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Effects of processing on pesticide residues in peaches intended for baby food

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1 **EFFECTS OF PROCESSING ON PESTICIDE RESIDUES IN**
2 **PEACHES INTENDED FOR BABY FOOD**

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For Peer Review Only

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4 **Abstract**
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6 Peaches containing incurred residues of chlorpyrifos-methyl, fenitrothion, procymidone
7 and vinclozolin were used for simulated industrial processing to manufacture baby food
8 puree. The residues were determined in raw material, in intermediate products at crucial
9 steps of the processing procedures and in final products. The determination of the residues
10 was performed by an analytical method distinguished by high sensitivity based on acetone
11 extraction, 2 steps clean-up/pre-concentration on graphitized carbon and SAX/PSA
12 sorbent and GC-ECD. The results of the study were interpreted with respect to
13 enforcement of the stringent Maximum Residue Limit of 0.01 mg.kg⁻¹ established by
14 European Commission (EC) for any pesticide in baby food. Peeling was identified as the
15 most effective procedure resulting in reduction of residues. Thermal treatment
16 (concentration and sterilisation) contributed to substantial reduction of organophosphate
17 (chlorpyrifos-methyl, fenitrothion) residues, but procymidone and vinclozolin residue
18 levels were increased by peach puree thermal concentration.
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16 **Key words:** pesticide residues, chlorpyrifos-methyl, fenitrothion, procymidone,
17 vinclozolin, peaches, processing, baby food

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2 Introduction

3 The risk assessment of dietary intake of pesticides is important for ensuring safe food
4 production (Lentza-Rizos 2001). Special attention should be given to the health protection
5 of babies and small children, as they are the most vulnerable group of population
6 (Matsumura 2004, Dencker et al. 1998). In order to protect the health of infants and small
7 children in the European Union (EU), very strict limit has been introduced by the
8 European Commission (EC) for any pesticide potentially occurring in baby food.
9 Directives 2003/13/EC and 2003/14/EC established a general maximum residue limit (EU
10 MRL) of 0.01 mg.kg⁻¹ for any individual pesticide in processed cereal-based foods and
11 baby foods (European Commission 2003 a, b).

12
13 Residues of pesticides in food are influenced by storage and processing that occurs
14 between harvesting of raw commodities and consumption of prepared foodstuffs (Holland
15 et al. 1994). Procedures used in food processing have been shown to have considerable
16 effects on residue levels (Holland et al. 1994, Scattenberg et al. 1996). In general
17 significant decrease in residues occurs (Scattenberg et al. 1996). However concentration
18 of residues in some food fractions has been occasionally observed (Hajslova 2000). The
19 extent and direction of the pesticide changes during processing depend on physico-
20 chemical properties of the chemicals, type and conditions of the processes and nature of
21 the crops (Pappas et al. 2003, Elkins 1989).

22
23 The insecticides chlorpyrifos-methyl, fenitrothion and the fungicides procymidone,
24 vinclozolin were selected to be included in the study taking into consideration the
25 statistical evaluation of the quantities applied in practice and especially their use on

1 peaches. The EU MRLs established for peaches intended for general nutritional uses are 2
3 mg/kg for procymidone, 0.5 mg/kg for chlorpyrifos-methyl and fenitrothion and 0.05
4 mg/kg for vinclozolin (European Commission 2002). The existence of international
5 MRLs indicates that the corresponding pesticides are authorised to be used on particular
6 crops and that detectable residues of the order of magnitude of the MRL are left at
7 harvest. Pesticides with international MRLs of the order of 0.01 mg.kg^{-1} were not
8 included in the study with the exception of pesticides of high frequency of occurrence of
9 samples with residues exceeding MRLs (monitoring studies).

10 The enforcement of the very stringent Directive on residues in baby food makes it
11 necessary to understand the behaviour of pesticides during processing. Optimisation of all
12 technological parameters would allow final products to be prepared in compliance with
13 baby food safety requirements after processing of potentially contaminated raw materials.

