

# Report on surveillance of infant food for pesticide residues

October, 2006

## **Table of Contents**

<u>Summary</u>	2
Introduction	3
Pesticide Residues in Food	3
<b>Legislation</b>	4
Study details	5
Results	8
Conclusion	12
Appendix I	18
Appendix II	19
Tables	
Table 1 Sample details	7
Table 2 Levels of pesticide residues detected in infant food sa	amples13
Table 3 Pesticides which shall not be present* in infant form	,
and processed cereal-based foods and baby foods for infants	and young children18
Table 4 Pesticides to which specific maximum residue levels	apply18
Table 5 Pesticides included in this survey	19

#### **Summary**

The Food Safety Authority of Ireland has carried out a surveillance study of infant food and infant juices available on the Irish market for the possible presence of pesticide residues, in order to establish levels of compliance with existing legislation in this area. This survey follows on from a previous study carried out by FSAI in 2004. The legislation on processed food for infants and young children ensures that these products generally contain no, or extremely low levels of, pesticide residues, in order to ensure maximum health protection for this vulnerable group of the population. Pesticide residue levels in processed food for infants and young children should not exceed a Maximum Residue Level (MRL) of 0.01 mg/kg (0.01 parts per million, ppm) for the majority of individual pesticides and even lower levels apply to certain specified pesticides.

50 samples of baby food, comprising juices, jars and boxes (cereal/meat/fruit/vegetable based) were analysed in this survey. Samples purchased were "ready to eat", and were analysed as such, or sold in the dry form, requiring reconstitution before use. In the case of the latter, samples were analysed in the reconstituted form in accordance with the manufacturers' instructions. A total of 386 pesticides were included in the survey.

Of the 50 samples tested for 386 pesticide compounds, representing 19,300 individual results, a total of ten positive results for the presence of residues were obtained. These residues comprised o-phenylphenol, piperonylbutoxide, fenhexamide, maleic hydrazide, propylenethiourea/ ethylenethiourea (PTU/ETU) and diphenylamine. However, only two out of ten residues detected were above the MRL established for these pesticides in baby food. The two residues detected above the MRL were o-phenylphenol, found in one product, and maleic hydrazide which was detected in two separate products.

Follow up investigations in the case of *o*-phenylphenol showed that the presence of the residue was not as a result of the use of *o*-phenylphenol as a plant protection product, but as a result of its presence in the packaging material of the baby food. As a result of this finding, the manufacturer of the baby food products has notified his packaging suppliers and new specifications have been drawn up for the packaging materials used in the manufacture of this product.

Maleic hydrazide was detected in two separate products at concentrations of 0.1mg/kg and 0.7 mg/kg. In the case of the product containing maleic hydrazide at 0.1mg/kg, investigations carried out by the manufacturer have revealed that the source of the residue was an onion powder used in the recipe. As a result the manufacturer has switched to a different onion powder supplier and has taken all necessary steps to correct the problem.

In the case of the second product, which contained maleic hydrazide at 0.7 mg/kg, all suppliers of the ingredients used in its manufacture have indicated that no maleic hydrazide was used on their raw materials. However, as a result of these findings the baby food manufacturer has set up a system of positive release to include testing for maleic hydrazide on the four raw ingredients present in the product and in addition full supplier agronomy audits will take place later this year.

FSAI considers that the residue levels found in the products concerned presented no risk to infants and young children. The manufacturers involved have taken appropriate follow up action

and have put in place the relevant checks and safeguards in order to ensure that this problem does not occur in the future. The FSAI is satisfied that all necessary steps have been taken to ensure legal compliance with the requirements of S.I. 242 of 2004 and S.I. 433 of 2004 as they relate to levels of pesticides. Overall the results of the study show good compliance with the existing legislation on pesticide residues in baby food and FSAI will continue to monitor such products for pesticide residues and other chemical contaminants, in order to safeguard the health of Irish children.

#### Introduction

The Food Safety Authority of Ireland has carried out a surveillance study of infant food and infant juices available on the Irish market for the possible presence of pesticide residues in order to establish levels of compliance with legal requirements for these products.

The Food Safety Authority of Ireland (FSAI) has a statutory responsibility to ensure the safety of food consumed, distributed, produced and sold on the Irish market. In this respect, the FSAI coordinates the collation of food safety surveillance information from laboratories run by its official agents including the Health Service Executive, the Department of Agriculture and Food, the Department of Communications, Marine and Natural Resources, the Marine Institute and the local authorities. The FSAI also conducts targeted food safety surveillance in areas where potential safety issues have been identified and/or on food contaminants for which there are currently no testing capacities in Ireland, such as very low levels of pesticide residues in infant foods. This report provides the results of a targeted surveillance study on levels of 386 pesticide residues in foods for infants and young children.

#### **Pesticide Residues in Food**

Pesticides residues may be present on crops as a result of direct application, incidentally through drift or through contamination from irrigated water, for example. There are currently about 980 pesticide products registered for use in Ireland by the Pesticides Control Service of the Department of Agriculture and Food (<a href="http://www.pcs.agriculture.gov.ie/">http://www.pcs.agriculture.gov.ie/</a>). Although use of pesticides has many benefits in terms of crop and animal production, some of these pesticides and/or their metabolites or breakdown products are relatively toxic substances and could potentially have harmful effects on consumers, wildlife and the environment. Before a plant protection product is approved for use, it must undergo a rigorous safety assessment. These products will not be approved for use if they demonstrate harmful effects on the health of humans, animals or the environment. The assessment of these substances includes an assessment of the parent substances, metabolites and breakdown products. It is only in cases of serious abuse, where plant protection products are not applied correctly, that a risk to the consumer or others arises.

The European Union has established maximum residue levels (MRLs) for about 150 pesticide active substances in food commodities, in order to ensure the safety of consumers. The MRL is the maximum concentration of pesticide residue legally permitted in a particular food commodity and is derived from field application trials with the pesticide, performed according to good

agricultural practice (GAP). An MRL of 0.01 mg/kg (0.01 parts per million, ppm) or lower has been established for pesticide residues in processed food for infants and young children. This MRL is legally distinct from the wider EU scheme for MRLs in a range of food commodities consumed by adults and older children, and reflects the need to ensure maximum health protection for this vulnerable group of the population, as detailed further below.

The MRL is not a health-based exposure limit for a pesticide in food, although an MRL is only established when it is shown that it is safe for consumers. The toxicological endpoints used to determine the acceptability of the MRL are the ADI (acceptable daily intake) and where relevant, for acutely toxic pesticides, the ARfD (the acute reference dose). The ADI is the amount of a pesticide or other chemical that can be consumed every day for a lifetime with reasonable confidence that no harmful effects on health will result, while the ARfD is the amount of a pesticide or other chemical that can be consumed at one meal or in one day with reasonable certainty that no harmful effects on health will result. The ADIs or ARfDs apply to adults and young children alike, but in considering the implications of an MRL exceedance in a particular food commodity, account has to be taken of the fact that young children may be higher consumers on a bodyweight basis of particular foods such as fruit when compared to adults. This may make them more susceptible or at greater risk of exceeding the ADI or ARfD.

