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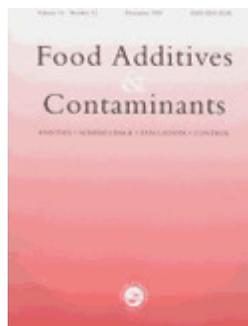
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Study on the effect of grain storage and processing on deltamethrin residues in post-harvest treated wheat with regard to baby food safety requirements

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1 2 3 1 **Abstract**

4 2 The effects of storage intervals and of milling procedures on dissipation of deltamethrin
5 3 residues in post-harvest treated wheat grain were studied with the aim of obtaining scientific
6 4 data on compliance of the processed products with the safety requirements concerning baby
7 5 foods. The insecticide formulation was applied on stored wheat at recommended rate of
8 6 active ingredient of 0.5 mg kg⁻¹ and at the higher rate of 4 mg kg⁻¹ providing the highest
9 7 protective effect. The loss of residues and their distribution in different fractions of the milled
10 8 grain were studied after various storage intervals, from 7 to 270 days after treatment. Eight
11 9 fractions - bran, semolina, 3 types of groats and 3 types of flour were collected after milling
12 10 of grain and analyzed for pesticide residues. The residues were determined by a GC method
13 11 characterized by limit of determination 0.005 mg kg⁻¹ - low enough for enforcement of the
14 12 maximum residue level of 0.01 mg kg⁻¹ established by European Commission Directive for
15 13 any pesticide in cereal-based foods. Deltamethrin applied post-harvest on wheat as a grain
16 14 protectant is characterised by low rate of degradation on the grain under practical storage
17 15 conditions. 180 days after treatment at an application rate 0.5 mg kg⁻¹ the residues were
18 16 between 0.03 and 0.2 mg kg⁻¹ in the various types of flour. 270 days after treatment at rate 4
19 17 mg kg⁻¹ the residues in flour were in the range 0.4 - 1.5 mg kg⁻¹.

20 18
21 19 **Keywords:** deltamethrin, wheat grain, post-harvest treatment, residues, processed products,
22 20 baby food

23 21 24 22 25 23 **Introduction**

26 24 Food safety is one of the most serious problems of the modern society. Contamination of
27 25 plant production with pesticide residues is a source of risk for human health. Obtaining a

1
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3 1 knowledge of the effects of processing on the fate of residues is one of the basic pre-
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5 2 conditions for refinement of pesticide risk assessment and especially of potential risk to
6
7 3 consumers of high vulnerability (Regulation (EC) No 396/2005). Special attention has to be
8
9 4 paid to the health protection of infants and young children as they represent the most
10
11 5 vulnerable group of population. Infants and young children have been shown to be very
12
13 6 sensitive to certain toxic effects (Matsumura F. 2004, National Research Council 1993).
14
15 7 Furthermore, babies and young children are exposed to relatively higher doses of toxicants
16
17 8 per unit body weight, compared to adults because of their proportionally higher consumption
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19 9 of food. Based on FDA data, children receive 3 - 5 times more cereals than adults per body
20
21 10 weight. For better protection of the health of infants and young children a general MRL of
22
23 11 0.01 mg kg⁻¹ for any individual pesticide in processed cereal-based foods and baby foods has
24
25 12 been established by EC (European Commission 2003^a, European Commission 2003^b).
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34 14 Post-harvest treatment for protection of raw grain against stored-product pests has been
35
36 15 reported as a source of contamination of cereals (JMPR 1991, JMPR 1992). A summary of
37
38 16 residue monitoring data sorted by frequency of incidents showed that grain protectants used
39
40 17 on cereals accounted for most of the positive findings (Hamilton et al. 1997). Studies on the
41
42 18 effect of storage on insecticides residues following post-harvest treatments have generally
43
44 19 shown that residues decline rather slowly (Holland et al. 1994). For refining of dietary intake
45
46 20 estimates of pesticide residues a generation of food processing studies data has been required.
47
48
49 21 Milling is one of the 10 processing methods identified as deserving main attention (World
50
51 22 Health Organization 1997). Studies on the fate of pesticide residues on grain subjected to
52
53 23 milling and processing showed that the bulk of the insecticides deposited on the grain
54
55 24 remained in the epidermis and was removed from the bran during milling. Most residues were
56
57 25 found in the outer portion of grains (Hajslova, 2000). The concentration on the bran was from
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60