14
15 The aim of this work was firstly to study the effect of fruit processing procedures on
16 behaviour of chlorpyrifos-methyl, fenitrothion, procymidone and vinclozolin residues in
17 peaches intended for production of baby food puree. Secondly, the aim was to establish
18 the processing factors for intermediate and final products of simulated industrial and
19 household processing of peaches, treated with the selected pesticides. Thirdly we aimed
20 to contribute to refinement of the plant protection technologies and processing
21 technologies in order to guarantee production of baby food in compliance with the
22 particular safety requirements.

23
24 The MRL for residues established in baby food is 10 -100 times below the MRLs for food
25 of general consumption. This very low limit necessitates considerable attention to be paid

1 to the problems of assurance the high quality of analytical methods both in respect of
2 quantitative parameters (sensitivity, trueness, precision, and range) and of qualitative
3 assessment (identification and confirmation). Implementation of analytical methods
4 capable of determining the pesticide residues at ultra-trace levels (at or below 0.01 mg.kg^{-1}
5 ¹) was mandatory in the study.

7 **Materials and methods**

8 **Chemicals and apparatus.** Acetone, ethyl acetate and methanol (gas chromatography
9 grade) were obtained from Merck KGaA (Darmstadt, Germany). Solid phase extraction
10 (SPE) sorbents: graphitized carbon ENVI-Carb 120/400 mesh, surface area $100 \text{ m}^2.\text{g}^{-1}$,
11 was obtained from Supelco (Bellefonte, PA, USA); Bond Elute SAX 250 mg-PSA 250
12 mg was from Varian Inc. (Lake Forest, CA 92630 USA). Certified analytical standards of
13 chlorpyrifos-methyl, fenitrothion, procymidone and vinclozolin were obtained from Dr.
14 Ehrenstorfer (Augsburg, Germany). Stock solutions (1.0 mg.ml^{-1}) of each pesticide were
15 prepared in ethyl acetate and stored in a freezer. Working standard solutions at
16 concentration rates of 5 ng.ml^{-1} - 500 ng.ml^{-1} were prepared by diluting of stock solutions
17 with ethyl acetate and stored at 4°C in dark. Double distilled water was used when needed
18 in experiments. Filter paper "blue spot 391", fine-pored, was obtained from Sartorius AG,
19 Goettingen, Germany.

20
21 A Hewlett-Packard gas chromatograph Model 5890 Series II equipped with ^{63}Ni electron
22 capture detector (ECD) was used. Split-splitless injector operated in the splitless mode.
23 Capillary column ATTM-5ms, ($30 \text{ m} \times 0.25 \text{ mm} \times 0.25 \mu\text{m}$) was obtained from Alltech
24 Associates, Inc. (Lokeren, Belgium). The purity of nitrogen used was greater than 99.999
25 % (SIAD, Bulgaria). Vacuum manifold (Varian Inc) was used for SPE.

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6 2 **Field treatment and sampling.** The field treatments of peach trees cultivar Fayette with
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9 3 the commercial formulations Reldan 50 EC (chlorpyrifos-methyl 500 g.l⁻¹ active
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11 4 ingredient (a.i.) and Sumilex 50 WP (procymidone 500 g.kg⁻¹ a.i.) were carried out in the
12
13 5 orchard belonging to the experimental station on peaches near the town of Sliven on 22
14
15 6 August 2003. The field treatments with the commercial formulations Agria 1050
16
17 7 (fenitrothion 500 g.l⁻¹a.i.) and Ronilan (vinclozolin 500 g.kg⁻¹ a.i.) were carried out in the
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19 8 orchard of the experimental station near the town of Petrich on 10 September 2004.
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25 10 All pesticides were applied at the recommended rate of 0.15% a.i. by using common
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27 11 orchard spraying equipment. Each pesticide working solution was applied on four trees
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29 12 and three trees received no treatment (control trees). Samples were collected 3 days after
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31 13 treatment. The aim was to obtain fruits containing incurred residues at concentration high
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33 14 enough to be traced in all intermediate products and in the final products of the simulated
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35 15 industrial processing to baby food puree. 40 kg of peaches were collected of each variant
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37 16 of treatment and of the untreated control trees. The sampling was carried out according to
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39 17 the recommendations of the EC for residue analysis sampling (European Commission
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41 18 2002).
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50 20 **Processing procedures.** All processes applied to the study for processing effects
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52 21 assessment were planned in a way to correspond as closely as possible to those that occur
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54 22 in actual practice. The field samples of each pesticide/peach combination were put into
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56 23 polyethylene bags and immediately transported to the laboratory. The following sets of
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58 24 samples of each type were prepared for processing and pesticide residues analysis:
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60