#### Legislation

MRLs are established for the majority of commonly used pesticides in various food commodities (fresh fruit and vegetables, cereals and foodstuffs of animal origin) via the legal framework of Directives 76/895/EEC, 86/362/EEC, 86/363/EEC, 90/642/EEC and Regulation (EC) No. 396 of 2005. In addition, it has been agreed at EU level that pesticide residues should preferably not be present in processed food for infants and young children, but if present, that such residues should be as low as reasonably achievable. Accordingly specific legislative requirements regarding the occurrence of pesticide residues in such food have been laid down in the following EU Directives:

- 1. Commission Directive 91/321/EEC on infant formulae and follow-on formulae as amended <sup>1</sup>
- 2. Commission Directive 96/5/EC on processed cereal-based foods and baby foods for infants and young children as amended<sup>2</sup>

The national Regulations implementing these Directives are the European Communities (Infant Formulae and Follow-on Formulae) Regulations 2004, S.I. No. 242 of 2004 and the European Communities (Processed Cereal-based foods and Baby Foods for Infants and Young Children) Regulations 2004, S.I. No. 433 of 2004.

<sup>1</sup> By Commission Directive 96/4/EC, Commission Directive 1999/50/EC and Commission Directive 2003/14/EC 2 By Commission Directive 1998/36/EC, Commission Directive 1999/39/EC and Commission Directive 2003/13/EC

Article 6 of both Directives requires that infant formulae, follow-on formulae and processed cereal-based foods and baby foods for infants and young children "shall not contain any substance in such quantity as to endanger the health of infants and young children". The Directives specifically require that these products must not contain residues of individual pesticides at levels exceeding 0.01 mg/kg. The MRL of 0.01 mg/kg applies to the product as sold ready for consumption (e.g. ready to eat baby food) or after reconstitution according to the instructions of the manufacturer (e.g. infant formulae, dried cereal-based foods, etc.)

In addition, in March 2005, more stringent restrictions on these foods were introduced for a small number of pesticides that are considered more toxic and have a very low acceptable daily intake (ADI). The level of residues of these pesticides allowed in infant formula and manufactured baby foods is lower than 0.01 mg/kg in order to ensure that babies and young children are at no risk of exceeding the ADI. However, since most of these pesticides are not permitted for use in the EU, these lower MRLs are primarily precautionary measures. They are divided into two groups:

- 1. Pesticides that should not be present in agricultural products intended for the production of infant formulae, follow-on formulae and processed cereal-based foods and baby foods for infants and young children (see Appendix I, Table 3)
- 2. Pesticides to which a specific maximum residue applies (see Appendix I, Table 4)

This study was undertaken in order to determine whether processed food for infants and young children available on the Irish market was compliant with the above legal requirements.

#### **Study Details**

In this survey, a total of 50 samples as detailed in Table 1 were purchased in retail outlets in Dublin, comprising the following:

•	Vegetable/meat based infant food	19 samples
•	Fruit-based infant food	13 samples
•	Cereal-based infant food	11 samples
•	Biscuits for infants	4 samples
•	Juices for infants and young children	3 samples

The survey was not intended to provide a comprehensive survey of all infant foods currently available on the Irish market. Rather, it was intended to provide a representative sample of those infant foods in which pesticide residues might possibly be detected (in the main, cereal-, fruit-and vegetable-based products). The foods sampled included organic baby foods, in which the presence of pesticide residues would not be expected. Almost 40% of all samples taken were stated to be organic, which approximately represents the relative proportion of organic to non-organic baby foods available on the Irish market.

Single samples only of each product selected for inclusion were analysed. The results obtained relate solely to the sample tested and may not necessarily be representative of the general pesticide residue status of that product. Since the survey was designed to measure broad compliance with the regulations and a risk assessment was not conducted, no inference can be drawn on the risk or the safety of the products concerned or from the results obtained for a particular brand of a product included in the survey.

The 50 samples were analysed for a total of 386 pesticide residues as detailed in Appendix II, Table 5. These 386 pesticide residues include those for which more stringent legislation came into force in March 2005 (Appendix I). The sampling phase was carried out in November 2005 and the analytical phase from December, 2005 until March 2006. Samples were analysed by Eurofins/Labor Specht & Partner in Germany, under contract to FSAI. The majority of compounds were analysed as part of an accredited Multi Residue Method (DFG S19) whilst the remainder were analysed using accredited Group Specific or Single Residue Methods. The specific chromatographic method of analysis and limit of quantitation (LOQ) of each pesticide is also provided in Table 5.

Ready-to-consume foods including juices and biscuits were analysed directly following thorough mixing. A number of the samples purchased were dried baby foods, requiring reconstitution before use. These samples are identified in Table 1 and were prepared according to the instructions of the manufacturer as indicated on the packaging of the products.

**Table 1: Sample Details** 

FSAI Sample Code	Sample Details	Ready-to-consume (rtc) or dried and reconstitution details if the latter	Best Before Date
1/2005	Boots organic apple & apricot cereal	Dried, reconstituted 1 in 5 with water	March 2007
2/2005	Cow & Gate Gluten Free fruit muesli	rtc	14.06.2007
3/2005	Cow & Gate Biscuits For Babies & Toddlers	Dried, analysed directly	14.07.2006
4/2005	Cow & Gate Organic Creamed Oat Porridge	rtc	16.05.2008
5/2005	Farleys Oats & Apple Cereal	Dried, reconstituted 1 in 3 with water	1.03.2007
6/2005	Farleys Gluten Free Rusks	Dried, analysed directly	1.03.2007
7/2005	Heinz Mums Own Creamed Porridge with pear juice	rtc	April 2006
8/2005	Heinz Mums Own Tropical muesli	rtc	August 2006
9/2005	Heinz organic apricot and strawberry breakfast	Dried, reconstituted 1 in 8 with milk (3.5% fat)	1.01.2007
10/2005	Liga Junior Biscuits	Dried, analysed directly	May 2006
11/2005	Milupa Seven Cereals	Dried, reconstituted 1 in 5 with water	8.10.2006
12/2005	Milupa Oat & Apple Cereal Breakfast	Dried, reconstituted 1 in 4 with water	28.07.2006
13/2005	Milupa Rusk With Mixed Fruit	Dried, reconstituted 1 in 4 with water	13.08.2006
14/2005	Hipp Organic Gluten Free Apple, Orange and Banana Cereal	Dried, reconstituted 1 in 8 with milk (3.5% fat)	September 2006
15/2005	Hipp Organic Creamed Porridge Breakfast	rtc	July 2006
16/2005	Boots organic Fruit and Yogurt	Dried, reconstituted 1 in 4 with milk (3.5% fat)	October 2006
17/2005	Cow & Gate Apple & Pear	rtc	6.05.2008
18/2005	Cow & Gate Concentrated Summer Fruits	Dried, reconstituted 1 in 6 with water	6.03.2007
19/2005	Cow & Gate Apple & Blackcurrant Concentrated Fruit Juice	Reconstituted 1 in 6 with water	25.05.2007
20/2005	Cow & Gate Organic Summer Fruit Compote	rtc	19.04.2008
21/2005	Heinz, Farleys recipe Strawberry Yogurt	Dried, reconstituted 1 in 3 with water	1.03.2007
22/2005	Heinz Apple & Banana	rtc	September 2006
23/2005	Heinz Apple and Orange juice from Concentrate	rtc	11.05.2006
24/2005	Heinz Organic Apple & Blueberry	rtc	30.05.2006
25/2005	Heinz Organic Banana, apple and Pear	rtc	January 2007
26/2005	Heinz Organic Apple & Apricot	rtc	November 2006
27/2005	Hipp Organic Apple & Blueberry Dessert	rtc	July 2006
28/2005	Milupa Fruit Salad	Dried, reconstituted 1 in 4 with water	15.07.2006
29/2005	Hipp Organic Apple & Pear pudding	rtc	April 2007
30/2005	Milupa Sunripe Banana	Dried, reconstituted 1 in 4 with water	19.07.2006
31/2005	Cow & Gate Dutch Apple Pudding	rtc	12.06.2008
32/2005	Heinz Mums Own Medley of Vegetables and Sweet Potatoes	rtc	January 2007
33/2005	Heinz Mums Own Cheesy Vegetable Pasta	rtc	September 2006

