOD = 2052.4 µg/kg)	< LOD (LOD = 1342.0 µg/kg)
DDP	< LOD (LOD = 33.9 µg/kg)	< LOD (LOD = 238.3 µg/kg)	< LOD (LOD = 22.7 µg/kg)	< LOD (LOD = 41.1 µg/kg)	< LOD (LOD = 98.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017459	S09-017460	S09-017461	S09-017462	S09-017463
DMP	< LOD (LOD = 33.8 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 29.6 µg/kg)	< LOD (LOD = 30.4 µg/kg)	< LOD (LOD = 25.2 µg/kg)
DEP	< LOD (LOD = 52.7 µg/kg)	< LOD (LOD = 2.4 µg/kg)	< LOD (LOD = 20.2 µg/kg)	< LOD (LOD = 22.2 µg/kg)	< LOD (LOD = 25.8 µg/kg)
DiPP	< LOD (LOD = 34.6 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 10.4 µg/kg)	< LOD (LOD = 21.2 µg/kg)	< LOD (LOD = 19.2 µg/kg)
DAP	< LOD (LOD = 134.1 µg/kg)	< LOD (LOD = 6.3 µg/kg)	< LOD (LOD = 38.6 µg/kg)	< LOD (LOD = 105.0 µg/kg)	< LOD (LOD = 52.0 µg/kg)
DiBP	< LOD (LOD = 39.9 µg/kg)	< LOQ (LOQ = 9.5 µg/kg)	< LOD (LOD = 18.7 µg/kg)	< LOD (LOD = 45.9 µg/kg)	< LOD (LOD = 19.3 µg/kg)
DBP	< LOD (LOD = 39.5 µg/kg)	< LOQ (LOQ = 11.6 µg/kg)	< LOD (LOD = 16.3 µg/kg)	< LOQ (LOQ = 41.2 3 µg/kg)	No data
DPP	No data	< LOD (LOD = 4.5 µg/kg)	No data	No data	No data
DHP	< LOD (LOD = 70.6 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 10.7 µg/kg)	< LOD (LOD = 33.9 µg/kg)	< LOD (LOD = 41.4 µg/kg)
BBP	< LOD (LOD = 89.0 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 23.5 µg/kg)	< LOD (LOD = 62.6 µg/kg)	< LOD (LOD = 47.9 µg/kg)
DCHP	No data	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 14.9 µg/kg)	No data	< LOD (LOD = 53.3 µg/kg)
DEHP	No data	< LOQ (LOQ = 38.5 µg/kg)	53.5 µg/kg (not confirmed)	No data	< LOQ (LOQ = 117.9 µg/kg)
DOP	No data	< LOD (LOD = 18.7 µg/kg)	< LOD (LOD = 25.8 µg/kg)	< LOD (LOD = 164.3 µg/kg)	< LOD (LOD = 229.5 µg/kg)
DiNP	< LOD (LOD = 3266.6 µg/kg)	< LOD (LOD = 284.7 µg/kg)	< LOD (LOD = 829.5 µg/kg)	< LOD (LOD = 2077.2 µg/kg)	No data
DiDP	< LOD (LOD = 3437.2 µg/kg)	< LOD (LOD = 213.7 µg/kg)	< LOD (LOD = 564.0 µg/kg)	< LOD (LOD = 1999.8 µg/kg)	No data
DDP	No data	< LOD (LOD = 35.7 µg/kg)	< LOD (LOD = 38.5 µg/kg)	No data	No data

Table 44 continued. Retail food sample results

	S09-017464	S09-017465	S09-017466	S09-017467	S09-017468
DMP	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 5.4 µg/kg)
DEP	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 5.3 µg/kg)	< LOD (LOD = 2.3 µg/kg)	2.7 µg/kg (not confirmed)	< LOD (LOD = 37.3 µg/kg)
DiPP	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 3.4 µg/kg)	2.6 µg/kg (not confirmed)	< LOD (LOD = 1.5 µg/kg)	29.3 µg/kg (not confirmed)
DAP	< LOD (LOD = 13.7 µg/kg)	< LOD (LOD = 7.3 µg/kg)	< LOD (LOD = 5.9 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 47.1 µg/kg)
DiBP	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 1.5 µg/kg)	17.8 µg/kg	< LOD (LOD = 1.8 µg/kg)	< LOD (LOD = 13.6 µg/kg)
DBP	< LOQ (LOQ = 9.9 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 6.0 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 18.0 µg/kg)
DPP	No data	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 1.6 µg/kg)	< LOD (LOD = 10.9 µg/kg)	< LOD (LOD = 276.8 µg/kg)
DHP	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 1.7 µg/kg)	< LOD (LOD = 29.2 µg/kg)	< LOD (LOD = 29.2 µg/kg)
BBP	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 10.5 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 5.5 µg/kg)	< LOD (LOD = 25.0 µg/kg)
DCHP	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 9.1 µg/kg)
DEHP	126.6 µg/kg	25.8 µg/kg (not confirmed)	< LOD (LOD = 39.4 µg/kg)	< LOQ (LOQ = 131.3 µg/kg)	< LOD (LOD = 25.4 µg/kg)
DOP	< LOD (LOD = 8.7 µg/kg)	< LOD (LOD = 20.4 µg/kg)	< LOD (LOD = 12.0 µg/kg)	< LOD (LOD = 16.8 µg/kg)	< LOD (LOD = 22.8 µg/kg)
DiNP	< LOD (= 231.9 µg/kg)	< LOD (LOD = 379.4 µg/kg)	< LOD (LOD = 596.5 µg/kg)	< LOD (LOD = 336.9 µg/kg)	< LOD (LOD = 1421.3 µg/kg)
DiDP	< LOD (LOD = 147.2 µg/kg)	< LOD (LOD = 214.3 µg/kg)	< LOD (LOD = 305.1 µg/kg)	< LOD (LOD = 184.2 µg/kg)	< LOQ (LOQ = 613.9 µg/kg)
DDP	< LOD (LOD = 32.4 µg/kg)	< LOD (LOD = 61.1 µg/kg)	< LOD (LOD = 34.7 µg/kg)	< LOD (LOD = 15.6 µg/kg)	< LOD (LOD = 82.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017469	S09-017470	S09-017471	S09-017472	S09-017473
DMP	< LOD (LOD = 3.8 µg/kg)	< LOD (LOD = 6.0 µg/kg)	< LOD (LOD = 12.9 µg/kg)	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 3.8 µg/kg)
DEP	< LOD (LOD = 86.6 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 12.6 µg/kg)	< LOD (LOD = 18.4 µg/kg)	< LOD (LOD = 7.8 µg/kg)
DiPP	< LOD (LOD = 15.0 µg/kg)	< LOD (LOD = 1.3 µg/kg)	< LOD (LOD = 9.4 µg/kg)	< LOD (LOD = 10.2 µg/kg)	< LOD (LOD = 6.7 µg/kg)
DAP	< LOD (LOD = 93.1 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 53.7 µg/kg)	< LOD (LOD = 53.7 µg/kg)	< LOD (LOD = 14.0 µg/kg)
DiBP	< LOD (LOD = 1.8 µg/kg)	< LOQ (LOQ = 6.0 µg/kg)	< LOD (LOD = 5.1 µg/kg)	< LOD (LOD = 7.7 µg/kg)	< LOD (LOD = 6.0 µg/kg)
DBP	40.1 µg/kg (not confirmed)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 11.2 µg/kg)	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 2.2 µg/kg)
DPP	< LOD (LOD = 27.5 µg/kg)	< LOD (LOD = 10.9 µg/kg)	< LOD (LOD = 9.4 µg/kg)	< LOD (LOD = 11.3 µg/kg)	< LOD (LOD = 13.8 µg/kg)
DHP	< LOD (LOD = 28.4 µg/kg)	< LOD (LOD = 29.2 µg/kg)	< LOD (LOD = 12.0 µg/kg)	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 8.0 µg/kg)
BBP	< LOD (LOD = 81.3 µg/kg)	< LOD (LOD = 5.2 µg/kg)	< LOD (LOD = 60.5 µg/kg)	< LOD (LOD = 53.4 µg/kg)	< LOD (LOD = 19.3 µg/kg)
DCHP	< LOD (LOD = 45.8 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 13.3 µg/kg)	< LOD (LOD = 15.2 µg/kg)	< LOD (LOD = 20.6 µg/kg)
DEHP	< LOQ (LOQ = 131.3 µg/kg)	< LOQ (LOQ = 131.3 µg/kg)	< LOQ (LOQ = 19.0 µg/kg)	20.0 µg/kg	< LOQ (LOQ = 19.0 µg/kg)
DOP	< LOD (LOD = 26.8 µg/kg)	< LOQ (LOQ = 56.0 µg/kg)	< LOD (LOD = 110.4 µg/kg)	< LOD (LOD = 70.7 µg/kg)	< LOD (LOD = 24.4 µg/kg)
DiNP	< LOD (LOD = 267.1 µg/kg)	< LOD (LOD = 5000.8 µg/kg)	< LOD (LOD = 156.7 µg/kg)	< LOD (LOD = 271.1 µg/kg)	< LOD (LOD = 697.8 µg/kg)
DiDP	< LOQ (LOQ = 613.9 µg/kg)	< LOD (LOD = 4999.5 µg/kg)	< LOD (LOD = 125.0 µg/kg)	< LOD (LOD = 243.2 µg/kg)	< LOD (LOD = 666.6 µg/kg)
DDP	< LOD (LOD = 23.9 µg/kg)	< LOQ (LOQ = 10.1 µg/kg)	< LOD (LOD = 16.9 µg/kg)	< LOD (LOD = 6.7 µg/kg)	< LOD (LOD = 18.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017474	S09-017475	S09-017476	S09-017477	S09-017478
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.0 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 5.2 µg/kg)
DEP	< LOD (LOD = 6.5 µg/kg)	6.0 µg/kg (not confirmed)	< LOQ (LOQ = 6.6 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 5.0 µg/kg)
DiPP	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 3.8 µg/kg)
DAP	< LOD (LOD = 13.7 µg/kg)	< LOD (LOD = 17.9 µg/kg)	< LOD (LOD = 5.6 µg/kg)	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 5.4 µg/kg)
DiBP	10.6 µg/kg (not confirmed)	< LOQ (LOQ = 6.0 µg/kg)	< LOQ (LOQ = 6.0 µg/kg)	11.0 µg/kg (not confirmed)	< LOD (LOD = 1.5 µg/kg)
DBP	12.7 µg/kg (not confirmed)	< LOD (LOD = 3.5 µg/kg)	< LOQ (LOQ = 11.7 µg/kg)	22.2 µg/kg (not confirmed)	< LOD (LOD = 2.2 µg/kg)
DPP	41.2 µg/kg (not confirmed)	< LOD (LOD = 13.5 µg/kg)	< LOQ (LOQ = 36.3 µg/kg)	< LOD (LOD = 10.9 µg/kg)	< LOD (LOD = 3.7 µg/kg)
DHP	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 29.2 µg/kg)	< LOD (LOD = 29.2 µg/kg)	< LOD (LOD = 4.4 µg/kg)
BBP	< LOD (LOD = 10.2 µg/kg)	< LOD (LOD = 8.0 µg/kg)	< LOQ (LOQ = 9.1 µg/kg)	< LOD (LOD = 9.5 µg/kg)	< LOD (LOD = 9.8 µg/kg)
DCHP	< LOD (LOD = 20.0 µg/kg)	< LOD (LOD = 9.1 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 12.4 µg/kg)	< LOD (LOD = 8.2 µg/kg)
DEHP	No data	< LOD (LOD = 39.4 µg/kg)	< LOQ (LOQ = 131.3 µg/kg)	< LOD (LOD = 270.0 µg/kg)	< LOQ (LOQ = 19.0 µg/kg)
DOP	No data	< LOD (LOD = 16.8 µg/kg)	< LOQ (LOQ = 56.0 µg/kg)	< LOD (LOD = 267.8 µg/kg)	< LOD (LOD = 17.9 µg/kg)
DiNP	No data	< LOD (LOD = 267.1 µg/kg)	< LOD (LOD = 5000.8 µg/kg)	< LOD (LOD = 5000.8 µg/kg)	< LOD (LOD = 663.8 µg/kg)
DiDP	No data	< LOD (LOD = 184.2 µg/kg)	< LOD (LOD = 4999.5 µg/kg)	< LOD (LOD = 4999.5 µg/kg)	< LOD (LOD = 617.6 µg/kg)
DDP	No data	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 282.3 µg/kg)	< LOD (LOD = 9.2 µg/kg)

