

## **VETERINARY RESIDUES COMMITTEE ANNOUNCEMENT ON THE RESULTS OF THE BRAND NAMING SURVEY OF NITROFURANS IN WARM WATER PRAWNS**

A brand naming survey carried out by the Veterinary Medicines Directorate (VMD) on behalf of the Veterinary Residues Committee (VRC) found evidence of the use of nitrofurans in three samples of imported warm water prawns.

The Committee recommended that warm water prawns should be tested for these veterinary medicines because they wished to continue with their message to producers that the use of unauthorised substances in farming should not be tolerated. Previous brand naming surveys were based on sample collection at Border Inspection Posts and retail outlets. The committee recommended that this survey focused on Border Inspection Posts and wholesalers because they wished to understand whether a problem exists with wholesale products. Nitrofurans are not authorised for use in the treatment of animals for food production in the EU and may not be used in the production of food of animal origin which is exported to the EU. Nitrofurans have been linked with a possible increased risk of cancer in humans through long-term consumption.

A total of 102 samples of imported warm water prawns were analysed in the survey carried out between October and December 2006. Three samples contained residues of nitrofurans metabolites. Residues of semicarbazide (SEM), a metabolite of nitrofurazone, and 3-amino-2-oxazolidinone (AOZ), a metabolite of furazolidone, were confirmed in one sample. A second sample contained a confirmed residue of SEM. Both of these samples were from the same batch of imported prawns but were purchased in different months. A third sample contained a confirmed residue of AOZ. Following the confirmation of these residues the Food Standards Agency (FSA) was informed of the results and action was taken to ensure that the affected stock was removed from the food chain to protect public health.

The VRC is concerned about the non-compliant findings of the survey, which show that substances banned from use in the EU continue to be used in the third country production of food of animal origin which is exported to the EU. These active substances were chosen for the survey because of their unauthorised presence in imported warm water prawn samples analysed as part of the non-statutory programme in previous years.

At the Committee's request, Defra wrote to the European Commission, copied to the European Food Safety Authority, about the continuing presence of these residues in imported crustaceans earlier this year. The Commission has replied to Defra's Chief Veterinary Officer setting out the action that it is taking to deal with this issue.

## **Results of the brand naming survey for nitrofurans in warm water prawns**

### **Summary**

A survey of nitrofurans in imported warm water prawns was carried out by the VMD between October and December 2006. The purpose of the survey was to assess the incidence of residues of these active substances in warm water prawns imported through Border Inspection Posts (BIPs) and available from wholesale outlets. The use of nitrofurans in the treatment of food producing animals is prohibited in the EU.

A total of 102 samples of warm water prawns were analysed for residues of nitrofurans. Samples for the survey were purchased from a range of wholesalers by the market research organisation Mintel International Group Ltd, by a market inspector at Billingsgate Market and also collected by Port Health Officers from consignments entering the UK. All the samples were analysed by the Central Science Laboratory using a validated method of analysis.

Nitrofurans residues were confirmed in three samples of raw prawns. Residues of SEM were found in two samples, one of which also contained residues of AOZ. These two samples were found to be from the same batch of prawns but had been purchased in different months. Residues of AOZ only were found in a third sample.

SEM residues in food may arise from sources other than nitrofurans use<sup>1</sup>. The Community Reference Laboratory (CRL) in France recommends that for raw products of animal origin, samples should be analysed for bound residues of SEM and therefore for this survey any free SEM was removed by washing during the confirmatory test. The CRL's advice states that where residues of SEM are confirmed in raw animal tissue these are indicative of the use of nitrofurazone. Additionally in this survey where shell-on samples screened non-compliant, the shell was removed prior to confirmatory analysis.

The Limit of Quantification for each of the nitrofurans metabolites covered by the survey is 1 µg/kg. This limit is in line with the EU Minimum Required Performance Limit (MRPL) for these substances.

In each case where non-compliant results were confirmed, the FSA was informed, the Local Authority was contacted and produce from the same batch as the non-compliant sample was withdrawn and destroyed. In the case of the sample found to contain only AOZ, the product was no longer in stock at the wholesalers but all customers were informed and any remaining prawns from this consignment were removed from the food chain.

Annex 1 gives details of all the samples collected as part of the survey. Annex 2 gives details of sample preparation and the method of analysis.

<sup>1</sup> [http://www.efsa.europa.eu/en/press\\_room/press\\_release/2005/1006.html](http://www.efsa.europa.eu/en/press_room/press_release/2005/1006.html)

The results show that the incidence of residues of these substances in warm water prawns continues to be a problem. Table 1 below sets out surveillance data from the non-statutory programme for the last four years which shows a high level of non-compliant samples.