**Table 1: Sample Details (continued)** 

FSAI Sample Code	Sample Details	Ready-to-consume (rtc) or dried and reconstitution details if the latter	Best Before Date
34/2005	Heinz Organic cheesy carrot, cauliflower and potato	rtc	October 2006
35/2005	Heinz Organic vegetable risotto	rtc	January 2007
36/2005	Hipp Organic mixed vegetable medley	rtc	August 2007
37/2005	Hipp Organic mild vegetable and chicken Korma	rtc	March 2007
38/2005	Milupa Vegetable Risotto	Dried, reconstituted 1 in 5 with water	11.06.2006
39/2005	Milupa Cauliflower & Creamed Potato	Dried, reconstituted 1 in 3 with water	10.02.2007
40/2005	Cow & Gate Organic Cauliflower Cheese Florets	rtc	28.02.2008
41/2005	Boots Organic vegetables & chicken	Dried, reconstituted 1 in 5 with water	September 2006
42/2005	Cow & Gate Vegetable & Turkey Casserole	rtc	27.05.2008
43/2005	Cow & Gate Sunday Lunch	rtc	26.08.2008
44/2005	Cow & Gate Vegetable & Pork Dinner	rtc	28.08.2008
45/2005	Heinz Vegetable Hotpot with Chicken	rtc	March 2007
46/2005	Heinz Sweet Potato and beef mince pie	rtc	October 2006
47/2005	Heinz Mums Own Pumpkin and Lamb dinner	rtc	June 2006
48/2005	Milupa Turkey and Vegetable Dinner	Dried, reconstituted 1 in 6 with water	6.07.2006
49/2005	Heinz Organic Vegetable and Beef Stew	rtc	January 2007
50/2005	Hipp Organic Vegetables with rice and Chicken	rtc	August 2007

#### **Results**

The results obtained (see Table 2) showed good compliance with the legislation on pesticide residues in baby food. A total of ten positive results for the presence of residues were found. Overall the residues detected were fenhaxamide, diphenylamine, PTU/ETU, o-phenylphenol and maleic hydrazide. The presence of these residues in the products concerned was confirmed by repeat analysis. The levels of fenhexamide, diphenylamine and PTU/ETU were all below the limits set in the legislation. Two products included in the survey contained residues of both diphenylamine and PTU/ETU, but as mentioned previously the levels of these residues were below the legislative limit. There were however, two pesticides which were found above the MRL. These were o-phenylphenol and maleic hydrazide and they were detected in three baby food products. One of the baby foods contained o-phenylphenol at 0.17 mg/kg and the other two products contained maleic hydrazide at concentrations of 0.1 mg/kg and 0.7 mg/kg. In addition the synergist piperonylbutoxide was detected in one product at a level of 0.016 mg/kg. This substance is however not considered to be a pesticide under Directive 91/414/EC concerning the placing of plant protection products on the market, and therefore this level is not considered as an exceedance of the legislation on pesticide residues in baby food.

Bromide and chloride were also analysed in this survey. The presence of bromide in baby food can be due to the use of methyl bromide or other bromine-containing fumigant in crop production. However, bromide also occurs naturally in combination with chloride and for this reason chloride was also included in the scope of the analysis. While the levels of bromide in the majority of products was above the legislative limit of 0.01 mg/kg, ranging from 0.1 – 3.8 mg/kg (Table 2), in each case it was accompanied by higher levels of chloride, and it was concluded that the presence of bromide in the samples was attributable to its association with chloride, a normal constituent of food. In the opinion of FSAI the level of inorganic bromide found in these products would present no risk to the health of infants and young children. However, the presence of inorganic bromide as a result of concomitant chloride indicates that it is not possible to enforce an MRL of 0.01 mg/kg for bromide residues in baby food which may occur as a result of the use of methyl bromide as a fumigant

Further details on the other residue findings are outlined below:

**Fenhexamide** is a hydroxyanilide protectant fungicide and has registered uses in a number of different countries in a range of horticultural crops. It inhibits spore germ tube development and hyphal growth. The residue was found in a strawberry yoghurt below the legislative limit i.e. < 0.01 mg/kg. The most likely source of the fenhexamid in this product is the strawberries.

**Diphenylamine (DPA)** is used to prevent a storage disorder of apples and sometimes pears known as scald. It is the only known use of DPA. The incidence and severity of the disease varies, depending upon locality, seasonal conditions prior to and at harvest. It is widely used due to its antioxidant properties. The residue was detected in three products, all below the legislative limit i.e. <0.01 mg/kg. Two of these products contained apples which is the likely source of the residue. In the case of the other product it is not known where this residue originated from and it may be present as a result of adventitious contamination due to the high volatility and motility of this chemical.

**PTU/ETU**, Propylenethiourea (PTU) is a plant and animal metabolite and a degradation product of propineb. Ethylenethiourea (ETU) is a metabolite and decomposition product of the ethylenebisdithiocarbamate (EBDC) fungicides. EBDCs are used on a wide range of crops worldwide including potatoes, cereals, apples, pears and leafy vegetables. They control many fungal diseases such as blight, leaf spot, rust, downy mildew and scab. The residues of PTU/ETU were detected in a vegetable and pork dinner and also a vegetable and turkey dinner. These two products also contained residues of DPA but both of these residues were below the legislative limit i.e. <0.01 mg/kg as mentioned above. The most likely source of PTU/ETU residues is the vegetables used in the products.