Table 44 continued. Retail food sample results

	S09-017479	S09-017480	S09-017481	S09-017482	S09-017483
DMP	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 19.7 µg/kg)	< LOD (LOD = 44.8 µg/kg)	< LOD (LOD = 7.6 µg/kg)	< LOD (LOD = 3.6 µg/kg)
DEP	< LOD (LOD = 7.7 µg/kg)	< LOD (LOD = 31.5 µg/kg)	< LOD (LOD = 61.0 µg/kg)	< LOD (LOD = 18.8 µg/kg)	< LOD (LOD = 16.8 µg/kg)
DiPP	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 16.7 µg/kg)	< LOD (LOD = 43.3 µg/kg)	< LOD (LOD = 12.5 µg/kg)	23.3 µg/kg (not confirmed)
DAP	< LOD (LOD = 11.5 µg/kg)	< LOD (LOD = 32.2 µg/kg)	< LOD (LOD = 73.2 µg/kg)	< LOD (LOD = 67.1 µg/kg)	< LOD (LOD = 23.0 µg/kg)
DiBP	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 4.1 µg/kg)	22.9 µg/kg (not confirmed)	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 6.1 µg/kg)
DBP	< LOD (LOD = 2.8 µg/kg)	< LOD (LOD = 6.2 µg/kg)	31.0 µg/kg (not confirmed)	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 14.8 µg/kg)
DPP	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 11.8 µg/kg)	< LOD (LOD = 46.4 µg/kg)	< LOD (LOD = 4.9 µg/kg)	< LOD (LOD = 13.8 µg/kg)
DHP	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 17.2 µg/kg)	< LOD (LOD = 73.7 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 29.2 µg/kg)
BBP	< LOD (LOD = 13.4 µg/kg)	< LOD (LOD = 66.0 µg/kg)	< LOD (LOD = 191.1 µg/kg)	< LOD (LOD = 16.5 µg/kg)	< LOD (LOD = 18.8 µg/kg)
DCHP	< LOD (LOD = 15.4 µg/kg)	< LOD (LOD = 20.0 µg/kg)	< LOD (LOD = 155.9 µg/kg)	< LOD (LOD = 18.4 µg/kg)	< LOD (LOD = 25.9 µg/kg)
DEHP	141.8 µg/kg	< LOQ (LOQ = 19.0 µg/kg)	315.8 µg/kg (not confirmed)	< LOQ (LOQ = 19.0 µg/kg)	< LOQ (LOQ = 131.3 µg/kg)
DOP	< LOD (LOD = 23.4 µg/kg)	< LOD (LOD = 14.0 µg/kg)	< LOD (LOD = 119.4 µg/kg)	< LOD (LOD = 49.4 µg/kg)	< LOD (LOD = 18.2 µg/kg)
DiNP	< LOD (LOD = 257.2 µg/kg)	< LOD (LOD = 229.2 µg/kg)	No data	< LOD (LOD = 266.3 µg/kg)	< LOD (LOD = 673.6 µg/kg)
DiDP	< LOD (LOD = 257.1 µg/kg)	< LOD (LOD = 98.4 µg/kg)	No data	< LOD (LOD = 255.7 µg/kg)	< LOD (LOD = 423.0 µg/kg)
DDP	< LOD (LOD = 9.0 µg/kg)	< LOD (LOD = 46.2 µg/kg)	No data	< LOD (LOD = 82.3 µg/kg)	< LOD (LOD = 43.5 µg/kg)

Table 44 continued. Retail food sample results

	S09-017484	S09-017485	S09-017486	S09-017487	S09-017488
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 6.9 µg/kg)	< LOD (LOD = 15.1 µg/kg)	< LOD (LOD = 13.7 µg/kg)
DEP	1.2 µg/kg (not confirmed)	1.5 µg/kg (not confirmed)	< LOD (LOD = 40.1 µg/kg)	6.7 µg/kg (not confirmed)	88.6 µg/kg (not confirmed)
DiPP	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 30.7 µg/kg)	< LOD (LOD = 9.9 µg/kg)	< LOD (LOD = 17.7 µg/kg)
DAP	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 7.9 µg/kg)	< LOD (LOD = 112.9 µg/kg)	< LOD (LOD = 16.8 µg/kg)	< LOD (LOD = 51.9 µg/kg)
DiBP	< LOQ (LOQ = 16.7 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 40.5 µg/kg)	38.9 µg/kg (not confirmed)	< LOD (LOD = 52.6 µg/kg)
DBP	< LOQ (LOQ = 21.2 µg/kg)	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 57.7 µg/kg)	< LOD (LOD = 132.5 µg/kg)
DPP	< LOD (LOD = 93.5 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 23.5 µg/kg)	268.3 µg/kg (not confirmed)	< LOD (LOD = 48.3 µg/kg)
DHP	< LOD (LOD = 16.7 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 32.7 µg/kg)	< LOD (LOD = 24.8 µg/kg)	< LOD (LOD = 39.3 µg/kg)
BBP	79.8 µg/kg (not confirmed)	< LOD (LOD = 5.4 µg/kg)	< LOD (LOD = 38.3 µg/kg)	< LOD (LOD = 28.1 µg/kg)	< LOD (LOD = 40.2 µg/kg)
DCHP	< LOD (LOD = 76.8 µg/kg)	< LOD (LOD = 4.6 µg/kg)	< LOD (LOD = 41.7 µg/kg)	< LOD (LOD = 39.3 µg/kg)	< LOD (LOD = 31.4 µg/kg)
DEHP	963.2 µg/kg (not confirmed)	< LOD (LOD = 20.2 µg/kg)	< LOQ (LOQ = 67.5 µg/kg)	< LOQ (LOQ = 67.5 µg/kg)	< LOD (LOD = 59.6 µg/kg)
DOP	< LOD (LOD = 44.6 µg/kg)	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 57.4 µg/kg)	< LOD (LOD = 55.6 µg/kg)	< LOD (LOD = 44.6 µg/kg)
DiNP	2837.7 µg/kg (not confirmed)	< LOD (LOD = 401.2 µg/kg)	< LOD (LOD = 1034.6 µg/kg)	< LOD (LOD = 2727.7 µg/kg)	< LOD (LOD = 1944.7 µg/kg)
DiDP	< LOD (LOD = 739.4 µg/kg)	< LOD (LOD = 196.7 µg/kg)	< LOD (LOD = 1097.5 µg/kg)	< LOD (LOD = 1371.8 µg/kg)	< LOD (LOD = 2249.8 µg/kg)
DDP	< LOD (LOD = 102.2 5 µg/kg)	< LOD (LOD = 14.3 µg/kg)	< LOD (LOD = 72.6 µg/kg)	< LOD (LOD = 66.4 µg/kg)	< LOD (LOD = 105.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017489	**S09-017490**	**S09-017491**	**S09-017492**	**S09-017493**
DMP	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 3.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 7.2 µg/kg)
DEP	< LOD (LOD = 1.4 µg/kg)	4.4 µg/kg (not confirmed)	0.7 µg/kg (not confirmed)	< LOD (LOD = 1.5 µg/kg)	< LOQ (LOQ = 4.9 µg/kg)
DiPP	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 2.4 µg/kg)	< LOD (LOD = 1.3 µg/kg)	< LOD (LOD = 2.7 µg/kg)	< LOD (LOD = 6.1 µg/kg)
DAP	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 9.2 µg/kg)	0.9 µg/kg (not confirmed)	< LOD (LOD = 3.3 µg/kg)	< LOD (LOD = 11.1 µg/kg)
DiBP	< LOQ (LOQ = 16.7 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 5.0 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOQ = 9.5 µg/kg)
DBP	< LOQ (LOQ = 21.2 µg/kg)	< LOQ (LOQ = 21.2 µg/kg)	< LOD (LOD = 6.4 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 9.0 µg/kg)
DPP	< LOD (LOD = 9.8 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 0.9 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 8.7 µg/kg)
DHP	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 1.0 µg/kg)	< LOD (LOD = 2.2 µg/kg)	< LOD (LOD = 13.5 µg/kg)
BBP	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 7.2 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 15.4 µg/kg)
DCHP	< LOD (LOD = 13.5 µg/kg)	< LOD (LOD = 5.9 µg/kg)	< LOD (LOD = 1.4 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 16.0 µg/kg)
DEHP	442.0 µg/kg (not confirmed)	< LOD (LOD = 20.2 µg/kg)	< LOQ (LOQ = 67.5 µg/kg)	44.6 µg/kg	< LOQ (LOQ = 38.5 µg/kg)
DOP	< LOD (LOD = 22.2 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 4.0 µg/kg)	< LOD (LOD = 7.1 µg/kg)	< LOD (LOD = 21.3 µg/kg)
DiNP	2071.3 µg/kg (not confirmed)	< LOD (LOD = 303.4 µg/kg)	299.1 µg/kg (not confirmed)	< LOD (LOD = 344.3 µg/kg)	< LOQ (LOQ = 1706.8 µg/kg)
DiDP	< LOD (LOD = 1478.7 µg/kg)	< LOD (LOD = 185.4 µg/kg)	< LOD (LOD = 48.1 µg/kg)	< LOD (LOD = 375.0 µg/kg)	< LOD (LOD = 1173.1 µg/kg)
DDP	< LOD (LOD = 228.0 µg/kg)	< LOD (LOD = 11.8 µg/kg)	< LOD (LOD = 4.8 µg/kg)	< LOD (LOD = 14.1 µg/kg)	< LOD (LOD = 69.8 µg/kg)