## Background

Testing of warm water prawns for veterinary residues has been included in the non-statutory surveillance programme for over ten years. As a result of the detection of residues of banned antibiotics in prawns and shrimps imported in to the EU in 2001, the Commission instigated an increased level of residue checks on consignments entering the EU.

A Food and Veterinary Office mission to China in 2001, revealed that substances such as chloramphenicol and nitrofurans, that are prohibited for use in food producing animals in the EU, were being used in some third countries. Testing of aquaculture for these banned antibiotics under the non-statutory and statutory surveillance programmes was encouraged by the committee and introduced in 2002 for exporting areas such as South East Asia and Asia.. Residues of the metabolite of the nitrofuran furazolidone were first detected in warm water prawns under the non-statutory surveillance programme in 2002.

Following further intelligence on the possible use of prohibited substances, testing for four nitrofuran metabolites was introduced in 2003. The four metabolites, with the parent compound given in brackets, were AOZ (furazolidone), AMOZ (furaltadone), SEM (nitrofurazone) and AHD (nitrofurantoin).

**Table 1 Results of non-statutory surveillance of imported warm water prawns from 2003 to 2006**

Year	Substance tested for	Number of samples tested	Number of samples non-compliant
2003	Nitrofurans (AOZ, AMOZ, AHD, SEM)	307	29 (7 AOZ, 22 SEM*)
2004	Nitrofurans (AOZ, AMOZ, AHD, SEM)	342	32 (6 AOZ, 26 SEM)
2005	Nitrofurans (AOZ, AMOZ, AHD, SEM)	301	19 (5 AOZ, 14 SEM)
2006	Nitrofurans (AOZ, AMOZ, AHD, SEM)	248	19 (3 AOZ, 16 SEM)

\*Prior to 2004 total SEM residues (free and bound) were analysed.

## Legislation

EC legislation requires that all active substances used in veterinary medicines for food producing animals must be assessed so that a Maximum Residue Limit can be set.

Nitrofurans are listed under Annex IV of Council Regulation 2377/90 and their use in food producing animals is prohibited in the EU. Countries that wish to export food of animal origin to the EU must respect the requirements of this legislation. Commission Decision 2002/657 introduced the concept of Minimum Required Performance Limits (MRPLs). This is the concentration level at which regulatory laboratories should at least be able to detect and confirm the presence of particular substances. In March 2003 the Commission set MRPLs of 1 µg/kg for nitrofurans metabolites in aquaculture products (Commission Decision 2003/181/EC).

## Sampling

In this survey, 102 samples of imported warm water prawns were analysed. The countries of origin of the samples were as follows:

Non-EU	Number of samples	More than 1 country of origin	Number of samples
Bangladesh	11	Thailand/Indonesia	1
China	5		
Ecuador	3		
Honduras	2		
India	30		
Indian Ocean	1		
Indonesia	11		
Malaysia	6		
Nicaragua	1		
Not provided	1		
Pacific Ocean	1		
Panama	1		
Philippines	2		
SE Asia	1		
Sri Lanka	1		
Surinam	2		
Thailand	9		
Vietnam	13		

Samples were collected by Port Health Officers at BIPs and also purchased by Mintel International Group Ltd and a market inspector at Billingsgate Market between October and December 2006. Samples were collected at five BIPs and ten wholesalers. The sample size was a minimum of 250g of warm water prawns. Details of all samples collected for the survey are given in Annex 1.

## Results

Full results of the survey are detailed in Annex 1. Details of the analytical methods are set out in Annex 2.

Fourteen samples tested non-compliant using an initial screening test. Of these three samples contained confirmed residues of nitrofurans in excess of the MRPL. The prawns in two of these samples had their shells on. The prawns in the third sample had had their shells removed.

A sample of raw black tiger prawns imported from India and collected at a UK Border Inspection Post was found to contain residues of AOZ at a concentration of 14 µg/kg.

One sample of prawns imported from Thailand, via Italy, and purchased from Billingsgate Market contained residues of SEM at a concentration of 1.6 µg/kg.

A second sample of prawns imported from Thailand, via Italy, and purchased from Billingsgate Market contained residues of SEM at a concentration of 1.6 µg/kg and residues of AOZ at a concentration of 1.1 µg/kg.

In accordance with the FSA survey guidelines, the importers or wholesalers of the contaminated product were asked if they wished to comment on the confirmed results. No comments were received.

### **Risk Assessment**

Nitrofurans are listed in Annex IV of Council Regulation 2377/90 and therefore are not authorised for use in the EU. Nitrofurans are considered to be genotoxic carcinogens and, as such, no safe level can be set for them in food. An MRPL of 1 µg/kg has been set for nitrofurans by Commission Decision 2003/181/EC amending 2002/657/EC. (MRPLs are agreed EU laboratory standards to detect a substance or its residues and are not safety limits and therefore food should not contain any traces of nitrofurans or their metabolites).