1 ~~2 to 3.2 times the concentration on the whole wheat~~ (Desmarchelier 1979^a, Desmarchelier
2 1979^b). Bran/grain concentration factors in the range of 2 to 6 were reported in many studies
3 (JMPR 1988, JMPR 1989, JMPR 1991, JMPR 1992). Generally the concentration of
4 insecticide in flour was 10% or less of the concentration on the whole wheat.

5
6 Deltamethrin has been used as grain protectant for more than 30 years. The MRL of 1 mg kg⁻¹
7 is established for wheat grain intended for general nutritional uses and this value
8 accommodates post-harvest treatment of the commodity (European Commission 2002). The
9 MRL established by EC (European Commission 2003^a, European Commission 2003^b). for
10 residues in baby food is 100 times below the deltamethrin MRL for food of general
11 consumption. The enforcement of this very stringent limitation of residues in processed
12 cereal-based baby food imposes the necessity of careful tuning of all technological parameters
13 in order to select procedures that ensure baby food production in compliance with the safety
14 requirements from potentially contaminated raw materials.

15
16 For the determination of residues at ultra-trace levels considerable attention has to be paid to
17 the problems of assurance the high quality of analytical methods both in respect of
18 quantitative parameters (sensitivity, trueness, precision, and range) and of qualitative
19 assessment (identification and confirmation). Implementation of analytical methods capable
20 to determine the pesticide studied at or below 0.01 mg kg⁻¹ was mandatory in the study.

21
22 The study was aimed at assessment of the risk of baby food MRL violation due to utilization
23 of contaminated wheat grain for processing and production of baby food. The objectives of
24 this work were: to study the influence of the wheat grain storage intervals and of milling on
25 the decline and distribution of residues of deltamethrin applied post-harvest on wheat grain; to

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3 1 establish the residue balance in processed products; to provide scientific data on compliance
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5 2 of the processed post-harvest treated wheat grain products with the safety requirements
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7 3 concerning baby foods.
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15 **Experimental**

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17 6 All processes applied to the study for processing effects assessment were planned in a way to
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19 7 correspond as closely as possible to those that occur in actual practices.
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25 *Wheat treatment, sampling and processing.*

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27
28 10 High grain quality wheat (*Triticum aestivum*) cultivar “Momchil” was used in the experiments.
29
30 11 The initial grain moisture was $14.0 \pm 0.5\%$. The commercial insecticide formulation was Decis
31
32 12 2.5 EC containing 25 g.l^{-1} active ingredient (a.i). It was applied at two rates – the
33
34 13 recommended rate of 20 ml.t^{-1} and the rate assessed to give highest effect in stored grain
35
36 14 protection – 160 ml.t^{-1} (Obretenchev, D., personal communication). 40 kg wheat grain were
37
38 15 spread in about 1 cm thick layer and sprayed with 40 ml of working solution. The application
39
40 16 was accomplished by means of a hand-operated spraying device used in the practice. In order
41
42 17 to achieve uniform distribution of the pesticide the working solution was divided into three
43
44 18 portions. After uniform spraying of each portion the bulk of the grain was mixed by
45
46 19 quartering, then spread again and the spraying procedure was repeated consecutively with the
47
48 20 portions left. Finally the treated wheat grain was mixed thoroughly and kept in open
49
50 21 containers in a storehouse at conditions simulating the practical grain storage. Untreated grain
51
52 22 was stored under the same conditions as control variant. The room temperature was $20 \pm 2^{\circ}\text{C}$
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54 23 and the air moisture - $70 \pm 5\%$.
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3 1 A sample of 0.5 kg of treated grain was taken 1 hour after treatment, when the grain became
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5
6 2 dry, for determination of the initial concentrations of pesticide in the whole grain. The
7
8 3 following samplings were made after 7, 90, 180 and 270 days storage periods. 3 kg of the
9
10 4 treated grain and of untreated control were taken on particular sampling dates and submitted
11
12 5 to milling in laboratory mill, simulating the industrial processing.
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15 6