Table 44 continued. Retail food sample results

	S09-017494	**S09-017495**	**S09-017496**	**S09-017497**	**S09-017498**
DMP	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	< LOD (LOD = 0.7 µg/kg)	0.9 µg/kg (not confirmed)	< LOD (LOD = 0.7 µg/kg)
DEP	< LOD (LOD = 1.5 µg/kg)	< LOQ (LOQ = 4.9 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 1.5 µg/kg)
DiPP	< LOD (LOD = 1.2 µg/kg)	< LOD (LOD = 2.0 µg/kg)	< LOD (LOD = 1.1 µg/kg)	2.0 µg/kg (not confirmed)	< LOD (LOD = 2.0 µg/kg)
DAP	< LOD (LOD = 8.5 µg/kg)	< LOD (LOD = 7.7 µg/kg)	< LOD (LOD = 4.1 µg/kg)	< LOD (LOD = 3.0 µg/kg)	< LOD (LOD = 6.6 µg/kg)
DiBP	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 2.9 µg/kg)	< LOD (LOD = 2.9 µg/kg)	13.3 µg/kg (not confirmed)	< LOD (LOD = 2.9 µg/kg)
DBP	< LOD (LOD = 3.6 µg/kg)	< LOD (LOD = 3.5 µg/kg)	< LOD (LOD = 5.5 µg/kg)	15.3 µg/kg (not confirmed)	< LOD (LOD = 3.5 µg/kg)
DPP	< LOD (LOD = 9.2 µg/kg)	< LOD (LOD = 3.1 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 3.2 µg/kg)	< LOD (LOD = 4.9 µg/kg)
DHP	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 3.9 µg/kg)	< LOD (LOD = 1.5 µg/kg)	< LOD (LOD = 12.6 µg/kg)	< LOD (LOD = 5.4 µg/kg)
BBP	< LOD (LOD = 8.9 µg/kg)	< LOD (LOD = 6.2 µg/kg)	< LOD (LOD = 2.6 µg/kg)	< LOD (LOD = 10.1 µg/kg)	< LOD (LOD = 7.6 µg/kg)
DCHP	< LOD (LOD = 2.5 µg/kg)	< LOD (LOD = 4.2 µg/kg)	< LOD (LOD = 2.1 µg/kg)	< LOD (LOD = 19.5 µg/kg)	< LOD (LOD = 8.5 µg/kg)
DEHP	73.6 µg/kg	51.5 µg/kg	74.4 µg/kg	960.3 µg/kg	< LOQ (LOQ = 38.5 µg/kg)
DOP	< LOD (LOD = 13.8 µg/kg)	< LOD (LOD = 12.1 µg/kg)	< LOD (LOD = 11.6 µg/kg)	< LOD (LOD = 56.0 µg/kg)	< LOD (LOD = 9.0 µg/kg)
DiNP	< LOD (LOD = 157.6 µg/kg)	1999.0 µg/kg (same profile)	< LOD (LOD = 157.6 µg/kg)	< LOD (LOD = 843.9 µg/kg)	< LOD (LOD = 1307.9 µg/kg)
DiDP	< LOD (LOD = 105.5 µg/kg)	< LOD (LOD = 237.3 µg/kg)	< LOD (LOD = 75.0 µg/kg)	< LOD (LOD = 999.9 µg/kg)	< LOD (LOD = 416.6 µg/kg)
DDP	< LOD (LOD = 9.3 µg/kg)	< LOD (LOD = 11.0 µg/kg)	< LOD (LOD = 4.3 µg/kg)	< LOD (LOD = 72.8 µg/kg)	< LOD (LOD = 27.9 µg/kg)

Table 44 continued. Retail food sample results

	S09-017499
DMP	< LOD (LOD = 1.5 µg/kg)
DEP	< LOD (LOD = 1.5 µg/kg)
DiPP	< LOD (LOD = 2.3 µg/kg)
DAP	< LOD (LOD = 4.4 µg/kg)
DiBP	< LOD (LOD = 2.9 µg/kg)
DBP	< LOD (LOD = 4.0 µg/kg)
DPP	< LOD (LOD = 3.7 µg/kg)
DHP	< LOD (LOD = 3.7 µg/kg)
BBP	< LOD (LOD = 6.9 µg/kg)
DCHP	< LOD (LOD = 5.7 µg/kg)
DEHP	< LOQ (LOQ = 38.5 µg/kg)
DOP	< LOD (LOD = 6.9 µg/kg)
DiNP	< LOD (LOD = 724.2 µg/kg)
DiDP	< LOD (LOD = 220.6 µg/kg)
DDP	< LOD (LOD = 11.4 µg/kg)

ANNEX 1.

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Standard operating procedure for determination of seventeen phthalate diesters in foodstuffs

0. INTRODUCTION

Phthalates are a group of compounds produced in large volumes that are widely used as additives in plastics and consumer products. Some phthalates have been reported to be endocrine disruptors. They can migrate into foods from food packaging but have also been proposed to be present in food due to general contamination of the environment and food chain. The analytical method described allows the determination of 17 phthalate diesters in foodstuffs.

1. SCOPE

This document describes a method for the determination of 17 phthalate diesters in a range of foodstuffs. The concentration range for which the method is suitable is analyte specific. For dimethyl phthalate, diethyl phthalate, diisopropyl phthalate, diallyl phthalate, diisobutyl phthalate, di-n-butyl phthalate, di-n-pentyl phthalate, di-n-hexyl phthalate, benzyl butyl phthalate, dicyclohexyl phthalate, di-(2-ethylhexyl) phthalate, di-n-heptyl phthalate, di-n-octyl phthalate, n-octyl-n-decyl phthalate and di-n-decyl phthalate the method is appropriate for the quantitative determination of the analytes in the concentration range 0.05 – 0.5 mg/kg foodstuff. For diisononyl phthalate and diisodecyl phthalate the method is appropriate for the quantitative determination of the analytes in the concentration range 0.5 – 10 mg/kg.

2. REAGENTS

2.1 Analytes

2.1.1 Dimethyl phthalate

2.1.2 Diethyl phthalate

2.1.3 Diisopropyl phthalate

2.1.4 Diallyl phthalate

2.1.5 Diisobutyl phthalate

2.1.6 Di-n-butyl phthalate

2.1.7 Di-n-pentyl phthalate

2.1.8 Di-n-hexyl phthalate

2.1.9 Benzyl butyl phthalate

2.1.10 Dicyclohexyl phthalate

2.1.11 Di-(2-ethylhexyl) phthalate

2.1.12 Di-n-heptyl phthalate

2.1.13 Di-n-octyl phthalate

2.1.14 n-Octyl-n-decyl phthalate

2.1.15 Diisononyl phthalate

2.1.16 Diisodecyl phthalate

2.1.17 Di-n-decyl phthalate

2.2 Internal standards

2.2.1 d_4-Dimethyl phthalate

2.2.2 d_4-Diethyl phthalate

2.2.3 d_4-Di-n-propyl phthalate

2.2.4 d_4-Diisobutyl phthalate

2.2.5 d_4-Di-n-butyl phthalate

2.2.6 d_4-Di-n-pentyl phthalate

2.2.7 d_4-Di-n-hexyl phthalate

2.2.8 d_4-Benzyl butyl phthalate

2.2.9 d_4-Dicyclohexyl phthalate

2.2.10 d_4-Di-(2-ethylhexyl) phthalate

2.2.11 d_4-Di-n-heptyl phthalate

2.2.12 d_4-Di-n-octyl phthalate

2.2.13 d_4-Di-n-nonyl phthalate

2.3 Chemicals

2.3.1 Acetonitrile, HPLC fluorescence grade

2.3.2 Dichloromethane, HPLC grade

2.3.3 Aluminium oxide

Heat the aluminium oxide in a muffle furnace for a minimum of 4 hours at 500°C.

Add the muffled aluminium oxide to the solvent bottles (2.3.1 and 2.3.2) for at least 24 hours prior to use (0.5g per litre).

2.4 Solutions

2.4.1 Mixed phthalates stock solution

Weigh to the nearest 0.1 mg approximately 10 mg of dimethyl phthalate, diethyl phthalate, diisopropyl phthalate, diallyl phthalate, diisobutyl phthalate, di-n-butyl phthalate, di-n-pentyl phthalate, di-n-hexyl phthalate, benzyl butyl phthalate, dicyclohexyl phthalate, di-(2-ethylhexyl) phthalate, di-n-heptyl phthalate, di-n-octyl phthalate, n-octyl-n-decyl phthalate and di-n-decyl phthalate and 200 mg of diisononyl phthalate and diisodecyl phthalate in a 10 ml volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of each phthalate per millilitre of solution. Store the solution at a temperature below 5℃.