### **Action taken on residue findings**

As part of the follow-up action for the three non-compliant samples, the VMD wrote to the importer/wholesaler of the non-compliant samples requesting import documentation. In the case of the two samples of prawns imported from Thailand, which were from the same lot but purchased in different months, no documentation was available, as the product had entered the UK from Italy. The Local Authority was contacted by the FSA and any remaining stock held by the wholesaler was removed from sale or recalled, and subsequently destroyed.

In the case of the third sample imported from India, import documentation was provided by Port Health Officers and passed to the FSA who contacted the Local Authority where the consignment was being held. The Local Authority confirmed that the product was no longer in stock, but that all customers of the wholesaler had been informed and the remaining prawns from this consignment had been traced and removed from the food chain.

Since the testing results showed nitrofurans at or above the MRPL, the FSA also raised a Rapid Alert (RASFF) for the attention of the Commission and Member States, for all three samples.

Defra's Chief Veterinary Officer wrote to the European Commission, copied to the European Food Safety Authority, about the continuing presence of these residues in imported crustaceans earlier this year. The Commission has replied setting out the steps it is taking to deal with this problem and emphasising the need for Member States

to take steps to ensure food from contaminated consignments does not enter the food chain.

### **Conclusions**

The purpose of the survey was to assess the incidence of residues of nitrofurans metabolites in warm water prawns imported through Border Inspection Posts and available from wholesale outlets. The results of this survey found evidence of the use of nitrofurans in food production in some samples of imported warm water prawns. The continuing detection of these illegal substances is a cause for concern, particularly as they have been found for a number of years in this imported food product.