**o-Phenylphenol** was detected in one savoury biscuit product at a concentration of 0.17 mg/kg. **o-Phenylphenol** is a chemical which has a number of different functions. It is used as a post harvest treatment fungicide, in which case it is regarded as a plant protection product (pesticide), but it is also used as a preservative in a variety of end-uses. The substance is also a permitted food additive (E 231) and is listed in Directive 95/2/EC where it is permitted for the surface treatment of citrus fruits only. This provision was deleted by Directive 2003/114/EC but the

deletion will only come into force as soon as requirements for the labelling of foodstuffs treated with this substance become applicable by virtue of Community legislation on MRLs for pesticides. This has not happened yet.

The Joint Expert Committee on Food Additives (JECFA) has established an ADI of 0-0.4 mg/kg bw per day for o-phenylphenol when used as a food additive. That means that a 10kg child should not consume more than 4 mg/day of o-phenylphenol. As mentioned earlier the levels found in the biscuit product was 0.17 mg/kg, this represents 4.25% of the ADI. Given that the level found was very low it would mean that a 10 kg infant would have to eat in excess of 23 kilograms of the product in a day to exceed the ADI. Therefore the levels found are considered not to pose any health risks to infants and young children.

Nevertheless, following its detection at levels above the maximum level of 0.01 mg/kg established in Directive 96/5/EC on processed cereal-based foods and baby foods for infants and young children, at the request of the FSAI the manufacturer concerned carried out extensive investigations into the possible source of the chemical in the product. These investigations showed that the presence of *o*-phenylphenol was not due to the use of pesticides on any of the raw materials/food commodities used in the manufacture of these products. Such use of pesticides is not anticipated, since manufacturers of infant formulae, cereal-based foods and baby foods for infants and young children have to source starting materials which have been produced without the use of pesticides, in order to meet the stringent specifications laid down in Directives 91/321/EEC and 96/5/EC, as amended. Rather, the presence of *o*-phenylphenol was due to its use in the food contact material (packaging) used for the product in question.

Similar results for *o*-phenylphenol in a packaged, dried baby food were found during a previous FSAI survey<sup>3</sup> on pesticides in baby food in 2004. European Union Legislation governing food contact materials is based on Regulation (EC) No 1935/2004 which is given effect in national legislation by S.I. No. 139/2006. The use of *o*-phenylphenol in food contact materials is permitted provided that the level of migration into the food is below any level that would endanger human health. Its use should also not cause any unacceptable changes in the composition of the product or bring about deterioration in the organoleptic characteristics thereof.

FSAI concludes that the level of *o*-phenylphenol found in this product presents no risk to infants and young children. The manufacturer concerned has ceased the use of the ALU-paper packaging and will now use PET-ALU-PE packaging in order to prevent the migration of the *o*-phenylphenol into any of their products. The FSAI is satisfied that all necessary steps have been taken to ensure legal compliance with the requirements of S.I. No. 242 of 2004 and S.I. No. 433 of 2004 as they relate to levels of pesticides.

Maleic Hydrazide was detected in two baby food products at concentrations of 0.1 mg/kg in a Sunday Lunch product and 0.7 mg/kg in a Medley of Vegetables and Sweet Potatoes product. Maleic hydrazide is a plant growth regulator. Its main uses are for the suppression of sprouting

\_

<sup>&</sup>lt;sup>3</sup> http://www.fsai.ie/surveillance/food/pesticide\_residues\_infant%20.pdf

in stored potatoes and bulb vegetables such as onions, the control of suckers on tobacco and as a grass growth retardant in non-crop situations.

The Joint FAO/WHO Meeting on Pesticides Residues (JMPR) established an ADI of 0-0.3 mg/kg bw per day in 1996. This means that a 10kg child should not consume more than 3 mg/day of maleic hydrazide. As mentioned earlier the levels found in these two products were 0.1 mg/kg (3.3% of the ADI) and 0.7 mg/kg (23.3% of the ADI). Given that the levels found are low it would mean that a 10 kg infant would have to eat in excess of 30 kilograms and 4.3 kilograms respectively of these products in a day in order to exceed the ADI. Therefore the levels found are considered not to have posed any health risks to infants and young children.

Nevertheless, following its detection at levels above the maximum level of 0.01 mg/kg established in Directive 96/5/EC on processed cereal-based foods and baby foods for infants and young children, at the request of the FSAI the manufacturers concerned carried out extensive investigations into the possible source/sources of the chemical in the products concerned.

In the case of the Sunday Lunch product the residue of 0.1 mg/kg maleic hydrazide detected is the same as the level of quantification (LOQ) reported by the laboratory for the analytical method. However, it should be noted that the methodology used by the laboratory to determine maleic hydrazide has not been officially validated yet and at present there is only one accepted, validated method for this testing ISO 4876:1980. Retained samples were also tested and maleic hydrazide residues were again only found at the LOQ of the analytical method. Although the level of residue in the final product was very low, the manufacturer tested all of the raw materials used in this product, and an onion powder used in the recipe was found to be the source of the residue. All other raw materials used in the product were found to be free from maleic hydrazide. However, in light of this result the manufacturers of the product have changed to another onion powder supplier. This supplier has a full supply chain controlled system in which maleic hydrazide is not used. All of the batches of this product which have been produced from the 15<sup>th</sup> of May have been produced with maleic hydrazide-free raw materials.

In the case of the Medley of Vegetables and Sweet Potatoes product the manufacturer concerned has also carried out analysis on the retained samples, using the Dr. Specht laboratory. The majority of the results were below the LOQ, however some of them were positive for maleic hydrazide at levels of 0.1 to 0.7 mg/kg. In addition, the manufacturer concerned has also contacted the suppliers of the four vegetables used as raw materials in the baby food. Three of the suppliers have confirmed that no maleic hydrazide was applied on their raw materials, while the fourth supplier has stated that his crops are managed according to organic rules and as a result they do not currently carry out analysis on the raw material in question. There is no remaining stock of the affected batch and the manufacturer involved has now set up a system of positive release to include analytical testing for maleic hydrazide for all four vegetables used in the implicated product. In addition full supplier agronomy audits will take place by the end of August.

In the case of maleic hydrazide the FSAI can conclude that the levels of this chemical found in the two baby food products presented no risk to infants and young children. The manufacturers concerned have taken the appropriate follow up action and have put in place the relevant checks and safeguards in order to ensure that this problem does not occur in the future.

**Piperonylbutoxide** is a synergist used in a wide variety of pesticides. Synergists are chemicals that lack pesticidal effects on their own but enhance the pesticidal properties of other chemicals. Piperonylbutoxide is used in pesticides containing chemicals such as pyrethrins, pyrethroids, rotenone and carbamates, and is considered to be a pesticide under Directive 91/414/EC. The residue was detected in a fruit and yoghurt product at levels of 0.016 mg/kg. This substance is however not considered to be a pesticide under Directive 91/414/EC, and therefore this level is not considered as an exceedance of the legislation on pesticide residues in baby food.