2.4.2 Mixed phthalates intermediate solution

Using a glass graduated pipette measure 1.5 ml of mixed phthalate stock solution (2.4.1) in a 10 ml volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of each phthalate per millilitre of solution. Store the solution at a temperature below 5℃.

2.4.3 Mixed phthalates working solution

Using a glass syringe add 150 µl of the mixed phthalate stock solution (2.4.1) to a 10 ml volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of each phthalate per millilitre of solution. Store the solution at a temperature below 5℃.

2.4.4 Internal standards stock solution

Weigh 10 mg of each d_4-labelled phthalate into a 10 ml volumetric flask. Make up to the mark with acetonitrile (2.3.1). Cap and mix thoroughly. Store the solution at a temperature below 5℃.

2.4.5 Internal standards working solution

Using a glass graduated pipette measure 1 ml of internal standards stock solution (2.4.4) in a 10 ml volumetric flask and make up to the mark with acetonitrile (2.3.1). Cap and mix thoroughly. Store the solution at a temperature below 5℃.

2.4.6 Extraction solution, acetonitrile: dichloromethane, 1:1 (v:v)

Using a measuring cylinder add acetonitrile (500 ml, 2.3.1) and dichloromethane (500 ml, 2.3.2) to a 1 L Duran bottle. Cap and mix thoroughly.

3. APPARATUS

NOTE: An instrument or item of apparatus is listed only where it is special or made to a particular specification, the usual laboratory glassware and equipment being assumed to be available.

All glassware should be muffled and/or solvent rinsed and overwrapped in aluminium foil prior to use.

3.1 Gas chromatograph, equipped with an automatic sampler and a mass spectrometric detector.

Appropriate operating conditions have to be established for the specific equipment used for the determination.

3.1.1 Gas chromatographic parameters

NOTE: The following column and parameters have been found to be suitable:

Column:	Zebron ZB-5ms (5% phenyl 95% dimethylpolysiloxane) 30 m x 0.25 mm id x 0.25 µm film thickness

GC parameters:	
Column oven	isothermal 3 minutes at 80°C, then at 10°C/minute to 320°C and held for 5 minutes
Split mode	splitless
Inlet temperature	280°C
Injection volume	1.0 µl (Merlin Micro syringe)
Carrier gas	helium
Flow rate	1.0 ml/minute
Transfer line temp.	280°C
MS detection:	
Mode	Selected ion monitoring (SIM) EI mode
SIM ions recorded	

Phthalate diester	Ions monitored
Dimethyl phthalate	77,163, 194
Diethyl phthalate	149, 177, 222
Diisopropyl phthalate	149, 192, 209
Diallyl phthalate	104, 149, 189
Diisobutyl phthalate	149, 205, 223
Di-n-butyl phthalate	149, 205, 223
Di-n-pentyl phthalate	149, 219, 237
Di-n-hexyl phthalate	149, 233, 251
Benzyl butyl phthalate	149, 206

Phthalate diester	Ions monitored
Dicyclohexyl phthalate	149, 167, 249
Di-(2-ethylhexyl) phthalate	149, 167, 279
Di-n-heptyl phthalate	149, 167, 265
Di-n-octyl phthalate	149, 167, 279
n-Octyl-n-decyl phthalate	149, 279, 307
Diisononyl phthalate	149, 167, 293
Diisodecyl phthalate	149, 167, 307
Di-n-decyl phthalate	149, 167, 307

Internal standard	Ions monitored
d_4-Dimethyl phthalate	167, 194
d_4-Diethyl phthalate	153, 181
d_4-Di-n-propyl phthalate	153, 213
d_4-Diisobutyl phthalate	153, 227
d_4-Di-n-butyl phthalate	153, 227
d_4-Di-n-pentyl phthalate	153, 241
d_4-Di-n-hexyl phthalate	153, 255
d_4-Benzyl butyl phthalate	153, 210
d_4-Dicyclohexyl phthalate	153, 253
d_4-Di-(2-ethylhexyl) phthalate	153, 283
d_4-Di-n-heptyl phthalate	153, 269
d_4-Di-n-octyl phthalate	153, 283
d_4-Di-n-nonyl phthalate	153, 297

GC apparatus should be optimised according to manufacturer's instruction.

NOTE: All glassware used in this method must be muffled in an oven at 500℃ for a minimum of 4 hours prior to use.

NOTE: PTFE septa must be used in all cases to minimise phthalate contamination.

4. PROCEDURE

4.1 Sample preparation

4.1.1 Sample logging

Assign each sample a unique number using the Fera LIMS system.

All samples should be homogenised prior to analysis.

4.1.2 Test sample preparation

Weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 ml glass vial. Record the accurate mass of the samples. Using a glass pipette add internal standard working solution (30 µl of a 100 µg/ml solution, 2.4.5). Stand the sample at room temperature for 15 minutes to allow the internal standards to infuse into the sample. Add extraction solution, acetonitrile: dichloromethane, 1:1 (15 ml, 2.4.6), cap and shake using an orbital shaker for 4 hours.

Centrifuge at 2000 rpm for 5 minutes and transfer the solvent layer to a clean 40 ml glass vial. Transfer the vials containing the solvent extracts to a freezer and store for 24 hours at –20℃.

Decant the organic solvent (not the fat) into a clean 40 ml glass vial and evaporate to dryness on a heating block at 60℃ (no nitrogen).

> NOTE: Evaporate just to dryness, do not leave the samples on the heating block as the more volatile phthalate diesters will be lost.

Reconstitute the dry residue in acetonitrile (1 ml, 2.2.1), cap and vortex mix for 30 seconds. Transfer the solution to a vial suitable for analysis by GC-MS.

Samples should be prepared in duplicate.

4.1.3 Spiked samples

Two spiking levels have been selected to allow analytical recovery to be determined irrespective of the concentration detected in the foodstuff.

Low spike - weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 ml glass vial. Record the accurate mass of the sample. Using a glass syringe add the mixed phthalates working solution (50 µl of 2.4.3) and internal standard working solution (30 µl of 2.4.5). Stand the sample at room temperature for 15 minutes to allow the standard to infuse into the sample. Proceed with the extraction as described in clause 4.1.2.

High spike - weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 ml glass vial. Record the accurate mass of the sample. Using a glass syringe add the mixed phthalates working solution (250 µl of 2.4.3) and internal standard working solution (30 µl of 2.4.5). Stand the sample at room temperature for 15 minutes to allow the standards to infuse into the sample. Proceed with the extraction as described in clause 4.1.2.

4.1.4 Blank food samples

For blank food samples weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 ml glass vial. Record the accurate mass of the sample. Do not add internal standard. Proceed with the extraction as described in clause 4.1.2.

4.1.5 Procedural blank samples

Prepare duplicate samples as described in clause 4.1.1 but in the absence of the foodstuff.

NOTE: It is essential that these procedural blank samples are prepared to check for phthalate contamination occurring as a consequence of the extraction and analysis.

4.2 Calibration standards

Prepare calibration standards adding the volumes of the mixed phthalates intermediate solution (2.4.2) and the internal standards working solution (2.4.5) and acetonitrile (2.3.1) described in the table below to a series of eight 10 ml volumetric flasks.

Standard	Volume (µl) of the mixed phthalates intermediate solution (2.4.2)	Volume (µl) of the internal standards working solution (2.4.5)
A	0	300
B	20	300
C	50	300
D	100	300
E	200	300
F	300	300
G	400	300
H	500	300

Make up to the mark with acetonitrile (2.3.1). Transfer a portion of each solution to a vial suitable for analysis by GC-MS.

Calculate the accurate concentration of each analyte in each of the calibration standard solutions.

5. PROCEDURE

5.1 GC-MS analysis

When starting measurements baseline stability and response linearity of the detector should be examined. The same operating conditions for the GC-MS system should be maintained throughout the analysis of all samples, spiked samples, blank samples and calibration standards set out in clause 4.

5.1.1 Sample treatment and execution of the determination

Place the vials with the samples prepared in 4.1.2 to 4.1.5 in the autosampler and analyse using the conditions given in clause 3.1.1.

Identify the analyte and internal standard peaks on the basis of their retention time and measure the respective peak areas of each analyte and internal standard peak area to obtain the peak area ratios.

The peak areas to be used for quantification are:

Dimethyl phthalate (m/z 163) versus d_4-dimethyl phthalate (m/z 167)

Diethyl phthalate (m/z 149) versus d_4-diethyl phthalate (m/z 153)

Diisopropyl phthalate (m/z 149) versus d_4-di-n-propyl phthalate (m/z 153)

Diallyl phthalate (m/z 149) versus d_4-di-n-propyl phthalate (m/z 153)

Diisobutyl phthalate (m/z 149) versus d_4-diisobutyl phthalate (m/z 153)

Di-n-butyl phthalate (m/z 149) versus d_4-di-n-butyl phthalate (m/z 153)

Di-n-pentyl phthalate (m/z 149) versus d_4-di-n-pentyl phthalate (m/z 153)

Di-n-hexyl phthalate (m/z 149) versus d_4-di-n-hexyl phthalate (m/z 153)

Benzyl butyl phthalate (m/z 206) versus d_4-benzyl butyl phthalate (m/z 210)

Dicyclohexyl phthalate (m/z 149) versus d_4-dicyclohexyl phthalate (m/z 153)

Di-(2-ethylhexyl) phthalate (m/z 279) versus d_4-di-(2-ethylhexyl) phthalate (m/z 283)

Di-n-heptyl phthalate (m/z 265) versus d_4-di-n-heptyl phthalate (m/z 269)

Di-n-octyl phthalate (m/z 149) versus d_4-di-n-octyl phthalate (m/z 153)

n-Octyl-n-decyl phthalate (m/z 149) versus d_4-di-n-nonyl phthalate (m/z 153)

Diisononyl phthalate (m/z 293) versus d_4-di-n-nonyl phthalate (m/z 153)

Diisodecyl phthalate (m/z 307) versus d_4-di-n-nonyl phthalate (m/z 153)

Di-n-decyl phthalate (m/z 149) versus d_4-di-n-nonyl phthalate (m/z 153)

5.1.2 Calibration

Inject each of the calibration standards as prepared in clause 4.2, onto the GC column.