**ANNEX 1**

**PRAWN SURVEY 2006**

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1561	Raw Headless	Royal Star	India	On	17/10/2006	Jun-08	Fish Bargain Warehouse, 32 Acre Lane, Brixton SW2 5SG	Nortrade Foods, Kent TN18 5HB	Negative
06N1562	Ice glazed Prawns	Seatek	China	Off	17/10/2006	Jan-08	"	Seatek, London	Negative
06N1563	King Prawns	Thaistar	Thailand	Off	17/10/2006	17/09/2007	"	Nortrade Foods, Kent TN18 5HB	Negative
06N1564	Tropical Shrimp	Adrien	Sri Lanka	On	17/10/2006	Jan-08	"	Nantes Atlantiqua, France	Negative
06N1565	Black Tiger Prawn	Royal Gold	India	On	17/10/2006	May-08	"	Falcon Marine Exports Ltd, A/22 Cuttack Road, Falcon House 1st Floor, Orissa Bhubaneswar, India	Negative
06N1566	King Prawns	Ocean Pearl	Pacific Ocean	Off	17/10/2006	Sep-07	Booker Cash and Carry, Excelsior Road Industrial Estate, Cardiff CF14 3XL	Sea Products International Ltd	Negative
06N1567	Warm Water Prawns	Chef's Larder	Not provided	Off	17/10/2006	Jan-08	"	Chef's Larder	Negative
06N1568	Tiger Prawns	Lyons	Thailand/Indonesia	Off	17/10/2006	May-07	"	Lyons Seafood	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1569	Black Tiger Shrimp	Shrimp	Vietnam	On	17/10/2006	Oct-07	Booker Cash and Carry, Excelsior Road Industrial Estate, Cardiff CF14 3XL	Thai Than Seafood Factory (Vietnam)	Negative
06N1538	Black Tiger Shrimp	Neptune Gambas	Bangladesh	On	12/10/2006	Jun-08	CT Holmes, Shop 17, Billingsgate Market, Trafalgar Way, London E14 5ST	Not available	Negative
06N1539	Black Tiger Prawn	Crystal	Malaysia	On	12/10/2006	Jun-08	CT Holmes, Shop 17, Billingsgate Market, Trafalgar Way, London E14 5ST	Eastern Global (M) Sdn. Dhd. Perak Malaysia	Negative
06N1540	Black Tiger Shrimp	Fresh & Fresh	Vietnam	On	12/10/2006	Feb-08	John Stockwell, 14 Billingsgate Market, Trafalgar Way, London E14 5ST	Cafatex Seafood Vietnam	Negative
06N1541	Black Tiger Prawns	Indian Star	India	On	12/10/2006	May-08	John Koch, Shop 1, Billingsgate Market, Trafalgar Way, London E14 5ST	Baby Marine Exports (Packers) India	Negative
06N1542	Gambero blu congelato	Gambero Blu	Thailand	On	13/10/2006	Nov-07	J & P Exotics, Shop 5 and 6, Billingsgate Market, Trafalgar Way, London E14 5ST	Thai Spring Fish Co.	<b>Positive 1.6µg/kg SEM</b>
06N1543	Shrimp	Surinam Pearl	Surinam	On	Oct-06	Jan-08	J & P Exotics, Shop 5 and 6, Billingsgate Market, Trafalgar Way, London E14 5ST	Not available	Negative
06N1232	Black Tiger Shrimp	SIRI Special	India	Off	08/10/2006	Not available	Marpeche Seafoods Ltd, 136 Westbury Leigh, Wilts BA13 3SH	M/s Jagadeesh Marine Exports, India	Negative
06N1233	Black Tiger Shrimp	Bay One	India	Off	09/10/2006	Not available	Sea Products International Ltd, Ocean House, Wholesale Market Precinct, Pershore Street, Birmingham B5 6UU	Axis Trading, India	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1234	Black Tiger King Prawn	Vital	India	On	11/10/2006	Not available	Sea Products International Ltd, Ocean House, Wholesale Market Precinct, Pershore Street, Birmingham B5 6UU	Premier Marine Products, India	Negative
06N1235	Ecuadorian Shrimp	Coral	Ecuador	Off	06/10/2006	"	Seafood Centre International, Prospect House, 67 Boston Manor Road, Brentford TW8 9JQ	Promarisco SA, Ecuador	Negative
06N1236	Shrimp	Wanya	China	Off	13/10/2006	Jul-08	Chan Brothers Ltd, 17-25 Shirley Street, London E16	Shantou Wanya Foods Fty Co Ltd, China	Negative
06N1237	Black Tiger Shrimp	Castle	Bangladesh	On	15/10/2006	Not available	Seamark PLC, Seamark House, Edge Lane, Droylsden, Manchester M43	Salam Seafoods Ltd, Bangladesh	Negative
06N1238	King Prawns	Ocean Eagle	India	Off	17/10/2006	"	Sea Products International Ltd, Ocean House, Wholesale Market Precinct, Pershore Street, Birmingham B5 6UU	Premier Marine Products, India	Negative
06N1239	Black Tiger Prawn	Vinner	India	Off	03/10/2006	"	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	Wellcome Fisheries Ltd, India	Negative
06N1240	Black Tiger Shrimp	Samudra Sea Foods	India	On	04/10/2006	"	Spiceway Supermarket, Unit 4, College Fields, Merton SW19 2PX	Amulya Sea Foods, India	Negative
06N1259	Black Tiger Shrimp	Chandra Premium	India	On	08/10/2006	"	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	Sri Chandrakantha Marine, India	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1571	Cooked Shrimp with Marie Rose Sauce	Asian Classic	Vietnam	Off	04/11/2006	07/07/2008	Costco, West Thurrock Way, Essex RM20 3WY	Icelandic Seafood	Negative
06N1572	Tiger Prawns	Kirkland	SE Asia	Off	04/11/2006	07/11/2006	"	Not available	Negative
06N1573	Cooked Shrimp	Asian Classic	Vietnam	Off	04/11/2006	27/11/2007	"	Icelandic Seafood	Negative
06N1574	Whole cooked Crevettes	Ocean Pearl	Indonesia	On	04/11/2006	Feb-08	"	Sea Products International Ltd	Negative
06N1575	Shrimp	Asian Classic	Vietnam	Off	04/11/2006	Jun-08	"	Icelandic Seafood	Negative
06N1576	Cooked and peeled Prawns	Seabest	Indonesia	Off	06/11/2006	Dec-07	Seewoo, Furlong House, Horn Lane, Greenwich SE10 0ET	Not available	Negative
06N1577	Black Tiger Shrimp	Coral	Bangladesh	On	06/11/2006	Jul-07	"	Saint Martin Seafood Ltd	Negative
06N1578	Pink Shrimp	Seabest	India	Off	06/11/2006	30/04/2008	"	Seewoo Foods Ltd	Negative
06N1579	Black Tiger Prawns	Vital	India	On	06/11/2006	30/04/2008	"	Kader Investments & Trading Co.	Negative
06N1580	Black Tiger King Prawns	Seahawk	India	Off	06/11/2006	Jul-08	"	Seahawk Marine Foods	Negative