#### **Conclusion**

The FSAI is pleased to report the results of this survey, showing that a range of different baby food products and juices available on the Irish market were in compliance with the stringent legislation on pesticide residues in such products. While a low incidence of residues were found (10 positive results out of a total of 19,300 individual results, or 0.05%), only two of these were above the MRL of 0.01 mg/kg. The results are essentially similar to those found in the 2004 survey, in which a total of 5 positive results were found out of 15006 individual results, or 0.03%, three of which were above the MRL of 0.01 mg/kg.

However, FSAI emphasises to manufacturers of baby food the need to continue to take every precaution in sourcing raw materials for use in the manufacture and packaging of these products. FSAI will continue to monitor such products for pesticide residues and other chemical contaminants, in order to safeguard the health of Irish children.

Table 2 Levels of pesticide residues (mg/kg, parts per million) detected in infant food samples

FSAI Sample Code	DFG S19-multi residue method	Fipronil	Fentin-acetat	inorganic Bromide	Chloride	Haloxyfop	PTU/ETU	Propinep (within Dithiocarbamates)	Diquat	Chlormequat	Maleic Hydrazide	QuECheRS (Carbendazim, Thiophanate-methyl, Fosthiazate,Spiroxami ne)
FSAI 1/2005	n.d.	n.d.	n.d.	0,9	270	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 2/2005	n.d.	n.d.	n.d.	0,9	170	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 3/2005	o- Phenylphenol 0.17	n.d.	n.d.	3,8	2150	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 4/2005	n.d.	n.d.	n.d.	1,7	320	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 5/2005	n.d.	n.d.	n.d.	1,5	280	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 6/2005	n.d.	n.d.	n.d.	1,2	325	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 7/2005	n.d.	n.d.	n.d.	1,4	350	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 8/2005	n.d.	n.d.	n.d.	1,5	275	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 9/2005	n.d.	n.d.	n.d.	1,6	430	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 10/2005	n.d.	n.d.	n.d.	2,3	1280	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 11/2005	n.d.	n.d.	n.d.	0,7	340	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Table 2 (contd.) Levels of pesticide residues (mg/kg, parts per million) detected in infant food samples

FSAI Sample Code	DFG S19-multi residue method	Fipronil	Fentin-acetat	inorganic Bromide	Chloride	Haloxyfop	PTU/ETU	Propinep (within Dithiocarbamates)	Diquat	Chlormequat	Maleic Hydrazide	QuECheRS (Carbendazim, Thiophanate-methyl, Fosthiazate,Spiroxami ne)
FSAI 12/2005	n.d.	n.d.	n.d.	0,5	220	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 13/2005	n.d.	n.d.	n.d.	0,7	315	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 14/2005	n.d.	n.d.	n.d.	1,1	305	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 15/2005	n.d.	n.d.	n.d.	1,0	300	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 16/2005	Piperonylbutoxid 0,016	n.d.	n.d.	1,7	480	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 17/2005	n.d.	n.d.	n.d.	0,1	10	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 18/2005	n.d.	n.d.	n.d.	0,1	15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 19/2005	n.d.	n.d.	n.d.	0,1	15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 19/2004	n.d.	n.d.	n.d.	0,1	15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 20/2005	n.d.	n.d.	n.d.	0,3	10	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 21/2005	Fenhexamide < 0,01	n.d.	n.d.	1,3	240	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Table 2 (contd.) Levels of pesticide residues (mg/kg, parts per million) detected in infant food samples

FSAI Sample Code	DFG S19-multi residue method	Fipronil	Fentin-acetat	inorganic Bromide	Chloride	Haloxyfop	PTUÆTU	Propinep (within Dithiocarbamates)	Diquat	Chlormequat	Maleic Hydrazide	QuECheRS (Carbendazim, Thiophanate- methyl, Fosthiazate,Spiroxa mine)
FSAI 22/2005	n.d.	n.d.	n.d.	0,6	195	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 23/2005	n.d.	n.d.	n.d.	0,2	15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 24/2005	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 25/2005	n.d.	n.d.	n.d.	1,0	215	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 26/2005	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 27/2005	n.d.	n.d.	n.d.	0,1	15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 28/2005	n.d.	n.d.	n.d.	0,7	220	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 29/2005	n.d.	n.d.	n.d.	0,1	7	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 30/2005	n.d.	n.d.	n.d.	1,0	350	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 31/2005	Diphenylamine <0,01	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 32/2005	n.d.	n.d.	n.d.	1,1	230	n.d.	n.d.	n.d.	n.d.	n.d.	0,7	n.d.
FSAI 33/2005	n.d.	n.d.	n.d.	1,1	485	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 34/2005	n.d.	n.d.	n.d.	1,1	410	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Table 2 (contd.) Levels of pesticide residues (mg/kg, parts per million) detected in infant food samples

FSAI Sample Code	DFG S19-multi residue method	Fipronil	Fentin-acetat	inorganic Bromide	Chloride	Haloxyfop	PTU/ETU	Propinep (within Dithiocarbamates)	Diquat	Chlormequat	Maleic Hydrazide	QuECheRS (Carbendazim, Thiophanate- methyl, Fosthiazate,Spiroxa mine)
FSAI 35/2005	n.d.	n.d.	n.d.	1,5	255	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 36/2005	n.d.	n.d.	n.d.	1,1	145	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 37/2005	n.d.	n.d.	n.d.	1,6	300	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 19/2004	n.d.	n.d.	n.d.	0,7	410	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 38/2005	n.d.	n.d.	n.d.	1,2	525	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 39/2005	n.d.	n.d.	n.d.	1,0	600	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI	n.d.	n.d.	n.d.	1,0	600	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
40/2005 FSAI 41/2005	n.d.	n.d.	n.d.	0,7	190	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 42/2005	Diphenylamine <0,01	n.d.	n.d.	1,0	575	n.d.	0,004	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 43/2005	n.d.	n.d.	n.d.	1,1	445	n.d.	n.d.	n.d.	n.d.	n.d.	0,1	n.d.
FSAI 44/2005	Diphenylamine <0,01	n.d.	n.d.	0,6	405	n.d.	0,005	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 45/2005	n.d.	n.d.	n.d.	1,4	860	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 46/2005	n.d.	n.d.	n.d.	1,1	340	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

Table 2 (contd.) Levels of pesticide residues detected in infant food samples

FSAI Sample Code	DFG S19-multi residue method	Fipronil	Fentin-acetat	inorganic Bromide	Chloride	Haloxyfop	PTU/ETU	Propinep (within Dithiocarbamates)	Diquat	Chlormequat	Maleic Hydrazide	QuECheRS (Carbendazim, Thiophanate- methyl, Fosthiazate,Spiroxa mine)
FSAI 47/2005	n.d.	n.d.	n.d.	1,0	265	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 48/2005	n.d.	n.d.	n.d.	0.6	635	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 49/2005	n.d.	n.d.	n.d.	0.8	60	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
FSAI 50/2004	n.d.	n.d.	n.d.	0,9	100	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

#### Appendix I

Pesticides which shall not be present in infant formulae, follow-on formulae and processed cereal-based foods and baby foods for infants and young children or for which specific MRLs have been set.