- 25 • Fruits, without washing

- 1 • Fruits, to be submitted to washing with tap water
- 2 • Fruits, to be submitted to mechanical peeling after washing
- 3 • Fruits, to be submitted to chemical peeling after washing

4 Approximately 3 kg of whole fruits were taken of each type. The stones were removed
5 from the fruit selected to be analysed without washing. The weights of the edible part and
6 of the stones were taken. The samples selected to be analysed after washing were
7 intensively washed with tap water and processed as described above. The washed fruits
8 were divided into two sub-samples and submitted to different types of peeling:
9 mechanical, typical for household processing and chemical, applied mostly in industrial
10 processing. The weights of the edible part of the fruits, stones and peels of mechanically
11 peeled fruits were taken. The peel of mechanically peeled fruit were weight separately.
12 The edible parts of the fruit were blended and stored in plastic bags in a freezer at (-23°C)
13 for simulated industrial processing and analyses.

14
15 The processing procedures were carried out under the following conditions: washing with
16 tap water at $16^{\circ}\text{C} \pm 2^{\circ}\text{C}$; chemical peeling with 4% NaOH solution at 90°C , then after
17 removing the peel - 2% citric acid solution and washing with water; concentration by
18 boiling under open conditions at $90^{\circ}\text{C} \pm 2^{\circ}$ to $24\% \pm 1.7\%$ dry content; thermal
19 sterilisation in closed jars at $90^{\circ}\text{C} \pm 2^{\circ}$ for 25 min.

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21 At each crucial step of the processing procedure samples were taken and submitted to
22 residue analysis. All procedure steps and corresponding samples taken for analysis were
23 given consecutive numbers as presented in figure 1.

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60**Analytical method.**

Extraction: Analytical samples (10 g) of the thoroughly homogenised material were weighed and taken at the selected steps of the processing procedure. Acetone (20 ml) was added and samples were extracted by mechanical shaking for 30 min. Extracts were filtered, residues washed by shaking with 2 x 20 ml acetone, and filtrates combined and homogenised. A half of the volume of each mixed filtrate was taken for further SPE clean-up and pre-concentration.

Sample clean-up. The aliquots of the acetone extracts (approximately 25 ml) were diluted with doubly-distilled water (200 ml) and the diluted extracts were submitted to two step SPE clean-up/preconcentration. The first step of SPE was carried out by using polypropylene cartridges manually packed with 400 mg ENVI-Carb, conditioned with 10 ml ethyl acetate: methanol (8:2) followed by 5 ml methanol and washed with 10 ml water. The sorbent was never allowed to dry during the conditioning and sample loading steps. The samples were forced to pass through the sorbent under vacuum at rate of 10 ml.min⁻¹. After the passage of the sample, the sorbent was washed with 0.4 ml methanol and dried under vacuum for 30 min. The pesticides were eluted with 6 ml ethyl acetate-methanol (8:2) at rate of 1 ml/min and collected in 10 ml graduated tubes. The eluates were submitted to the second clean-up step by using SAX-PSA Bond Elut cartridges. The sorbent was washed with 2 ml methanol and conditioned with 2 ml ethyl acetate-methanol (8:2). The samples were passed through the cartridge at rate of approximately 1 ml.min⁻¹. After the passage of the sample, the elution of the analytes was completed by washing twice with 1 ml ethyl acetate-methanol (8:2). Combined eluates were collected in 10 ml graduated tubes. The initial volumes of the samples were adjusted at 5 ml (1g matrix.ml⁻¹) by evaporation under gentle stream of nitrogen. Samples containing high-level residues

1 (unpeeled fruits, peels) were diluted to bring them within the calibrated range. Samples
2 containing low-level residues (peeled fruits, puree of peeled fruits) were concentrated to
3 bring the residue levels at or over the lowest calibrated level.