06N1581	Prawn	Classic	Indian Ocean	On	06/11/2006	Mar-08	"	Seamark PLC	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1544	Black Tiger Gambas	Neptune	Bangladesh	On	07/11/2006	31/12/2008	CT Holmes, Shop 17, Billingsgate Market, Trafalgar Way, London E14 5ST	Conception Food	Negative
06N1545	Frozen Raw Prawns	Gold Ribbon	Malaysia	On	07/11/2006	Feb-08	CT Holmes, Shop 17, Billingsgate Market, Trafalgar Way, London E14 5ST	Ocean Pack	Negative
06N1546	Black Tiger Shrimp	Fresh & Fresh	Vietnam	On	07/11/2006	Aug-08	John Stockwell, 14 Billingsgate Market, Trafalgar Way, London E14 5ST	Cafatex Seafood Vietnam	Negative
06N1547	Black Tiger Prawns	Indian Star	India	On	07/11/2006	May-08	John Koch, Shop 1, Billingsgate Market, Trafalgar Way, London E14 5ST	Baby Marine Export	Negative
06N1548	Frozen Shrimp	Surinam Pearl	Surinam	On	07/11/2006	Nov-07	J & P Exotics, Shop 5 and 6, Billingsgate Market, Trafalgar Way, London E14 5ST	Not available	Negative
06N1549	Prawn	Gambero Blu	Thailand	On	07/11/2006	Nov-07	J & P Exotics, Shop 5 and 6, Billingsgate Market, Trafalgar Way, London E14 5ST	Thai Spring Fish Co.	<b>Positive</b> <b>1.6µg/kg SEM 1.1µg/kg</b> <b>AOZ</b>
06N1280	King Prawns in Tempura Butter	Seahawk	Thailand	Off	17/10/2006	Not available	Seahawk Marine Foods Ltd, Broadleigh House, Woodmarsh, Trowbridge, Wilts BA14 0JA	Kingfisher Holding Ltd, Bangkok, Thailand	Negative
06N1288	Aqua/Raw	Farm Delight	Ecuador	Off	25/10/2006	Mar-08	Youngs Bluecrest Seafood Ltd, PO Box 513, Ross House, Wickham Road, Grimsby DN31 3WB	Promarisco SA, Guayaquil, Ecuador	Negative
06N1226	Raw peeled pulled vein Tail-on farmed shrimp	No brand	Nicaragua	Off	19/10/2006	Not available	Lyons Seafoods Ltd, Fairfield House, Fairfield Road, Warminster, Wiltshire BA12 9DA	Camerones De Nicaragua SA, Nicaragua	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1265	Shrimp	Seadragon	Thailand	Off	13/10/2006	Not available	Yet Sun Hon Ltd, Aldermans Ind. Est, Coventry CV2 2PJ	Fung Lea Food PTY Ltd, NSW 2210, Australia	Negative
06N1266	Black Tiger Shrimp	Indian Star	India	Off	25/10/2006	"	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	Baby Marine (Eastern) Exports, Tamil Nadu State, India	Negative
06N1267	Breaded King Prawn Skewers	Iceland	Thailand	Off	27/10/2006	"	Iceland Foods Ltd, Second Ave, Deeside Ind. Park, Flintshire CH5 2NW	Charoen Pokphand Foods Public Company Ltd, Thailand	Negative
06N1268	Raw Prawns	Ameena	India	Off	01/11/2006	"	Lyons Seafoods Ltd, Fairfield House, Fairfield Road, Warminster, Wiltshire BA12 9DA	High Seas Exim, Kerala Cochin, India	Negative
06N1241	Black Tiger Shrimp	Nila	India	On	25/10/2006	"	Ice Pak International, 49 Barkley Road, Leeds LS11 7EN	Nila Seafoods (P) Ltd, Tuticorin, Tamilnadu, India	Negative
06N1242	Cooked and peeled Prawns	L & T Red	Indonesia	Off	27/10/2006	"	Scotch Frost of Glasgow Ltd, Bothwell Park Industrial Estate, Hornal Road, Uddington, Glasgow G71 6NZ	PT Sekar Bumi TBK, Jalan, Raya Darmo 23-25, Indonesia	Negative
06N1243	Shrimp	King	Bangladesh	On	26/10/2006	"	Bloomsbury International Ltd, 27 Knightsbridge, London SW1X 7LY	Bagerhat Seafood Industries Ltd, 51 Khan-a-Sabur Road, Khulna, Bangladesh	Negative
06N1244	Black Tiger Shrimp	HJR	Philippines	On	26/10/2006	"	Seamark Plc, Seamark House, Edge Lane, Droylsden, Manchester M43 6BB	Bulacan HJR International Corp. Lot 3 Block 1 Phase III, Sterling Industrial Park, Libtong Meycauayan, Bulacan, Philippines	Negative
06N1245	Black Tiger Prawns	Sea Gold	India	Off	25/10/2006	"	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	Falcon Marine Exports Ltd, A/22 Cuttack Road, Falcon House 1st Floor, Orissa Bhubaneswar, India	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1246	Black Tiger Prawns	Brakes	Vietnam	On	24/10/2006	Not available	Diamond Seafoods (UK) Ltd, 3rd Floor 107, Cannon Street, London EC4N 5AD	Kim Anh Co Ltd, 49 National Highway, 1 Quarter, 2 Soc Trang Province, Soc Trang, Vietnam	Negative
06N1247	Scampi	Akasaka Special	India	On	25/10/2006	"	Landauer Ltd, 24 Beaufort Court, Admirals Way, London E14 9XL	Nekkanti Sea Foods Ltd, 1 Jayaprada Apts, Nowroji Road, MR Peta, Visakhapatnam, India	Negative
06N1248	Shrimp	Tiger	Thailand	Off	24/10/2006	"	Seamark Plc, Seamark House, Edge Lane, Droylsden, Manchester M43 6BB	Narong Seafood Co Ltd, 5/4-6 Moo 4. Seatakit Road, Samutsakhon, Thailand	Negative
06N1249	Tiger Prawns	Sindhu Special	India	Off	20/10/2006	"	Sea Products International Ltd, Ocean House, Wholesale Market Precinct, Pershore Street, Birmingham B5 6UU	M/S Devi Fisheries Ltd, 7-8-20/1 Kasturiba Marg, Visakhapatnam - 530 003, India	<b>Positive 14µg/kg AOZ</b>
06N1587	Black Tiger	Bay Star	India	Off	09/12/2006	Aug-08	Wing Yip, Oldham Road, Ancoats, Manchester M4 5HU	Sai Marine Exports Ltd	Negative
06N1586	Black Tiger Prawns	Tiger Delight	Vietnam	Off	Dec-06	Jul-08	Wing Yip, Oldham Road, Ancoats, Manchester M4 5HU	Vision Seafoods	Negative
06N1585	Black Tiger	Golden Ribbon	Malaysia	On	19/12/2006	14/02/2008	Wing Yip, Oldham Road, Ancoats, Manchester M4 5HU	Oceanpac SDN	Negative
06N1584	Metapenaeus	Admiral	Indonesia	Off	09/12/2006	Jan-08	Wing Yip, Oldham Road, Ancoats, Manchester M4 5HU	Mega Marine Pride	Negative
06N1583	Tiger Prawns	Sea Rainbow	Malaysia	Off	09/12/2006	24/05/2008	Wing Yip, Oldham Road, Ancoats, Manchester M4 5HU	Kuok Sui Sea Products	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1582	Pink Tiger Prawns	Cooks Brand	Malaysia	Off	09/12/2006	May-08	Wing Yip, Oldham Road, Ancoats, Manchester M4 5HU	Not available	Negative