Plot the peak area ratio values derived in 5.1.1 against the concentration of the analyte (in units of µg/ml) in the calibration standards (4.2). Do this for each analyte.

The calibration curves shall be rectilinear and the correlation coefficient shall be 0.99 or better.

5.2 Evaluation of data

5.2.1 Calculation of analyte levels in the foodstuff

Calculate the average of the peak area ratio values obtained from the test samples (5.1.1) and read the analyte concentrations in the extract from the calibration graphs produced in 5.1.2.

If the regression line equation is

$y = a * x + b$

$C_{extract} = (y-b)/a$

Where:

$C_{extract}$ is the concentration of the analyte in the extract.

The concentration in the foodstuff is then calculated as:

$C_{food} = (C_{extract} \times V)/M$

Where:

$C_{extract}$ is the concentration of the analyte in the extract.

V is total final volume of the extract.

M is the mass of the foodstuff extracted.

C_{food} is the concentration of the analyte in the foodstuff.

NOTE: Following this SOP the total volume of the extract is 1 ml.

5.2.2 Calculation of analyte levels in the spiked foods

Calculate the average of the peak area ratio values obtained from the spiked samples (5.1.1) and read the analyte concentrations from the calibration graphs produced in 5.1.2. Calculate the analyte concentrations in the spiked food as described in clause 5.2.1.

5.2.3 Correction for recovery

The average concentration of each anaylte determined in the food (5.2.1) is subtracted from the concentration in the spiked sample (5.2.2) and the recovery is calculated relative to the known concentration spiked. The concentration in the food (5.2.1) is then corrected for recovery.

5.3 Confirmation

The phthalate diesters are confirmed by applying the obligatory criteria:

(i) Calculate the ion ratios for the analytes using the following ions:

Phthalate diester	**Ratios**
Dimethyl phthalate	163/77 and 163/194
Diethyl phthalate	149/177 and 149/222
Diisopropyl phthalate	149/192 and 149/209
Diallyl phthalate	149/104 and 149/189
Diisobutyl phthalate	149/205 and 149/223
Di-n-butyl phthalate	149/205 and 149/223
Di-n-pentyl phthalate	149/219 and 149/237
Di-n-hexyl phthalate	149/233 and 149/251
Benzyl butyl phthalate	149/206

Dicyclohexyl phthalate	149/167 and 149/249
Di-(2-ethylhexyl) phthalate	279/167 and 279/149
Di-n-heptyl phthalate	265/167 and 265/149
Di-n-octyl phthalate	149/167 and 149/279
n-Octyl-n-decyl phthalate	149/279 and 149/307
Diisononyl phthalate	149/167 and 149/293
Diisodecyl phthalate	149/167 and 149/307
Di-n-decyl phthalate	149/167 and 149/307

The criteria is that the ratios obtained for the samples should agree to ± 20% of that obtained from the calibration standards analysed at the same time.

(ii) Calculate the analyte retention times relative to the appropriate internal standard (RRT). The criteria is that the RRT of the samples should agree to ± 2% of that obtained from the calibration analysed at the same time.

ANNEX 2.

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Standard operating procedure for determination of phthalate monoesters in foodstuffs

0. INTRODUCTION

Phthalates (diesters) are a group of toxic compounds produced in large volumes that are widely used as additives in plastics and consumer products. Some phthalates have been reported to be endocrine disruptors. They can migrate into foods from food packaging but have also been proposed to be present in food due to general contamination of the environment and food chain. In previous studies differences were observed in the total phthalate concentration (calculated by converting all phthalates to dimethyl phthalate) and the sum of the individual phthalate diesters measured in foods. It was proposed that the difference in these two values could be due to the presence of phthalic acid and phthalate monoesters in the food samples tested as these substances would also be converted to dimethyl phthalate and therefore included in the total phthalate measurement. To ascertain whether or not this proposal is correct a method of analysis has been developed for the commercially available phthalate monoesters. The analytical method described allows the determination of nine phthalate monoesters.

1. SCOPE

This document describes a method for the determination of nine phthalate monoesters in a range of foodstuffs. The method is appropriate for the quantitative determination in approximate analyte concentration range of 0.005 – 0.1 mg/kg.

2. REAGENTS

2.1 Analytes

2.1.1 Monoisopropyl phthalate (100 µg/mL)

2.1.1 Monoisobutyl phthalate (100 µg/mL)

2.1.2 Mono-n-butyl phthalate (100 µg/mL)

2.1.3 Monobenzyl phthalate (100 µg/mL)

2.1.4 Monocyclohexyl phthalate (100 µg/mL)

2.1.5 Mono-n-pentyl phthalate (100 µg/mL)

2.1.6 Mono-2-ethylhexyl phthalate (100 µg/mL)

2.1.7 Mono-n-octyl phthalate (100 µg/mL)

2.1.9 Monoisononyl phthalate (100 µg/mL)

2.2 Internal standards

2.2.1 (ring- 1,2-13C2, dicarboxyl- 13C2) Mono-2-ethylhexyl phthalate (100 µg/mL)

2.2.2 (ring- 1,2-13C2, dicarboxyl- 13C2) Mono-n-octyl phthalate (100 µg/mL)

2.2.3 (ring- 1,2-13C2, dicarboxyl- 13C2) Monoisononyl phthalate (100 µg/mL)

2.2.4 (ring- 1,2-13C2, dicarboxyl- 13C2) Monobenzyl phthalate (100 µg/mL)

2.2.5 (ring- 1,2-13C2, dicarboxyl- 13C2) Monocyclohexyl phthalate (100 µg/mL)

2.3 Chemicals

2.3.1 Acetonitrile, HPLC fluorescence grade

2.3.2 Dichloromethane, HPLC grade

2.3.3 Glacial acetic acid, 99-100%, Technical grade

2.4 Solutions

2.4.1 Mixed phthalate monoesters working solution, 7.5 µg/mL

Using a glass syringe transfer 750 µL of each of the unlabelled phthalate monoesters (2.1.1 to 2.1.9) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Store the solution at a temperature below 5℃.

2.4.2 Internal standards working solution, 7.5 µg/mL

Using a glass syringe transfer 750 µL of each of the labelled phthalate monoesters (2.2.1 to 2.2.5) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Store the solution at a temperature below 5℃.

2.4.3 Extraction solution, acetonitrile : dichloromethane, 1:1 (v:v)

Using a measuring cylinder add acetonitrile (500 mL, 2.3.1) and dichloromethane (500 mL, 2.3.2) to a 1 L Duran bottle. Cap and mix thoroughly.

3. APPARATUS

NOTE: An instrument or item of apparatus is listed only where it is special or made to a particular specification, the usual laboratory glassware and equipment being assumed to be available.

3.1 Liquid chromatograph, equipped with an automatic sampler and a mass spectrometric detector

Appropriate operating conditions have to be established for the specific equipment used for the determination.

3.1.1 Liquid chromatographic parameters

NOTE: The following column and parameters have been found to be suitable:

Column: SunFire™ C18 150 x 2.1 mm x 3.5 µm (Waters)

Liquid chromatograph: Waters Alliance 2695 system

Mobile phase A: Water + 0.1% acetic acid (v/v)

Mobile phase B: Acetonitrile + 0.1% acetic acid (v/v)

Run time: 20 minutes

Column temperature: 35℃

Flow rate: 0.3 mL/min

Injection volume 10 µL

Gradient:

Time (minutes)	% A	% B
0	55	45
2	55	45
9.5	0	100
12	0	100
13	55	45
20	55	45

Mass Spectrometer: Micromass Quattro Ultima Mass Spectrometer

Ionisation: Negative electrospray

Dwell time: 0.1 sec

Cone voltage: 35 V

Capillary: 2.8 kV

Multiple reaction monitoring (MRM)

Analyte	**Transitions**	**Collision energy (eV)**
Monoisopropyl phthalate	206.8 → 120.8 206.8 → 134.9	17 10
Total mono-n-butyl phthalate and monoisobutyl phthalate	220.9 → 133.9 220.9 → 177.0	13 10
Mono-n-butyl-phthalate ONLY	220.9 → 69.0	10

Monobenzyl phthalate	255.0 → 106.9 255.0 → 182.8	15 12
Monocyclohexyl phthalate	246.9 → 97.0 246.9 → 146.9	15 15
Mono-n-pentyl phthalate	234.9 → 191.1 234.9 → 85.0	10 15
Mono-2-ethylhexyl phthalate	277.1 → 133.9 277.1 → 127.0	15 15
Mono-n-octyl phthalate	277.1 → 127.0 277.1 → 233.0	18 15
Monoisononyl phthalate	291.0 → 140.9 291.0 → 247.0	18 15

Internal standard	**Transitions**	**Collision energy (eV)**
Labelled mono-n-butyl phthalate	225.0 → 179.8	10
Labelled monobenzyl phthalate	259.0 → 107.0	12
Labelled monocyclohexyl phthalate	250.9 → 97.1	20
Labelled mono-2-ethylhexyl phthalate	281.0 → 137.0	15
Labelled mono-n-octyl phthalate	281.1 → 127.0	17
Labelled monoisononyl phthalate	295.0 → 250.1	25

4. PROCEDURE

4.1 Sample preparation

4.1.1 Sample logging

Assign each sample a unique number using the Fera LIMS system.

All samples should be homogenised prior to analysis.

4.1.2 Test sample preparation

Weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the samples. Using a glass syringe add internal standard working solution (100 µL of a 7.5 µg/mL solution, 2.4.2). Stand the sample at room temperature for 15 minutes to allow the internal standards to infuse into the sample. Add extraction solution, acetonitrile: dichloromethane, 1:1 (15 mL, 2.4.3) and glacial acetic acid (1 mL, 2.3.3), cap and shake using an orbital shaker for 4 hours.

Filter the samples through Whatman N 113V 185 mm filter paper. Collect the filtrate in a clean 40 mL glass vial and store overnight at –20℃.