16
17 7 Milling was carried out in a fractionating laboratory mill Buhler model MLU 202 M/s
18
19 8 (Buhler, Uzwil, Switzerland). After milling the following fractions were collected: bran,
20
21 9 semolina, 3 fractions of groats, and 3 fractions of flour. These processed products fully
22
23 10 resemble the industrial processing products. The collected fractions as well as the whole grain
24
25 11 were subjected to analysis.
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29 12

30 31 13 *Chemicals*

32 33 14 *Pesticide standards.*

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35
36 15 Analytical standard of deltamethrin, purity 99.9% Pestanal was obtained from Sigma-Aldrich
37
38 16 Laborchemicalien GmbH (Seelze, Germany). Stock solution (1.0 mg.ml^{-1}) was prepared in
39
40 17 ethyl acetate and stored in a freezer at -23°C . Working standard solutions at concentration
41
42 18 rates of 10 ng.ml^{-1} to 500 ng.ml^{-1} were prepared by diluting of stock solution with ethyl
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44 19 acetate and stored at 4°C in the dark.
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49 50 21 *Solvents and reagents.*

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53 22 All organic solvents were of residue analysis grade and were obtained from Merck KGaA
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55 23 (Darmstadt, Germany). Double distilled water was used when needed in experiments. Solid
56
57 24 phase extraction (SPE) sorbent Bond Elute SAX 250 mg-PSA 250 mg was from Varian Inc.
58
59 25 (Lake Forest, CA 92630 USA).
60

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6 2 *Instrumentation.*
7
8 3 Hewlett-Packard gas chromatograph Model 5890 Series II equipped with ⁶³Ni electron
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10 4 capture detector (ECD) was used. Split-splitless injector operated in the splitless mode.
11
12 5 Capillary column AT™-5ms, (30 m × 0.25 mm × 0.25 μm) was obtained from Alltech
13
14 6 Associates, Inc. (Lokeren, Belgium).
15
16
17 7 Nitrogen of purity 99.9995 % was used for gas chromatography.
18
19
20 8 Vacuum manifold (Varian BV) was used for SPE.
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25 10 *Analytical method*
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28 12 *Extraction.* The weight of the samples taken of different products varied depending on the
29
30 13 expected levels of the residue concentrations. The sample: solvent ratios varied because of the
31
32 14 different swelling capacity of the matrices. The whole wheat grain laboratory samples were
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34 15 homogenized by mixing, analytical samples of 20 g were weighed, 40 ml ethyl acetate and 10
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36 16 g anhydrous sodium sulfate were added and samples were extracted by shaking for 30 min on
37
38 17 a shaking machine Labortechnik HS 501 (IKA Werke GmbH & Co. KG, Staufen, Germany).
39
40 18 Analytical samples of 20 g were weighed of bran and semolina, 10 g anhydrous sodium
41
42 19 sulfate and 100 ml of the solvent were added for extraction. 40 g analytical samples of flour
43
44 20 and groats were extracted with 80 ml of the solvent after adding of 10 g anhydrous sodium
45
46 21 sulfate. The raw extracts were filtered through paper filter, 5 ml aliquot of each extract was
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48 22 taken in a graduated tube and the volume of the extract was adjusted at 4 ml by evaporation
49
50 23 under gentle stream of air. 1 ml methanol was added to the extracts. The samples dissolved in
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52 24 the resulted 5 ml mixed solvent ethyl acetate-methanol (4:1) were submitted to further SPE
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54 25 cleanup and pre-concentration.
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3 1 *Sample clean-up.*
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5 2 The mixed mode SAX-PSA sorbent combines the hydrogen bonding and weak anion-
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7 3 exchange capability of primary/secondary amines of PSA sorbent with the strong anion-
8
9 4 exchange capability of quaternary amines of SAX sorbent. This SPE product possesses
10
11 5 powerful ability to remove polar interferences such as fatty acids, other organic acids, various
12
13 6 sugars. A pre-packed cartridge SAX-PSA was washed with 2 ml methanol and conditioned
14
15 7 with 2 ml ethyl acetate-methanol (4:1). The samples were passed through the sorbent at a rate
16
17 8 of 1 ml.min⁻¹. The eluate was collected in a graduated tube. After the passage of the sample
18
19 9 the elution of the pesticides continued with ethyl acetate-methanol (4:1). A total volume of 10
20
21 10 ml of the eluate was collected. 1 µl was injected in GC system and if the obtained detector
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23 11 response was out of the calibration range, the sample volume was adjusted by dilution or
24
25 12 evaporation under gentle stream of air in order to obtain analyte concentrations appropriate to
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27 13 quantitative determination.
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36 15 *Gas-chromatographic determination.*
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38 16 GC-ECD operating conditions: carrier gas nitrogen at flow rate 4 ml.min⁻¹; make up nitrogen
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40 17 30 ml.min⁻¹; injector temperature 270°C; detector temperature 320°C. Oven temperature
41
42 18 programme: 150°C, hold 1 min; rate 30°C/min to 290°C final temperature, hold 8 min.
43
44 19 Injection volume was 1 µl for all samples. Quantification was performed by using external
45
46 20 standards. The recovery studies were performed by using blank samples of untreated wheat
47
48 21 grain. The weights of blanks were the same as the weights of the analytical samples of treated
49
50 22 grains. The fortification was done by addition of calculated volumes of working standard
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52 23 solutions in order to obtain 3 concentration levels of the insecticide studied: at MRL (0.01 mg
53
54 24 kg⁻¹), below MRL (0.005 mg kg⁻¹) and ten times MRL (0.1 mg kg⁻¹). Five parallel samples
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56 25 were prepared for each fortification level. After waiting time of 1 h for evaporation of the
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1 ethyl acetate from standard solutions the fortified samples were analyzed according to the
2 method described above.