06N1556	Prawn	W Wing Yip PLC	Thailand	Off	10/12/2006	May-08	Wing Yip, 375 Nechells Park Road, Birmingham B7 5NT	Not available	Negative
06N1557	Prawn	Tiger Delight	Malaysia	On	10/12/2006	Feb-09	"	Vision Seafoods	Negative
06N1558	King Prawns	W Wing Yip PLC	Bangladesh	On	10/12/2006	May-08	"	Not available	Negative
06N1559	King Prawns	W Wing Yip PLC	India	Off	10/12/2006	May-08	"	"	Negative
06N1560	King Prawns	Royal Star	Vietnam	On	10/12/2006	24/07/2008	"	"	Negative
06N1552	Black Tiger	Platinum	Indonesia	On	13/12/2006	16/07/2008	John Koch, Shop 1, Billingsgate Market, Trafalgar Way, London E14 5ST	Leeuwarden	Negative
06N1553	Black Tiger Shrimp	Fresh & Fresh	Vietnam	On	13/12/2006	Aug-08	John Stockwell, 14 Billingsgate Market, Trafalgar Way, London E14 5ST	Leeuwarden	Negative
06N1554	Black Tiger Shrimp	Conch brand	Bangladesh	On	13/12/2006	Not available	John Stockwell, 14 Billingsgate Market, Trafalgar Way, London E14 5ST	M.U Seafoods Ltd	Negative
06N1555	Black Tiger	SKB	Indonesia	On	13/12/2006	Sep-08	R. A. O, 21 Billingsgate Market, Trafalgar Way, London E14 5ST	Sekar Bumi	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1271	Prawn	Lucky Star	Indonesia	On	12/11/2006	Not available	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	PT Laura Indo JL Sabar no 59, Prajen Muba, Indonesia	Negative
06N1272	Prawn	Lyons	Indonesia	Off	13/12/2006	"	Lyons Seafoods Ltd, Fairfield House, Fairfield Road, Warminster, Wiltshire BA12 9DA	PT Centalperti Bahari, Indonesia	Negative
06N1273	Prawn	Java Village	Indonesia	Off	20/11/2006	"	Diamond Seafoods (UK) Ltd, 3rd Floor 107, Cannon Street, London EC4N 5AD	PT Sumber Kalimantan Abadi, Indonesia	Negative
06N1274	Prawn	Chandra Premium	India	On	21/11/2006	"	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	Sri Chandrantha Marine, India	Negative
06N1275	Prawn	Amantha Premium	India	Off	03/12/2006	"	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	Jaya Satya Marine, India	Negative
06N1276	Shrimp	SL	Indonesia	Off	05/12/2006	"	Libra Seafoods Ltd, Viking Business Park, Rolling Mill Road, Jarrow, Tyne and Wear	Not available	Negative
06N1227	Prawn	Naturland	Ecuador	Off	16/11/2006	"	Lyons Seafoods Ltd, Fairfield House, Fairfield Road, Warminster, Wiltshire BA12 9DA	Expalsa Exportadora De Alimentos SA, Ecuador	Negative
06N1228	Prawn	None	Honduras	Off	09/11/2006	"	Lyons Seafoods Ltd, Fairfield House, Fairfield Road, Warminster, Wiltshire BA12 9DA	Empacadora Deli SA, Honduras	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1229	Frozen shrimp	Farallon	Panama	Off	10/11/2006	"	Salmon Poachers Ltd, Units 1-3 Salisbury Road Business Park, Pewsey, Wiltshire SN9 5PZ	Farallon Aquaculture SA, Panama	Negative
06N1283	Raw Prawns	Spring	India	On	09/11/2006	Not available	Northcoast Seafoods Ltd, Europarc Innovation Centre, Innovation Way, Grimsby, NE Lincs DN37 9TT	Star Agro Marine Exports Pvt Ltd, India	Negative
06N1250	Black Tiger Prawn	Choi-Sun	India	Off	15/11/2006	"	Chans Limited, 37-39 Farnell Street, Glasgow G4 9SE	Sai Marine Exports Ltd, India	Negative
06N1251	Prawn	CPF	Thailand	Off	15/11/2006	"	Bloomsbury International Ltd, 27 Knightsbridge, London SW1X 7LY	Charoen Pokphand Foods Public Company Ltd, Thailand	Negative
06N1252	Black Tiger Prawn	Mehnaz	Bangladesh	Off	15/11/2006	"	J Sykes & Sons, New Smithfield Market, Manchester M11 2WJ	Kuliarchar Seafoods (Cox's Bazar) Ltd, Bangladesh	Negative
06N1253	Prawn	Harvest	India	Off	15/11/2006	Not available	Sea Products International Ltd, Ocean House, Wholesale Market Precinct, Pershore Street, Birmingham B5 6UU	Asvini Fisheries PVT Limited, India	Negative
06N1254	Iced King Prawns	Zoy	Bangladesh	On	16/11/2006	"	Scotch Frost of Glasgow Ltd, Bothwell Park Industrial Estate, Hornal Road, Uddington, Glasgow G71 6NZ	Apex Foods Ltd, Bangladesh	Negative
06N1255	Prawn	Royal Gold	India	On	15/11/2006	"	Nortrade Seafood, Cranbrook Road, Gills Green Hawkhurst, Cranbrook TN18 5HB	Falcon Marine Exports Ltd, A/22 Cuttack Road, Falcon House 1st Floor, India	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1256	Prawn	Lilly	Bangladesh	Off	16/11/2006	"	Seamark Plc, Seamark House, Edge Lane, Droylsden, Manchester M43 6BB	Seamark (BD) Ltd, Bangladesh	Negative
06N1257	Prawn	Bamboo	India	Off	29/11/2006	"	Landauer Ltd, 24 Beaufort Court, Admirals Way, London E14 9XL	Five Star Marine Exports Private Ltd, India	Negative
06N1258	Prawn	Landauer	China	On	23/11/2006	"	Landauer Ltd, 24 Beaufort Court, Admirals Way, London E14 9XL	Yacheng Haiteng Aquatic Products and Food Co Ltd, China	Negative
06N1647	Black Tiger Prawns	HJR	Philippines	On	01/12/2006	"	Seamark Plc, Seamark House, Edge Lane, Droylsden, Manchester M43 6BB	Bulacan HJR International Corp. Lot 3 Block 1 Phase III, Sterling Industrial Park, Libtong Meycauayan, Bulacan, Philippines	Negative
06N1648	Prawn	Lucky	Indonesia	Off	11/12/2006	"	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	P.T Indra Surya Indoperkasa, Indonesia	Negative
06N1649	Black Tiger	Beseaco	Vietnam	Off	06/12/2006	<b>Not availbale</b>	Ruskim Seafoods Ltd, Marine House, Stafford Park 15, Telford, Shropshire TF3 3BB	Bentre Seafood Joint Stock Company, Vietnam	Negative
06N1650	Black Tiger	Tiger Delight	India	Off	08/12/2006	"	Bloomsbury International Ltd, 27 Knightsbridge, London SW1X 7LY	Asvini Fisheries PVT Limited, India	Negative
06N1652	Black Tiger	Minh Phu	Vietnam	On	22/11/2006	"	Nortrade Seafood, Cranbrook Road, Gills Green Hawkhurst, Cranbrook TN18 5HB	Minh Phu Seafood Corp., Vietnam	Negative