Centrifuge at 2000 rpm for 3 minutes and decant the organic solvent (not the fat) into a 40 mL glass vial and evaporate to dryness on a heating block at 75℃.

Reconstitute the dry residue in acetonitrile (1.5 mL, 2.3.1), cap and sonicate for 1 minute. Filter the sample through a 0.2 µm PTFE syring filter into a glass vial for analysis by LC-MS/MS.

Samples should be prepared in duplicate.

4.1.3 Spiked sample

Weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the sample. Using a glass syringe add the mixed phthalate monoesters working solution (100 µL of a 7.5 µg/mL solution, 2.4.1), and internal standard working solution (100 µL of a 7.5 µg/mL solution, 2.4.2). Stand the sample at room temperature for 15 minutes to allow the standard to infuse into the sample. Proceed with the extraction as described in clause 4.1.2.

4.1.4 Blank sample

For blank samples weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the sample. Do not add the internal standards. Proceed with the extraction as described in clause 4.1.2.

4.1.5 Procedural blank samples

Prepare duplicate samples as described in clause 4.1.1 but in the absence of the foodstuff.

4.2 Calibration standards

Prepare calibration standards adding the volumes of the mixed phthalate monoesters working solution (2.4.1) and the internal standards working solution (2.4.2) and acetonitrile (2.3.1) described in the table below to a series of seven glass vials suitable for LC-MS/MS analysis.

Concentration (µg/mL)	**Volume (µL) of the mixed phthalate monoesters working solution (2.4.1)**	**Volume (µL) of the internal standards working solution (2.4.2)**	**Volume (µL) of acetonitrile**
0	0	100	1400
0.05	10	100	1390
0.1	20	100	1380
0.2	40	100	1360
0.5	100	100	1300
0.75	150	100	1250
1.0	200	100	1200

5. PROCEDURE

5.1 LC-MS/MS analysis

When starting measurements baseline stability and response linearity of the detector should be examined. The same operating conditions for the LC-MS/MS system should be maintained throughout the analysis of all samples, spiked samples, blank samples and calibration standards set out in clause 4.

5.1.1 Sample treatment and execution of the determination

Place the vials with the samples prepared in 4.1.2 to 4.1.5 in the autosampler and analyse using the conditions given in clause 3.1.1.

Identify the analyte and internal standard peaks on the basis of their retention time and measure the respective peak areas of each analyte and internal standard peak area to obtain the peak area ratios.

The peak areas to be used for quantification are:

Monoisopropyl phthalate (206.8 → 120.8) versus labelled monobenzyl phthalate (259.0 → 107.0)

Total monoisobutyl phthalate and mono-n-butyl phthalate (206.8 → 120.8) versus labelled monobenzyl phthalate (259.0 → 107.0)

Mono-n-butyl phthalate only (206.8 → 69.0) versus labelled monobenzyl phthalate (259.0 → 107.0)

Monobenzyl phthalate (220.9 → 133.9) versus labelled monobenzyl phthalate (259.0 → 107.0)

Monocyclohexyl phthalate (246.9 → 97.0) versus labelled monocyclohexyl phthalate (250.9 → 97.1)

Mono-n-pentyl phthalate (234.9 → 191.1) versus labelled mono-2-ethylhexyl phthalate (281.0 → 137.0)

Mono-2-ethylhexyl phthalate (277.1 → 133.9) versus labelled mono-2-ethylhexyl phthalate (281.0 → 137.0)

Mono-n-octyl phthalate (277.1 → 127.0) versus labelled mono-n-octyl phthalate (281.1 → 127.0)

Monoisononyl phthalate (291.0 → 140.9) versus labelled monoisononyl phthalate (295.0 → 250.1)

5.1.2 Calibration

Inject each of the calibration standards as prepared in clause 4.2, onto the LC column.

Plot the peak area ratio values derived in 5.1.1 against the concentration of analyte in solution. Do this for each analyte.

The calibration curves shall be rectilinear and the correlation coefficient shall be 0.99 or better.

5.2 Evaluation of data

5.2.1 Calculation of analyte levels in the foodstuff

Calculate the average of the peak area ratio values obtained from the test samples (5.1.1) and read the analyte concentrations from the calibration graphs produced in 5.1.2.

If the regression line equation is:

$$y = a * x + b \qquad (1)$$

$$C = (y-b)/a \qquad (2)$$

Where:

C is the concentration of the analyte in solution.

The concentration in the foodstuff is then calculated as:

$C_{food} = (C * 1.5)/M$

Where:

C is the concentration of the analyte in solution.

M is the mass of food extracted

C_{food} is the concentration of the analyte in the foodstuff.

5.2.2 Calculation of analyte levels in the spiked foods

Calculate the average of the peak area ratio values obtained from the spiked samples (5.1.1) and read the analyte concentrations from the calibration graphs produced in 5.1.2. Calculate the analyte concentrations in the spiked food as described in clause 5.2.1.

5.2.3 Correction for recovery

The average concentration of each analyte determined in the food (5.2.1) is subtracted from the concentration in the spiked sample (5.2.2) and the recovery is calculated relative to the known concentration spiked. The concentration in the food (5.2.1) is then corrected for recovery.

5.2.4 Monoisobutyl phthalate and mono-n-butyl phthalate quantification

For the monoisobutyl phthalate and mono-n-butyl phthalate carry out the quantification as described in sections 5.2.1 to 5.2.3 for the total monoisobutyl phthalate and mono-n-butyl phthalate channel (206.8 → 120.8) and for the mono-n-butyl phthalate channel (206.8 → 69.0). To calculate the amount of monoisobutyl phthalate subtract the amount of mono-n-butyl phthalate from the total of monoisobutyl phthalate and mono-n-butyl phthalate.

5.3 Confirmation

The phthalate monoesters are confirmed by applying the obligatory criteria:

(i) Calculate the relative retention times (RRT) af the analytes to the internal standards. The criteria is that the RRT of the samples should agree to ± 2% of that obtained from the calibration standards analysed at the same time.

(ii) Calculate the ratio of the peak area of the quantification channel to the peak area of the confirmation channel were calculated using the transitions:

Analyte	**Quantification transition**	**Confirmation transition**
Monoisopropyl phthalate	206.8 → 120.8	206.8 → 134.9
Monoisobutyl phthalate	220.9 → 133.9	220.9 → 177.0
Mono-n-butyl phthalate	220.9 → 69.0	None
Monobenzyl phthalate	255.0 → 106.9	255.0 → 182.8
Monocyclohexyl phthalate	246.9 → 97.0	246.9 → 146.9
Mono-n-pentyl phthalate	234.9 → 191.1	234.9 → 85.0
Mono-2-ethylhexyl phthalate	277.1 → 133.9	277.1 → 127.0
Mono-n-octyl phthalate	277.1 → 127.0	277.1 → 233.0
Monoisononyl phthalate	291.0 → 140.9	291.0 → 247.0

The criteria is that the ratios obtained for the samples should agree to ± 25% of that obtained from the calibration standards.

ANNEX 3.

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Standard operating procedure for determination of phthalic acid in foodstuffs

0. INTRODUCTION

Phthalates (diesters) are a group of toxic compounds produced in large volumes that are widely used as additives in plastics and consumer products. Some phthalates have been reported to be endocrine disruptors. They can migrate into foods from food packaging but have also been proposed to be present in food due to general contamination of the environment and food chain. In previous studies differences were observed in the total phthalate concentration (calculated by converting all phthalates to dimethyl phthalate) and the sum of the individual phthalate diesters measured in foods. It was proposed that the difference in these two values could be due to the presence of phthalic acid and phthalate monoesters in the food samples tested as these substances would also be converted to dimethyl phthalate and therefore included in the total phthalate measurement. To ascertain whether or not this proposal is correct a method of analysis has been developed for phthalic acid. This report describes the performance characteristics of this analytical method.

1. SCOPE

This document describes a method for the determination of phthalic acid in a range of foodstuffs. The method is appropriate for quantitative determination in approximate analyte concentration range of 0.005 – 0.1 mg/kg.

2. REAGENTS

2.1 Analyte

2.1.1 Phthalic acid

2.2 Internal standard

2.2.1 Phthalic acid-ring-d_4

2.3 Chemicals

2.3.1 Acetonitrile, HPLC fluorescence grade

2.3.2 Dichloromethane, HPLC grade

2.3.3 Glacial acetic acid, 99-100%, Technical grade

2.4 Solutions

2.4.1 Phthalic acid stock solution (1 mg/mL)

Weigh to the nearest 0.1 mg approximately 10 mg of phthalic acid (2.1.1) in a 10 ml volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of phthalic acid per millilitre of solution.

Store the solution at a temperature below 5℃.

2.4.2 Phthalic acid working solution (7.5 µg/mL)

Using a glass syringe transfer 75 µL of the unlabeled phthalic acid solution (2.4.1) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of phthalic acid per millilitre of solution.

Store the solution at a temperature below 5℃.

2.4.3 Internal standard stock solution (1 mg/mL)

Weigh to the nearest 0.1 mg approximately 10 mg of labelled phthalic acid (2.2.1) in a 10 ml volumetric flask and make up to the mark with acetonitrile (2.3.1).

Store the solution at a temperature below 5℃.

2.4.4 Internal standard working solution (7.5 µg/mL)

Using a glass syringe transfer 75 µL of the labeled phthalic acid stock solution (2.4.3) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Store the solution at a temperature below 5℃.

2.4.5 Extraction solution, acetonitrile : dichloromethane, 1:1 (v:v)

Using a measuring cylinder add acetonitrile (500 mL, 2.3.1) and dichloromethane (500 mL, 2.3.2) to a 1 L Duran bottle. Cap and mix thoroughly.

3. APPARATUS

NOTE: An instrument or item of apparatus is listed only where it is special or made to a particular specification, the usual laboratory glassware and equipment being assumed to be available.

3.1 Liquid chromatograph, equipped with an automatic sampler and a mass spectrometric detector

Appropriate operating conditions have to be established for the specific equipment used for the determination.