4 **Results and discussion**

5 *Method validation*

6 The method was validated for each matrix studied.

7 The ECD response for deltamethrin was linear in the range of 0.02 ng – 0.5 ng and the
8 correlation coefficient r^2 was 0.9897 (five-point calibration curve). The concentrations of the
9 analytes in the samples were calculated by using a single-level calibration method. This
10 approach is recommended in case the detector response is variable with time (Document
11 SANCO/10476/2003). In our studies it was confirmed that the single-level calibration gave
12 more accurate results than multi-level calibration when the analyte concentrations in the
13 samples were adjusted in an interval $\pm 10\%$ of the calibration level used.

14
15 The mean recoveries and the repeatability expressed as relative standard deviation (RSD),
16 determined at three fortification levels (number of samples $n=5$) are presented in table 1. The
17 limit of quantification (LOQ), determined as the lowest concentration of the analyte that yield
18 signal-to-noise ratio of 10:1 was established at 0.005 mg.kg^{-1} . The LOQ was specified at this
19 concentration level because it ensured compliance with the European Commission criteria for
20 quantitative residue methods (Document SANCO/10476/2003).

22 *Processing.*

23 The laboratory fractionating mill is a conventional device used for simulated fractionation that
24 corresponds closely to the fractionation under the real industrial process. The results of
25 residue analysis obtained from processing experiments carried out on stored wheat grain,

1 treated post harvest with 0.5 mg kg⁻¹ deltamethrin are presented in table 2 and of grain, treated
2 with 4 mg kg⁻¹ deltamethrin – in table 3. Deltamethrin residues were persistent under the
3 storage conditions studied. No decrease was observed of the average residue levels on the
4 whole grain and in the processed fractions after storage periods of 180 and 270 days. The
5 measured slight increase of deltamethrin residues after 180 days storage period in the higher
6 dose variant is within the estimated uncertainty of the method. This result confirms that no
7 measurable degradation of deltamethrin residues on stored wheat grain occurred during the
8 storage period. These findings correspond with the results of deltamethrin residues evaluation
9 by FAO (Food and Agriculture Organization 1983). The data reported by several countries
10 confirmed that deltamethrin was stable on stored wheat grain for more than a year. Some
11 increase of the concentrations on grain after 6 – 9 months of storage was also observed (Food
12 and Agriculture Organization 1983).