4 *Chromatographic conditions.* GC-ECD operating conditions: carrier gas nitrogen, at flow
5 rate 2.5 ml.min⁻¹; make up nitrogen 30 ml.min⁻¹; injector temperature 270°C, splitless
6 time 1.5 min; detector temperature 300°C. Oven temperature program: 100°C, hold 1 min;
7 rate 20°C/min to 260°C final temperature, hold 3 min. Injection volume was 1 µl for all
8 samples. Quantification was performed by using external standards. Matrix-matched
9 calibration standards were used for all quantifications.

10 *Preparation of matrix-matched calibration standards.* Blank extracts of each type of
11 samples were submitted to SPE clean up as described above. Series of standard solutions
12 were prepared by adding 0.1 ml of pesticide standards of desired concentration to cleaned
13 extracts aliquot to 5g blank matrix and the volumes were adjusted to 5 ml. In cases when
14 final sample extracts were diluted or concentrated the matrix extracts in calibration
15 standards were adjusted in order to ensure similar matrix concentration in sample and
16 standard solutions.

18 **Results and Discussion**

19 **Method validation**

20 The method was validated for peaches. The linearity of ECD response was determined in
21 the range of 0.005 ng – 0.05 ng. The correlation coefficients r^2 of the four pesticides
22 studied were higher than 0.98 (five-point calibration curve). Concentrations of the
23 analytes in the samples were calculated by using single-level calibration method. This
24 approach is recommended in case the detector response is variable with time (Document
25 SANCO/10476/2003). In our studies it was confirmed that single-level calibration gave

1 more accurate results than multi-level calibration when the analytes concentrations in
2 samples were adjusted in an interval $\pm 10\%$ of the calibration level used.

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4 The mean recoveries and the repeatability expressed as standard deviation (SD),
5 determined at three fortification levels (number of samples $n=5$) are presented in table 1.

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7 [Table 1 here]

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9 The lowest calibrated levels were adopted at 0.005 mg/kg for chlorpyrifos-methyl,
10 procymidone and vinclozolin and at 0.01 mg/kg for fenitrothion. The limits of
11 quantification (LOQ) were specified at these concentration levels because it ensured
12 compliance with European Commission criteria for quantitative residue methods
13 (Document SANCO/10476/2003). The LOQ values of the compounds confirmed that the
14 method was suitable for analyses of these residues in baby food for compliance with the
15 EU MRL. The combination of graphitized carbon (Envi-carb) and quaternary
16 amine/primary-secondary amine (SAX-PSA) results in a clean-up that removes a wide
17 spectrum of matrix co-extractives. Graphitized carbon in reversed-phase mode effectively
18 removes pigments and sterols. Quaternary amine/primary-secondary amines are effective
19 in retention of fatty acids, organic acids and some sugars. The applied two-step clean-up
20 procedure by using sorbents of different mechanisms of action helped to reduce the matrix
21 enhancement effect and avoid contamination of the GC system. It resulted in a decrease of
22 the base line noise and increase of the sensitivity expressed as signal-to-noise ratio.
23 Chromatograms of pure standards, blank sample and blank sample fortified for
24 determination of recovery, presented in figure 2, show the efficiency of the clean-up
25 procedure applied.

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11 **Processing studies.** Peeling, juicing and thermal processing are identified as processing
12 procedures deserving attention with respect to their effect on pesticide residues (Holland
13 et al. 1994, Hajslova 2000, WHO Guidelines for predicting dietary intake of pesticide
14 residues 1997). The effect of each of these processes on residues in peach puree is
15 dependent on the physico-chemical properties of the pesticides applied, on their
16 mechanism of action, processing conditions etc. The residue concentrations of the
17 pesticides in intermediate products at crucial steps of the processing procedure and in
18 final products are presented in table 2.

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15 The extent and direction of the processing effects are expressed by Processing Factors (P)
16 defined as a ratio between residues in processed commodity (PC) and raw commodity
17 (RC). Values of $P < 1$ indicate that residues undergo processes of dilution/degradation and
18 values of $P > 1$ – processes of concentration (Joint Meeting on Pesticide Residues 2000).
19 Processing factors calculated for the individual procedures and for the entire processing
20 are presented in table 3.