3.1.1 Liquid chromatographic parameters

NOTE: The following column and parameters have been found to be suitable:

Liquid chromatography-mass spectrometry (LC-MS/MS)

Column: SeQuant ZIC®-HILIC (100 x 2.1 mm, 3.5 µm)

Liquid chromatograph: Waters Alliance 2695 system

Mobile phase A: 10 mM ammonium acetate at pH 6

Mobile phase B: Acetonitrile

Mobile phase composition: Isocratic, 5% A, 95% B

Run time: 10 minutes

Column temperature: 35℃

Flow rate: 0.2 mL/min

Injection volume 5 µL

Mass Spectrometer: Micromass Quattro Ultima Mass Spectrometer

Ionisation: Negative electrospray

Dwell time: 0.1 sec

Cone voltage: 35 V

Capillary: 3.0 kV

Multiple reaction monitoring (MRM)

Analyte	**Transitions**	**Collision energy (eV)**
Phthalic acid (quantification) Phthalic acid (confirmation)	164.75 → 120.81 164.75 → 76.95	10 15
d_4-Phthalic acid (Internal standard)	169.28 → 124.81	15

4. PROCEDURE

4.1 Sample preparation

4.1.1 Sample logging

Assign each sample a unique number using the Fera LIMS system.

All samples should be homogenised prior to analysis.

4.1.2 Test sample preparation

Weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the samples. Using a glass syringe add internal standard working solution (100 µL of a 7.5 µg/mL solution, 2.4.4). Stand the sample at room temperature for 15 minutes to allow the internal standard to infuse into the sample. Add extraction solution, acetonitrile: dichloromethane, 1:1 (15 mL, 2.4.5) and glacial acetic acid (1 mL, 2.3.3), cap and shake using an orbital shaker for 4 hours.

Filter the samples through Whatman N 113V 185 mm filter paper. Collect the filtrate in a clean 40 mL glass vial and store overnight at –20℃.

Centrifuge at 2000 rpm for 3 minutes and decant the organic solvent (not the fat) into a 40 mL glass vial and evaporate to dryness on a heating block at 75℃.

Reconstitute the dry residue in acetonitrile (1.5 mL, 2.3.1), cap and sonicate for 1 minute. Filter the sample through a 0.2 µm PTFE syring filter into a glass vial for analysis by LC-MS/MS.

Samples should be prepared in duplicate.

4.1.3 Spiked sample

Weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the sample. Using a glass syringe add the phthalic acid working solution, 100 µL of a 7.5 µg/mL solution (2.4.2), and internal standard working solution, 100 µL of a 7.5 µg/mL solution (2.4.4). Stand the sample at room temperature for 15 minutes to allow the standard and internal standard to infuse into the sample. Proceed with the extraction as described in clause 4.1.2.

4.1.4 Blank food sample

For the blank food sample weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the sample. Do not add internal standard. Proceed with the extraction as described in clause 4.1.2.

4.1.5 Procedural blank samples

Prepare duplicate samples as described in clause 4.1.1 but in the absence of the foodstuff.

4.2 Calibration standards

Prepare calibration standards adding the volumes of the phthalic acid working solution (2.4.2) and the internal standard working solution (2.4.4) and acetonitrile (2.3.1) described in the table below to a series of seven glass vials suitable for LC-MS/MS analysis.

Concentration (µg/mL)	Volume (µL) of the phthalic acid working solution (2.4.2)	Volume (µL) of the internal standard working solution (2.4.4)	Volume (µL) of acetonitrile
0	0	100	1400
0.05	10	100	1390
0.1	20	100	1380
0.2	40	100	1360
0.5	100	100	1300
0.75	150	100	1250
1.0	200	100	1200

5. PROCEDURE

5.1 LC-MS/MS analysis

When starting measurements baseline stability and response linearity of the detector should be examined. The same operating conditions for the LC-MS/MS system should be maintained throughout the analysis of all samples, spiked samples, blank samples and calibration standards set out in section 4.

5.1.1 Sample treatment and execution of the determination

Place the vials with the samples prepared in 4.1.2 to 4.1.5 in the autosampler and analyse using the conditions given in clause 3.1.1.

Identify the analyte and internal standard peaks on the basis of their retention time and measure the respective peak areas to obtain the peak area ratios.

The peak areas to be used for quantification are:

Phthalic acid (164.75 → 120.81) versus labelled phthalic acid (169.28 → 124.81)

5.1.2 Calibration

Inject each of the calibration standards as prepared in clause 4.2, onto the LC column.

Plot the peak area ratio values derived in 5.1.1 against the concentration of the analyte in solution.

The calibration curves shall be rectilinear and the correlation coefficient shall be 0.99 or better.

5.2 Evaluation of data

5.2.1 Calculation of analyte levels in the foodstuff

Calculate the average of the peak area ratio values obtained from the test samples (5.1.1) and read the analyte concentrations from the calibration graphs produced in 5.1.2.

If the regression line equation is:

$$y = a * x + b \qquad (1)$$

$$C = (y-b)/a \qquad (2)$$

Where:

C is the concentration of the analyte in solution.

The concentration in the foodstuff is then calculated as:

$$C_{food} = (C * 1.5)/M$$

Where:

C is the concentration of the analyte in solution.

M is the mass of food extracted

C_{food} is the concentration of the analyte in the foodstuff.

5.2.2 Calculation of the phthalic acid concentration in the spiked foods

Calculate the average of the peak area ratio values obtained from the spiked samples (5.1.1) and read the analyte concentrations from the calibration graphs produced in 5.1.2. Calculate the analyte concentrations in the spiked food as described in clause 5.2.1.

5.2.3 Correction for recovery

The average concentration of phthalic acid determined in the food (5.2.1) is subtracted from the concentration in the spiked sample (5.2.2) and the recovery is calculated relative to the known concentration spiked. The concentration in the food (5.2.1) is then corrected for recovery.

5.3 Confirmation

The phthalic acid is confirmed by applying the obligatory criteria:

(i) Calculate the phthalic acid retention time relative to the labelled internal standard (RRT). The criteria is that the RRT of the samples should agree to ± 2% of that obtained from the calibration analysed at the same time.

(ii) Calculate the ratio of the peak area of the quantification channel to the peak area of the confirmation channel using the transitions:

Analyte	**Quantification channel**	**Confirmation channel**
Phthalic acid	164.75 → 120.81	169.28 → 124.81

The criteria is that the ratios obtained for the samples should agree to ± 25% of that obtained from the calibration standards analysed at the same time.

ANNEX 4.

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Standard operating procedure for determination of total phthalates in foodstuffs

0. INTRODUCTION

Phthalates (diesters) are a group of toxic compounds produced in large volumes that are widely used as additives in plastics and consumer products. Some phthalates have been reported to be endocrine disruptors. They can migrate into foods from food packaging but have also been proposed to be present in food due to general contamination of the environment and food chain. In previous studies differences were observed in the total phthalate concentration (calculated by converting all phthalates to dimethyl phthalate) and the sum of the individual phthalate diesters measured in foods. It was proposed that the difference in these two values could be due to the presence of phthalic acid and phthalate monoesters in the food samples tested as these substances would also be converted to dimethyl phthalate and therefore included in the total phthalate measurement as well as the presence of any phthalate diesters that were not included in the analysis. The analytical method described allows the determination of the total phthalate molecules present i.e. diesters, monoesters and phthalic acid. These are extracted from the foodstuff, hydrolysed and methylated to form dimethyl phthalate prior to measurement by GC-MS.

1. SCOPE

This document describes a method for the determination of total phthalates in a range of foodstuffs. The method is appropriate for the quantitative determination in approximate analyte concentration range of 100 – 1000 µg/kg.

2. REAGENTS

2.1 Analytes

2.1.1 Dimethyl phthalate, 99%

2.1.2 Diethyl phthalate, 99%

2.1.3 Diisobutyl phthalate, 99%

2.1.4 Di-n-decyl phthalate, 99%

2.2 Internal standard

2.2.1 d_4-Dimethyl phthalate, 99%

2.3 Chemicals

2.3.1 Acetonitrile, HPLC fluorescence grade

2.3.2 Dichloromethane, HPLC grade

2.3.3 Glacial acetic acid, 99-100%, Technical grade

2.3.4 Potassium hydroxide, Analytical reagent grade

2.3.5 Methanol, HPLC fluorescence grade

2.3.6 Calcium acetate monohydrate, ACS reagent grade, >99%

2.3.7 Water, HPLC fluorescence grade

2.3.8 Boron trifluoride methanol solution, 14%

2.3.9 Hexane, HPLC fluorescence grade

2.4 Solutions

2.4.1 Dimethyl phthalate stock solution, 1 mg/mL

Weigh to the nearest 0.1 mg approximately 10 mg of dimethyl phthalate (2.1.1) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of dimethyl phthalate per millilitre of solution. Store the solution at a temperature below 5℃.

2.4.2 Dimethyl phthalate working solution, 100 µg/mL

Using a glass syringe transfer 1 mL of dimethyl phthalate stock solution (1 mg/mL, 2.4.1) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of dimethyl phthalate per millilitre of solution. Store the solution at a temperature below 5℃.

2.4.3 d_4-Dimethyl phthalate stock solution, 1 mg/mL

Weigh to the nearest 0.1 mg approximately 10 mg of the d_4-dimethyl phthalate (2.2.1) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of d_4-dimethyl phthalate per millilitre of solution. Store the solution at a temperature below 5℃.

2.4.4 d_4-Dimethyl phthalate working solution, 100 µg/mL

Using a glass syringe transfer 1 mL of d_4-dimethyl phthalate stock solution (1 mg/mL, 2.4.3) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of dimethyl phthalate per millilitre of solution. Store the solution at a temperature below 5℃.

2.4.5 Diethyl phthalate stock solution, 1 mg/mL

Weigh to the nearest 0.1 mg approximately 10 mg of diethyl phthalate (2.1.2) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of diethyl phthalate per millilitre of solution. Store the solution at a temperature below 5°C.

2.4.6 Diisobutyl phthalate stock solution, 1 mg/mL

Weigh to the nearest 0.1 mg approximately 10 mg of diisobutyl phthalate (2.1.3) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of diisobutyl phthalate per millilitre of solution. Store the solution at a temperature below 5°C.