Sample number	Type	Brand	Country of Origin	Shell	Date of Purchase/ Collection	Best Before Date	Wholesaler/ Importer	Supplier/Exporter	Result and Value at or above *MRPL/RL (µg/kg)
06N1653	Black Tiger	Oriental Harvest	Vietnam	Off	21/11/2006	"	Meadowvale Ltd, Wrexham Road, Malpas, Cheshire SY14 7EJ	Fine Foods Limited, Vietnam	Negative
06N1655	Prawn	None	China	On	16/11/2006	"	Landauer Ltd, 24 Beaufort Court, Admirals Way, London E14 9XL	Yacheng Haiteng Aquatic Products and Food Co Ltd, China	Negative
06N1656	Prawn	Wanya	China	On	22/11/2006	"	Chan Brothers Ltd, 17-25 Shirley Street, London E16	Shantou Wanya Foods Fty Co Ltd, China	Negative
06N1657	Prawn	Kohinoor	Bangladesh	On	08/12/2006	"	Seamark Plc, Seamark House, Edge Lane, Droylsden, Manchester M43 6BB	Asian Seafood Ltd, Bangladesh	Negative
06N1658	Prawn	Sea Farms	Honduras	Off	03/12/2006	"	Aqua Star Europe, Eagle House, The Slough, Studley B80 7EN	Empacadora De Productos Acuaticos San Lorenzo, Honduras	Negative