# Table 3 Pesticides which shall not be present\* in infant formulae, follow-on formulae and processed cereal-based foods and baby foods for infants and young children

- Disulfoton (sum of disulfoton, disulfoton sulfoxide and disulfoton sulfone expressed as disulfoton)
- Fensulfothion (sum of fensulfothion, its oxygen analogue and their sulfones, expressed as fensulfothion)
- Fentin expressed as triphenyltin cation
- Haloxyfop (sum of haloxyfop, its salts and esters including conjugates, expressed as haloxyfop)
- Heptachlor and trans-heptachlor epoxide, expressed as heptachlor
- Hexachlorobenzene
- Nitrofen
- Omethoate
- Terbufos (sum of terbufos, its sulfoxide and sulfone, expressed as terbufos)
- Aldrin and dieldrin, expressed as dieldrin
- Endrin

Table 4 Pesticides to which a specific maximum residue level applies

Specific maximum residue levels of pesticides or metabolites of pesticides	MRL (mg/kg)
Cadusafos	0.006
Demeton-S-methyl/demeton-S-methyl, sulfone/oxydemeton-methyl (individually or combined, expressed as demeton-S-methyl)	0.006
Ethoprophos	0.008
Fipronil (sum of fipronil and fipronil-desulfinyl, expressed as fipronil)	0.004
Propineb/propylenethiourea (sum of propineb and propylenethiourea)	0.006

<sup>\*</sup> Pesticides are considered not to be present if their residues do not exceed a level of  $\underline{0.003 \text{ mg/kg}}$ . This level, which is considered to be the limit of quantification of the analytical method, will be kept under regular review in the light of technical progress.

## **Appendix II**

**Table 5: Pesticides included in survey**<sup>4</sup>

Group OC (Organochlorine Pesticides), Methodology: GC/ECD (DFG S19)

Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg
Aclonifen	0.005	alpha-HCH	0.001
Aldrin	0.001	beta-HCH	0.002
Benfluralin	0.001	delta-HCH	0.002
Benzoylprop-ethyl	0.005	epsilon-HCH	0.002
Binapacryl	0.005	Heptachlor	0.001
Bromoxynil-octanoat	0.002	cis-Heptachlorepoxid	0.001
alpha-Chlordan	0.001	Trans-Heptachlorepoxid	0.001
gamma-Chlordan	0.001	Hexachlorbenzol	0.001
Chlorfenapyr	0.005	Hexaflumuron	0.01
Chlorfenprop-methyl	0.005	Ioxynil-octanoat	0.001
Chlorfenson	0.005	Isobenzan	0.001
Chloroneb	0.01	Isodrin	0.001
Chlorthal-dimethyl	0.001	Isopropalin	0.002
Chlorthalonil	0.005	delta-Ketoendrin	0.005
o,p'-DDD	0.002	Lindan	0.001
p,p'-DDD	0.002	Methoxychlor	0.005
o,p'-DDE	0.002	Mirex	0.001
p,p'-DDE	0.001	Nitrapyrin	0.002
o,p'-DDT	0.002	Nitrofen	0.002
p,p'-DDT	0.001	trans-Nonachlor	0.001
Dichlobenil	0.002	Octachlorstyrol	0.001
Dicloran	0.001	Oxychlordan	0.001
p,p'-Dicofol	0.005	Pendimethalin	0.002
Dieldrin	0.001	Pentachloranilin	0.001
Dienochlor	0.005	Pentachloranisol	0.001
Dinitramin	0.002	Plifenate	0.001
Dinobuton	0.005	Polychlorterpene (Camphechlor)	0.04
alpha-Endosulfan	0.001	Profluralin	0.001
beta-Endosulfan	0.001	Quintozen	0.001
Endosulfansulfat	0.002	S 421, Octachlordipropylether	0.005
Endrin	0.002	Tecnazen	0.001
Ethalfluralin	0.002	2.3.4.6-Tetrachloranisol	0.001
Etridiazol	0.002	Tetradifon	0.002
Fenson	0.005	Tetrasul	0.005
Flubenzimin	0.002	Triallat	0.005
Fluchloralin	0.002	Trichloronat	0.002
Flumetralin	0.002	Trifluralin	0.001
Genite	0.005		

\_

<sup>&</sup>lt;sup>4</sup> grouped according to Multi Residue screen used

Group OC3 Organochlorine Pesticides, Methodology: GC/ECD (DFG S19)

Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg)	
Bifenox	0.005	Folpet	0.01	
Brompropylat	0.005	Indoxacarb	0.005	
Captafol	0.005	Iprodion	0.01	
Captan	0.005	Kresoxim-Methyl	0.005	
Chlorbenzilat	0.01	Metribuzin	0.01	
Chlorfluazuron	0.01	Nitralin	0.002	
Chlorpropylat/Chlorbenzilate	0.01	Oxadiazon	0.005	
Chlozolinate	0.005	Picolinafen	0.01	
Dichlofluanid	0.005	Picoxystrobin	0.005	
Diflufenican	0.005	Procymidon	0.01	
Hexaflumuron	0.01	Propyzamid	0.01	
Famoxadone	0.01	Spirodiclofen	0.01	
Fipronil	0.005	Tolylfluanid	0.005	
Flufenoxuron	0.01	Trifloxystrobin	0.005	
Fluorochloridon	0.005	Vinclozolin	0.005	

#### Group PY (Pyrethroid Pesticides), Methodology GC/ECD (DFG S19)

Compound	LOQ	Compound	LOQ (mg/kg)
	(mg/kg)		
Acrinathrin	0.01	Fenvalerat (RR- u. SS-Isomere)	0.005
Bifenthrin	0.01	Fenvalerat (RS- u. SR-Isomere)	0.005
Cyfluthrin	0.01	Flucythrinate	0.01
lambda-Cyhalothrin	0.005	tau-Fluvalinat	0.01
Cypermethrin	0.01	Halfenprox	0.01
Cyphenothrin	0.01	Permethrin	0.01
Deltamethrin	0.01	Tefluthrin	0.005
Fenfluthrin	0.005	Transfluthrin	0.005
Fenpropathrin	0.005		

# Group PCB (Polychlorinated Biphenyls, "Marker PCBs"), Methodology GC/ECD (DFG S19)

Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg)
PCB 28	0.005	PCB 138	0.005
PCB 52	0.005	PCB 153	0.005
PCB 101	0.005	PCB 180	0.005
PCB 118	0.005		

These are not considered pesticides but are included in the OC screen

Group OP (Organophosphorus Pesticides), Methodology: GC/FPD (DFG S19)

Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg)
Acephat	0.01	Isocarbofos	0.01
Amidothion	0.01	Isofenphos	0.01
Azinphos-ethyl	0.02	Isoxathion	0.02
Azinphos-methyl	0.03	Jodfenphos	0.01
Bomyl	0.01	Leptophos	0.02
Bromfenvinphos	0.01	Malaoxon	0.01
Bromophos (-methyl)	0.01	Malathion	0.01
Bromophos-ethyl	0.01	Mecarbam	0.01
Cadusafos	0.01	Mephosfolan	0.01
Carbophenothion (-ethyl)	0.01	Methacrifos	0.01
Carbophenothion-methyl	0.01	Methamidophos	0.01
Chlorfenvinphos	0.01	Methidathion	0.01
Chlormephos	0.01	Mevinphos	0.01
Chlorpyrifos (-ethyl)	0.01	Monocrotophos	0.01
Chlorpyrifos-methyl	0.01	Morphothion	0.02
Chlorthion	0.01	N-Desethyl-pirimiphos-methyl	0.01
Chlorthiophos	0.01	Omethoat	0.01
Coumaphos	0.03	Oxydemeton-methyl	0.01
Crufomat	0.01	Paraoxon	0.01
Cyanofenphos	0.02	Paraoxon-methyl	0.01
Cyanophos	0.01	Parathion	0.01
Demeton-S-methyl	0.01	Parathion-methyl	0.01
Demeton-S-methyl-sulfon	0.01	Phenkapton	0.02
Diazinon	0.01	Phenthoat	0.01
Dicapthon	0.01	Phorat	0.01
Dichlofenthion	0.01	Phorat-sulfon	0.01
Dichlorvos	0.01	Phosalon	0.01
Dicrotophos	0.01	Phosfolan	0.01
Dimefox	0.01	Phosmet	0.02
Dimethoat	0.01	Phosphamidon	0.01
Dioxabenzofos (Salithion)	0.01	Pirimiphos-ethyl	0.01
Dioxathion	0.01	Pirimiphos-methyl	0.01
Disulfoton	0.01	Pirimithat	0.01
Disulfoton-PS-sulfon	0.01	Profenofos	0.01
Ditalimfos	0.01	Propetamphos	0.01
Edifenphos	0.02	Prothiophos	0.01
EPN	0.02	Prothoat	0.01
Ethion	0.01	Pyraclophos	0.02
Ethoprophos	0.01	Pyrazophos	0.02
Etrimfos	0.01	Pyridaphenthion	0.01
Famophos	0.02	Quinalphos	0.01
Fenamiphos	0.01	Quintiophos	0.01
Fenchlorphos	0.01	Sulfotep	0.01
Fenitrothion	0.01	Sulprofos	0.02
Fensulfothion	0.01	TEPP	0.01
Fensulfothion-PO-sulfon	0.03	Terbufos	0.01

Fenthion	0.01	Tetrachlorvinphos	0.01
Fenthion-PO-sulfon	0.02	Thiometon	0.01
Fenthion-PS-sulfon	0.02	Thionazin	0.01
Fonofos	0.01	Tolclofosmethyl	0.01
Formothion	0.01	Triamiphos	0.02
Fosthietan	0.01	Triazophos	0.01
Heptenophos	0.01	Trichlorfon	0.03
Iprobenfos (IBP)	0.01	Trichloronat	0.01

Group LC OP (Organophosphorus Pesticides), Methodology LC-MS/MS (DFG S19)

Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg)
Cadusafos	0.001	Fensulfothion-PO-sulfoxid	0.001
Demeton-S-methyl	0.001	Fensulfothion-PO-sulfon	0.001
Demeton-S-methylsulfon	0.001	Fensulfothion-PS- sulfon	0.001
Disulfoton	0.001	Ometoat	0.001
Disulfoton-sulfon(PS)	0.001	Oxydemeton-methyl	0.001
Disulfoton-sulfoxid (PS)	0.001	Terbufos	0.001
Ethoprophos	0.001	Terbufos-sulfon	0.001
Fensulfothion	0.001	Terbufos-sulfoxid	0.001

Group ON/MS-1 (N-containing pesticides), Methodology GC/MSD (DFG S19)

Alachlor         0.01         Oxadiazon         0.01           Ametryn         0.01         Pendimethalin         0.01           Atrazin         0.01         Piperonylbutoxid         0.01           Bendiocarb         0.01         Pirimicarb         0.01           Bitertanol         0.01         Procymidon         0.01           Brompropylat         0.01         Prometryn         0.01           Bupirimat         0.01         Propachlor         0.01           Carbaryl         0.01         Propazin         0.01           Carbofuran         0.01         Propham         0.01           Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propoxur         0.01           Dichlobenil         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenarimol         0.01         Terbuthylazin         0.01	Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg)
Atrazin         0.01         Piperonylbutoxid         0.01           Bendiocarb         0.01         Primicarb         0.01           Bitertanol         0.01         Procymidon         0.01           Brompropylat         0.01         Prometryn         0.01           Bupirimat         0.01         Propachlor         0.01           Carbaryl         0.01         Propazin         0.01           Carbofuran         0.01         Propham         0.01           Chlorpopham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Triadimefon         0.01	Alachlor	0.01	Oxadiazon	0.01
Bendiocarb         0.01         Pirimicarb         0.01           Bitertanol         0.01         Procymidon         0.01           Brompropylat         0.01         Prometryn         0.01           Bupirimat         0.01         Propachlor         0.01           Carbaryl         0.01         Propazin         0.01           Carbofuran         0.01         Propham         0.01           Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimenol         0.01	Ametryn	0.01	Pendimethalin	0.01
Bitertanol         0.01         Procymidon         0.01           Brompropylat         0.01         Prometryn         0.01           Bupirimat         0.01         Propachlor         0.01           Carbaryl         0.01         Propazin         0.01           Carbofuran         0.01         Propham         0.01           Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimenol         0.01           Metalaxyl         0.01         Trifluralin         0.01 </td <td>Atrazin</td> <td>0.01</td> <td>Piperonylbutoxid</td> <td>0.01</td>	Atrazin	0.01	Piperonylbutoxid	0.01
Brompropylat         0.01         Prometryn         0.01           Bupirimat         0.01         Propachlor         0.01           Carbaryl         0.01         Propazin         0.01           Carbofuran         0.01         Propham         0.01           Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimenol         0.01           Metalaxyl         0.01         Trifluralin         0.01	Bendiocarb	0.01	Pirimicarb	0.01
Bupirimat         0.01         Propachlor         0.01           Carbaryl         0.01         Propazin         0.01           Carbofuran         0.01         Propham         0.01           Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Trifluralin         0.01	Bitertanol	0.01	Procymidon	0.01
Carbaryl         0.01         Propazin         0.01           Carbofuran         0.01         Propham         0.01           Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Brompropylat	0.01	Prometryn	0.01
Carbofuran         0.01         Propham         0.01           Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Bupirimat	0.01	Propachlor	0.01
Chlorpropham         0.01         Propiconazol         0.01           Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Carbaryl	0.01	Propazin	0.01
Chlorbenzilat/Chlorpropylat         0.01         Propargit         0.01           Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Carbofuran	0.01	Propham	0.01
Cyanazin         0.01         Propoxur         0.01           Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Chlorpropham	0.01	Propiconazol	0.01
Desmetryn         0.01         Propyzamid         0.01           Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Chlorbenzilat/Chlorpropylat	0.01	Propargit	0.01
Dichlobenil         0.01         Sebuthylazin         0.01           Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Cyanazin	0.01	Propoxur	0.01
Fenarimol         0.01         Simazin         0.01           Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Desmetryn	0.01	Propyzamid	0.01
Fenpropimorph         0.01         Terbuthylazin         0.01           Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Dichlobenil	0.01	Sebuthylazin	0.01
Fluazifop-P-butyl         0.01         Tetradifon         0.01           Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Fenarimol	0.01	Simazin	0.01
Hexazinon         0.01         Thiabendazol         0.01           Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Fenpropimorph	0.01	Terbuthylazin	0.01
Iprodion         0.01         Tolylfluanid         0.01           Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Fluazifop-P-butyl	0.01	Tetradifon	0.01
Lenacil         0.01         Triadimefon         0.01           Metalaxyl         0.01         Triadimenol         0.01           Metolachlor         0.01         Trifluralin         0.01	Hexazinon	0.01	Thiabendazol	0.01
Metalaxyl0.01Triadimenol0.01Metolachlor0.01Trifluralin0.01	Iprodion	0.01	Tolylfluanid	0.01
Metolachlor 0.01 Trifluralin 0.01	Lenacil	0.01	Triadimefon	0.01
	Metalaxyl	0.01	Triadimenol	0.01
Metribuzin 0.01 Vinclozolin 0.01	Metolachlor	0.01	Trifluralin	0.01
<u> </u>	Metribuzin	0.01	Vinclozolin	0.01