13
14 The distribution of deltamethrin residues in fractions from processed wheat is presented in
15 tables 2 and 3. The bulk of the insecticide deposited on the grain remained in the bran. The
16 concentrations of deltamethrin in the bran were from 3.3 to 4.7 times as high as those present
17 on the whole wheat grain. A considerable part of the insecticide was distributed in the
18 semolina fractions. The residues determined in semolina were 1.8 – 2.7 times as high as those
19 on the whole grain. Generally the residue levels in flour were 3 – 7 times lower than the
20 concentration on the whole grain. The reduction of residues in flour and groats is mostly due
21 to the removal of the insecticides with bran and partly with semolina fractions.

22
23 In order to determine the percentage loss of residues during processing the residual amounts
24 (mg) in the separate fractions were calculated by multiplication of the weight of each fraction
25 by the corresponding residue concentration. The balance of the residual amounts in all

1
2
3 1 fractions compared to the amount on whole grain before milling, determined in samples taken
4
5 2 after 180 days storage (table 4), shows that deltamethrin undergoes negligible loss of residues
6
7 3 (0.7 %) during milling. Taking into consideration that volatilization is one of the basic
8
9 4 processes acting on pesticide residues during grinding, screening etc., the negligible losses
10
11 5 could be explained by the very low vapor pressure of deltamethrin - $V.p.(25^{\circ}C)=1.25 \times 10^{-5}$
12
13 6 mPa (Tomlin 2000).
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22 8 **Conclusions**

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25 9 The analytical method used was sensitive enough for reliable determination of deltamethrin in
26
27 10 wheat at concentration 0.01 mg kg^{-1} . The storage time and milling of wheat grain, treated
28
29 11 post-harvest against stored-product pests with deltamethrin at recommended application rate,
30
31 12 are not effective enough to reduce the residues in milled products to levels at or below the
32
33 13 general MRL of 0.01 mg kg^{-1} established by EC for any individual pesticide in processed
34
35 14 cereal-based foods and baby foods. The bulk of the insecticide deposited on the grain is
36
37 15 removed with the bran and partly with the semolina fractions during milling but yet the flour
38
39 16 and groats fractions, used directly for preparing of baby foods, could contain deltamethrin in
40
41 17 quantities which do not comply with the infants and young children safety requirements. The
42
43 18 results of this study indicate that processed products (flour, groats) of wheat, treated post-
44
45 19 harvest with deltamethrin at the recommended rate are in compliance with the safety
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47 20 requirements for general population. The increase of the application rate beyond the
48
49 21 recommended rate should be avoided because it resulted in exceeding the MRL of 1 mg.kg^{-1}
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51 22 for general population uses.
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5

For Peer Review Only

Table 1.

| Fortification level, mg.kg^{-1} | Recovery \pm RSD, % |
|--|-----------------------|
| 0.005 | 97.0 ± 7.1 |
| 0.01 | 89.6 ± 16.6 |
| 0.1 | 97.6 ± 15.3 |

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Table 2.