## Methodology

All prawn samples were transported to Central Science Laboratory frozen and maintained frozen on arrival. A portion of each sample was homogenised by passing twice through a mincer. Homogenised sample, including shell where shell-on samples received, was used for screening analysis. A portion of unhomogenised sample was stored separately for confirmatory analysis should this be necessary. For confirmatory analysis shell was removed before homogenisation. All sub samples were stored frozen at -18°C prior to analysis.

Samples were analysed by validated UKAS accredited methodology. The principles of the method are as follows:

Nitrofuran metabolites are extracted from tissue with 0.2M hydrochloric acid. The metabolites are derivatised with 2-nitrobenzaldehyde, and the sample neutralised with 0.2M dipotassium hydrogen orthophosphate and 2M sodium hydroxide. The extract is applied to a Solid Phase Extraction (SPE) cartridge. The 2-nitrobenzaldehyde derivatives are eluted with ethyl acetate. The eluate is evaporated to dryness and the residue redissolved in methanol/water mobile phase. The nitrofuran derivatives are determined by reversed phase LC-MSMS. Extracted matrix standards are used for calibration and d<sub>5</sub>-AMOZ, d<sub>4</sub>-AOZ, 2,4,5-[<sup>13</sup>C<sub>3</sub>]-AHD & 1,2-[<sup>15</sup>N<sub>2</sub><sup>13</sup>C]-SEM are used as internal standards which gives implicit recovery correction for all analytes.

For confirmation of SEM residues initial washing of the confirmation sub sample is performed according to EU CRL guidelines to remove free SEM and leave bound SEM for measurement. The sample pre-treatment involves washing with methanol, ethanol, and diethyl ether prior to hydrolysis and derivatisation.

## Quality Assurance

Samples were screened using LCMSMS. Suspect non-compliant samples were confirmed quantitatively by LCMSMS. Confirmatory analysis was performed on a separate sub sample.

All screening batches included known blank samples and extracted two level matrix matched standards (i.e. a known amount of analyte added to a known blank prior to extraction). All results were recovery corrected against d<sub>5</sub>-AMOZ, d<sub>4</sub>-AOZ, 2,4,5-[<sup>13</sup>C<sub>3</sub>]-AHD & 1,2-[<sup>15</sup>N<sub>2</sub><sup>13</sup>C]-SEM internal standards.

All quantitation batches included multilevel matrix matched standards and duplicate sample extracts in addition to the QA samples included for screening.

Quality assurance was also determined by participation in UK and EU proficiency schemes and collaborative studies.

**Method validation**

The LCMSMS method for determining nitrofurantoin metabolites was validated at CSL according to the requirements of Commission Decision 2002/657/EC. This required the determination of a number of key parameters including “CC $\alpha$ ” (Decision limit) and “CC $\beta$ ” (Detection capability):

	<b>SEM</b>	<b>AOZ</b>	<b>AHD</b>	<b>AMOZ</b>
<b>CC<math>\alpha</math> (<math>\mu\text{g}/\text{kg}</math>)</b>	0.01	0.03	0.09	0.20
<b>CC<math>\beta</math> (<math>\mu\text{g}/\text{kg}</math>)</b>	$\leq 0.5$	$\leq 0.5$	$\leq 1.0$	$\leq 0.25$