**Group ON/MS-2 (N-containing pesticides), Methodology GC/MSD (DFG S19)** 

Compound	LOQ	Compound	LOQ (mg/kg)
	(mg/kg)		
Aminocarb	0.01	Fenoxycarb	0.01
Benalaxyl	0.01	Flusilazol	0.01
Biphenyl	0.01	Furathiocarb	0.01
Bromacil	0.01	Hexaconazol	0.01
Buprofezin	0.01	Imazalil	0.01
Chloridazon	0.01	Methiocarb	0.01
Cyproconazol	0.01	Myclobutanil	0.01
Desmethyl-pirimicarb	0.01	Oxadixyl	0.01
Diflubenzuron	0.01	Penconazol	0.01
Dioxacarb	0.01	Promecarb	0.01
Diphenylamin	0.01	Pyrimethanil	0.01
Diuron	0.01	Tebuconazol	0.01
Ethofenprox	0.01	Teflubenzuron	0.01
Fenazaquin	0.01	Triflumizol	0.01

Group ON/MS 4 (N-containing pesticides), Methodology GC/MSD (DFG S19)

Compound	LOQ	Compound	LOQ (mg/kg)
	(mg/kg)		
Aclonifen	0.01	Iprovalicarb	0.01
Azoxystrobin	0.01	Isoprocarb	0.01
Bromuconazol	0.01	Kresoxim-methyl	0.01
Chlozolinate	0.01	Landrin	0.01
Climbazol	0.01	Metamitron	0.01
Clodinafop-propargyl	0.01	Metazachlor	0.01
Clomazone	0.01	Metconazol	0.01
Cycloat	0.01	Napropamid	0.01
Cyprodinil	0.01	Norflurazon	0.01
Diclobutrazol	0.01	Nuarimol	0.01
Diethofencarb	0.01	Paclobutrazol	0.01
Diflufenican	0.01	Prosulfocarb	0.01
Diniconazol	0.01	Pyridaben	0.01
Epoxiconazol	0.01	Pyrifenox	0.01
EPTC	0.01	Pyriproxyfen	0.01
Etoxazol	0.01	Quinoxyfen	0.01
Etaconazol	0.01	Tebutam	0.01
Fenbuconazol	0.01	Terbacil	0.01
Fludioxonil	0.01	Terbumenton	0.01
Fluotrimazol	0.01	Tetraconazol	0.01
Fluquinconazol	0.01	Uniconazol	0.01

Fipronil, Methodology GC/MSD-NCI (DFG S19)

Compound	LOQ (mg/kg)
Fipronil	0.004

Group LC-1 (Determination of selected pesticides), Methodology LC-MS/MS (DFG S19)

Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg)
Acetamiprid	0.01	Indoxacarb	0.01
Carbaryl	0.01	Lufenuron	0.01
Carbofuran	0.01	Metamitron	0.01
3-Hydroxycarbofuran	0.01	Methiocarb	0.01
Chloridazon	0.01	Methiocarbsulfon	0.01
Clofentezin	0.01	Methiocarbsulfoxid	0.01
Clothianidin	0.01	Methomyl	0.01
Cymoxanil	0.01	Methoxyfenozid	0.01
Desmedipham	0.01	Oxamyl	0.01
Difenoconazol	0.01	Phenmedipham	0.01
Diflubenzuron	0.01	Prochloraz	0.01
Dimethomorph	0.01	Tebufenozid	0.01
Ethofumesat	0.01	Tebufenpyrad	0.01
Fenhexamid	0.01	Teflubenzuron	0.01
Fenpyroximat	0.01	Tepraloxydim	0.01
Fluazinam	0.01	Thiacloprid	0.01
Flufenoxuron	0.01	Thiametoxam	0.01
Hexythiazox	0.01	Thiodicarb	0.01
Imidacloprid	0.01		

Group LC-2 (Determination of N-methyl Carbamates), Methodology LC-MS/MS (DFG S19)

Compound	LOQ (mg/kg)	Compound	LOQ (mg/kg)
Carbofuran (incl. 3- Hydroxycarbofuran)	0.01	Isoprocarb	0.01
Aldicarb (incl. Aldicarbsulfon,-sulfoxid)	0.01	Methiocarb (incl. Methiocarbsulfon, -sulfoxid)	0.01
Aminocarb	0.01	Methomyl (incl. Thiodicarb)	0.01
Bendiocarb	0.01	Oxamyl	0.01
Butocarboxim (incl. Butocarboximsulfon,- sulfoxid)	0.01	Phenmedipham	0.01
Carbaryl	0.01	Pirimicarb (incl. Desmethylpirimicarb)	0.01
Desmedipham	0.01	Promecarb	0.01
Dioxacarb	0.01	Propoxur	0.01
Ethiofencarb (incl. Ethiofencarbsulfon, - sulfoxid)	0.01	Thiofanox (incl. Thiofanoxsulfon, -sulfoxid)	0.01

### **Group QuEChERS**

Compound	LOQ (mg/kg)
Carnendazime	0.005
Thiophanate-methyl	0.005
Fosthiazate	0.01
Spiroxamine	0.01

**Group ETU** 

Compound	LOQ (mg/kg)
ETU	0.003
PTU	0.003

**Single Residue Methods** 

Compound	LOQ (mg/kg)
Compound	LOQ (llig/kg)
Inorganic Bromide	0.1
Chloride	5
Chlormequat	0.005
Deiquat	0.01
Fentin-acetat	0.003
Haloxyfop	0.003
Propinep (within Dithiocarbamates)	0.005
Maleic Hydrazide	0.1