| Matrix | Mean residue, $\text{mg.kg}^{-1} \pm \text{RSD}\%$ | | | |
|---------------------------------|--|-----------|-----------|-----------|
| | Days after treatment | | | |
| | 0 | 30 | 90 | 180 |
| Whole grain | 0.53±9.7 | 0.52±9.4 | 0.52±5.0 | 0.51±7.5 |
| Bran | | 2.4±5.2 | 2.5±5.3 | 2.3±5.3 |
| Semolina | | 1.2±0.5 | 1.2±6.7 | 1.1±3.3 |
| Groats 1 st fraction | | 0.15±5.6 | 0.10±11.5 | 0.16±10.5 |
| Groats 2 ^d fraction | | 0.08±7.4 | 0.09±9.7 | 0.09±3.0 |
| Groats 3 rd fraction | | 0.14±2.7 | 0.22±4.6 | 0.16±4.7 |
| Flour 1 st fraction | | 0.05±11.0 | 0.06±10.3 | 0.03±3.6 |
| Flour 2 ^d fraction | | 0.09±2.8 | 0.05±4.6 | 0.05±11.9 |
| Flour 3 rd fraction | | 0.16±13.8 | 0.07±11.4 | 0.23±3.3 |

Table 3.

| Matrix | Mean residue, $\text{mg}\cdot\text{kg}^{-1} \pm \text{RSD} \%$ | | | | |
|---------------------------------|--|----------|----------|-----------|----------|
| | Days after treatment | | | | |
| | 0 | 7 | 90 | 180 | 270 |
| Whole grain | 2.4±4.1 | 2.1±3.8 | 2.2±4.6 | 3.2±4.7 | 3.5±0.6 |
| Bran | | 9.9±0.7 | 9.0±9.4 | 10.5±4.7 | 13.4±7.9 |
| Semolina | | 3.9±1.9 | 4.8±1.5 | 8.7±5.7 | 5.3±4.7 |
| Groats 1 st fraction | | 0.22±2.6 | 0.42±4.7 | 0.41±13.2 | 0.19±3.7 |
| Groats 2 ^d fraction | | 0.13±2.2 | 0.41±3.9 | 0.40±5.6 | 0.13±0.6 |
| Groats 3 rd fraction | | 0.19±6.0 | 0.49±4.5 | 1.25±2.6 | 0.43±0.7 |
| Flour 1 st fraction | | 0.69±2.9 | 1.9±3.0 | 1.9±4.4 | 1.55±2.1 |
| Flour 2 ^d fraction | | 0.58±3.9 | 1.1±9.2 | 1.5±2.3 | 0.39±1.6 |
| Flour 3 rd fraction | | 0.77±8.8 | 1.4±5.9 | 1.6±0.3 | 0.45±6.3 |

Table 4

| Product | Deltamethrin distribution | |
|---------------------------------|---------------------------|-------------|
| | Amount (mean), mg | % |
| Bran | 1.2 | 75.0 |
| Semolina | 0.24 | 15.7 |
| Groats 1 st fraction | 0.03 | 1.9 |
| Groats 2 ^d fraction | 0.02 | 1.3 |
| Groats 3 rd fraction | 0.01 | 0.7 |
| Flour 1 st fraction | 0.04 | 2.6 |
| Flour 2 ^d fraction | 0.02 | 1.3 |
| Flour 3 rd fraction | 0.02 | 1.3 |
| Total | 1.52 | 99.3 |
| Whole grain | 1.53 | 100 |

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5 Captions to tables
6

7 Table 1. Mean recoveries and standard deviations of deltamethrin in wheat (n=5)

8
9 Table 2. Decline of deltamethrin residues in whole grain and processed products with
10 time after post-harvest treatment at range 0.5 mg/kg (n=3)
11

12 Table 3. Decline of deltamethrin residues in whole grain and processed products with
13 time after post-harvest treatment at range 4 mg/kg (n=3)
14

15
16 Table 4 Residue balance of deltamethrin in processed products after storage intervals
17 of 180 days (application rate 0.5 mg/kg)
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Captions to tables

Table 1. Mean recoveries and standard deviations of deltamethrin in whole grain (n = 5)

Table 2. Decline of deltamethrin residues in whole grain and processed products with time after post-harvest treatment at range 0.5 mg.kg⁻¹ (n = 3)

Table 3. Decline of deltamethrin residues in whole grain and processed products with time after post-harvest treatment at range 4 mg.kg⁻¹ (n = 3)

Table 4 Residue balance of deltamethrin in processed products after storage intervals of 180 days (application rate 0.5 mg.kg⁻¹)

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