2.4.7 Di-n-decyl phthalate stock solution, 1 mg/mL

Weigh to the nearest 0.1 mg approximately 10 mg of di-n-decyl phthalate (2.1.4) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Calculate the actual concentration in milligrams of di-n-decyl phthalate per millilitre of solution. Store the solution at a temperature below 5°C.

2.4.8 Diethyl phthalate, diisobutyl phthalate and di-n-decyl phthalate spiking solution, 100 µg/mL

Using a glass syringe transfer 1 mL of diethyl phthalate stock solution (1 mg/mL, 2.4.5), 1 mL of diisobutyl phthalate stock solution (1 mg/mL, 2.4.6) and 1 mL of di-n-decyl phthalate stock solution (1 mg/mL, 2.4.7) into a 10 mL volumetric flask and make up to the mark with acetonitrile (2.3.1).

Store the solution at a temperature below 5°C.

2.4.9 Extraction solution, acetonitrile:dichloromethane, 1:1 (v:v)

Using a measuring cylinder add acetonitrile (500 mL, 2.3.1) and dichloromethane (500 mL, 2.3.2) to a 1 L Duran bottle. Cap and mix thoroughly.

2.4.10 Methanolic potassium hydroxide solution, 1 M

Weigh to the nearest 0.1 g approximately 56.1 g of potassium hydroxide (2.3.4) into a 500 mL beaker, add approximately 400 mL of methanol (2.3.5) and dissolve by shaking. Once dissolved transfer to a 1 L volumetric flask, make up to the mark with methanol (2.3.5) and shake well.

2.4.11 Methanolic potassium hydroxide solution, 0.1 M

Using a measuring cylinder transfer 100 mL of 1 M methanolic potassium hydroxide solution (2.4.10) to a 1 L volumetric flask. Make up to the mark with methanol (2.3.5) and shake well.

2.4.12 Calcium acetate solution, 5%

Weigh to the nearest 0.1 g approximately 50 g of calcium acetate monohydrate (2.3.6) into a 1 L volumetric flask, add approximately 500 mL of water (2.3.7) and dissolve by shaking. Make up to the mark with water (2.3.7) and shake well.

3. APPARATUS

NOTE: An instrument or item of apparatus is listed only where it is special or made to a particular specification, the usual laboratory glassware and equipment being assumed to be available.

3.1 Gas chromatograph, equipped with an automatic sampler and a mass spectrometric detector

Appropriate operating conditions have to be established for the specific equipment used for the determination.

3.1.1 Gas chromatographic parameters

GC apparatus should be optimised according to manufacturer's instruction.

NOTE: The following column and parameters have been found to be suitable:

Column:	Zebron ZB-5ms (5% phenyl 95% dimethylpolysiloxane) 30 m x 0.25 mm id x 0.25 µm film thickness
Column oven	Isothermal 3 minutes at 80℃, then at 10℃/minute to 320℃ and held for 5 minutes
Split mode	Splitless
Inlet temperature	280°C
Injection volume	1.0 µL (Merlin Micro Seal and Syringe)
Carrier gas	Helium
Flow rate	1.0 mL/minute
Transfer line temp.	280°C
MS detection mode	Selected ion monitoring (SIM) EI mode
SIM ions recorded	Dimethyl phthalate: 77, 163, 194
	d_4-Dimethyl phthalate: 167, 198

4. PROCEDURE

NOTE: All glassware used in this method must be muffled in an oven at 500℃ for a minimum of 4 hours prior to use.

NOTE: PTFE septa must be used in all cases to minimise phthalate contamination.

4.1 Sample preparation

4.1.1 Sample logging

Assign each sample a unique number using the Fera LIMS system. All samples should be homogenised prior to analysis.

4.1.2 Test sample preparation

Weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the samples. Using a glass syringe add internal standard working solution (60 µL of a 100 µg/mL solution, 2.4.4). Stand the sample at room temperature for 15 minutes to allow the internal standard to infuse into the sample. Add extraction solution, acetonitrile: dichloromethane, 1:1 (15 mL, 2.4.9) and glacial acetic acid (1 mL, 2.3.3), cap and shake using an orbital shaker for 4 hours.

Decant the liquid into a clean 40 mL glass vial and store overnight at –20℃.

Centrifuge at 2000 rpm for 3 minutes and decant the organic solvent (not the fat) into a 40 mL glass vial and evaporate to dryness on a heating block at 45℃ (no nitrogen).

> NOTE: Evaporate just to dryness at a temperature not greater than 45℃ and do not leave the samples on the heating block over night, as the more volatile phthalates will be lost.

Reconstitute the dry residue in methanolic potassium hydroxide solution (0.1 M, 4 mL, 2.4.11), cap and vortex mix for 1 minute. Measure the pH using universal pH paper. If the pH is less than 10 add methanolic potassium hydroxide solution (1 M, 1 mL increments, 2.4.10) until the pH reaches 10. Cap and vortex mix for 1 minute. Place in an oven at 90^{o}C for 1 hour. Remove the vials, allow them to cool to ambient temperature and add boron trifluoride methanol solution (2 mL, 2.3.8). Cap, vortex mix for 1 minute and place in an oven at 90^{o}C for 1 hour.

Remove the vials, allow them to cool to ambient temperature and add aqueous calcium acetate solution (5%, 10 mL, 2.4.12). Cap and vortex mix for 30 seconds. Add hexane (4 mL, 2.3.9), cap, vortex mix for 30 seconds, centrifuge at 2000 rpm for 3 minutes and transfer the top hexane layer to a clean 12 mL glass vial. Add acetonitrile (1 mL, 2.3.1), cap, vortex mix for 30 seconds and transfer the bottom acetonitrile layer to a glass vial suitable for GC-MS analysis.

Samples should be prepared in duplicate.

4.1.3 Spiked sample

Weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the sample. Using a glass syringe add diethyl phthalate, diisobutyl phthalate and di-n-decyl phthalate mixed spiking solution (30 µL of a 100 µg/mL mixed solution, 2.4.8), and internal standard working solution (60 µL of a 100 µg/mL solution, 2.4.4). Stand the sample at room temperature for 15 minutes to allow the standard to infuse into the sample. Proceed with the extraction as described in clause 4.1.2.

4.1.4 Blank sample

For blank samples weigh to the nearest 0.01 g approximately 15 g of homogenised foodstuff into a 40 mL glass vial. Record the accurate mass of the sample. Do not add the internal standard. Proceed with the extraction as described in clause 4.1.2.

4.1.5 Procedural blank samples

Prepare duplicate samples as described in clause 4.1.1 but in the absence of the foodstuff.

NOTE: It is essential that these procedural blank samples are prepared to check for phthalate contamination occurring as a consequence of the extraction and analysis.

4.2 Calibration standards

Prepare calibration standards adding the volumes of the dimethyl phthalate working solution (2.4.2), internal standard working solution (2.4.4) and acetonitrile (2.3.1) described in the table below to a series of glass vials suitable for GC-MS analysis.

Concen-tration (µg/mL)	Equivalent concentration in the food (µg/kg)	Volume (µL) of the dimethyl phthalate working solution (2.4.2)	Volume (µL) of the d_4-dimethyl phthalate working solution (2.4.4)	Volume (µL) of acetonitrile (2.3.1)
0	0	0	60	940
1.5	100	15	60	925
3.0	200	30	60	910
6.0	400	60	60	880
9.0	600	90	60	850
12	800	120	60	820
15	1000	150	60	790

5. PROCEDURE

5.1 GC-MS analysis

When starting measurements baseline stability and response linearity of the detector should be examined. The same operating conditions for the GC-MS system should be maintained throughout the analysis of all samples, spiked samples, blank samples and calibration standards set out in clause 4.

5.1.1 Sample treatment and execution of the determination

Place the vials with the samples prepared in 4.1.2 to 4.1.5 in the autosampler and analyse using the conditions given in clause 3.1.1.

Identify the analyte and internal standard peaks on the basis of their retention time and measure the respective peak areas of the analyte and internal standard peak area to obtain the peak area ratios.

The peak areas to be used for quantification are m/z 163 (dimethyl phthalate) versus m/z 167 (d_4-dimethyl phthalate).

5.1.2 Calibration

Inject each of the calibration standards as prepared in clause 4.2, onto the GC column.

Plot the peak area ratio values derived in 5.1.1 against the concentration of the analyte (in units of µg/kg) in the calibration standards (4.2).

The calibration curves shall be rectilinear and the correlation coefficient shall be 0.99 or better.

5.2 Evaluation of data

5.2.1 Calculation of analyte levels in the foodstuff

Calculate the average of the peak area ratio values obtained from the test samples (5.1.1) and read the analyte concentrations in the extract from the calibration graphs produced in 5.1.2.

If the regression line equation is

$$y = a * x + b \qquad (1)$$

$$x = (y-b)/a \qquad (2)$$

Where:

x is the concentration of the analyte in the extract,

a is the gradient of the calibration graph,

b is the y-intercept of the calibration graph

y is the peak area ratio.

5.2.2 Calculation of analyte levels in the spiked foods

Calculate the average of the peak area ratio values obtained from the spiked samples (5.1.1) and read the analyte concentrations from the calibration graphs produced in 5.1.2. Calculate the analyte concentrations in the spiked food as described in clause 5.2.1.

5.2.3 Correction for recovery

The average concentration of each analyte determined in the food (5.2.1) is subtracted from the concentration in the spiked sample (5.2.2) and the recovery is calculated relative to the known concentration spiked. The concentration of diethyl phthalate, diisobutyl phthalate and di-n-decyl phthalate should be converted into the theoretical concentration of dimethyl phthalate formed assuming complete hydrolysis. The concentration in the food (5.2.1) is then corrected for recovery.

5.3 Confirmation

The dimethyl phthalate is confirmed by applying the obligatory criteria:

(ii) Calculate the ion ratios for dimethyl phthalate using the 163/77 and 163/194 ions. The criteria is that the ratios obtained for the samples should agree to ± 20% of that obtained from the calibration standards analysed at the same time.

(ii) Calculate the relative retention time (RTT) as the analyte retention times divided by the internal standard retention time. The criteria is that the RRT of the samples should agree to ± 2% of those obtained from the calibration analysed at the same time.