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22 [Table 3 here].

24 The key factors for the effectiveness of washing in reduction of residue concentration are
25 the age of the residues and the mode of action (systemic or non-systemic). Sampling was

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4 1 carried out 3 days after treatment in order to obtain residues incurred into cuticular waxes
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6 2 or deeper for closely simulating the processes in actual practice. The results presented in
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9 3 table 3 show that the cold water washing is a procedure that has limited effectiveness in
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11 4 reduction of residues and the reduction does not correlate with solubility and polarity of
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13 5 the compounds. This was also observed by Krol et al. (2000). Mechanical peeling, typical
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15 6 for household processing and chemical peeling, applied mostly in industrial processing,
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17 7 are the procedures which make a substantial contribution to reduction of residue levels in
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19 8 the fruit flesh. The bulk of the residues are removed with the peel. Residues of systemic
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21 9 pesticides penetrate into the plant tissues. Comparatively less of the systemic pesticide
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23 10 residues are therefore removed with the peel. The relatively higher value of P_2 calculated
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25 11 after mechanical peeling (table 3) for the systemic fungicide procymidone, compared to
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27 12 the other non-systemic compounds supports this concept. Chemical peeling results in the
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29 13 same relative efficiency in residue reduction.
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37 15 Thermal processing (cooking, sterilisation) may have highly variable effects depending on
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39 16 conditions (time, temperature, degree of moisture loss, whether the system is opened or
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41 17 closed) (Holland 1994) and on the properties of the compounds. Under the conditions of
42
43 18 the boiling process, loss of the phosphorous compounds chlorpyrifos-methyl and
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45 19 fenitrothion occurred. Despite the loss of moisture the residue concentrations of both
46
47 20 pesticides in concentrated puree were reduced significantly (P_4 values calculated for the
48
49 21 step of concentration were 0.8 and 0.6 respectively). The effect of cooking temperature
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51 22 was the main reason for accelerating the loss of organophosphates, as these compounds
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53 23 are susceptible to volatilisation and hydrolysis under such conditions.
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4 1 The concentration procedure resulted in a substantial increase of procymidone and
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6 2 vinclozolin residues (P_4 values calculated for the step of concentration were 2.3 and 4.5
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8 3 respectively). Procymidone, derivative of dicarboximide and vinclozolin, an oxazolidine
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10 4 derivative, are characterised by relatively high thermal stability (Tomlin 2000).
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16 6 The process of sterilisation in hermetically closed jars did not further influence the residue
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18 7 levels of the pesticides studied. The reduction of residue concentrations resulting from the
19
20 8 whole processing procedure of peeled fruits, calculated as final P value is significant,
21
22 9 especially for chlorpyrifos-methyl and fenitrothion. The reduction of residues of these
23
24 10 compounds in the final products was of the order of more than a hundred-fold (table 2).
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26 11 The residues of procymidone and vinclozolin in the final products obtained through the
27
28 12 processing of peeled fruit were reduced about ten times.
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35 14 Processing of unpeeled fruit is substantially less effective in reducing the residue levels in
36
37 15 final products. While the residues of organophosphates still undergo reductions of 2.5 to 5
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39 16 times, vinclozolin residue levels in peach puree produced from unpeeled fruit does not
40
41 17 change. The systemic fungicide procymidone is concentrated under this processing
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43 18 procedure ($P_{\text{final, (unpeeled fruit)}}$ is 1.3).
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49 20 All EC Directives concerning baby food safety stipulate that the exposure of infants and
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51 21 children to food contaminants is of particular concern because of their possible increased
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53 22 susceptibility for adverse effects. Growing and developing organisms have particular
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55 23 sensitivity to toxic compounds (Dencker et al. 1998, National Research Council 1993).
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57 24 The EC Directives on infant formulas and baby foods state that these products shall not
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4 1 contain any substance in such quantity as to endanger the health of infants and young
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6 2 children.
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11 4 As discussed above, detectable residues of the order of magnitude of the MRL may be
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13 5 present in the particular crop at harvest. Procymidone residues of the order of magnitude
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15 6 of 2 mg/kg could be expected in individual peach fruit. A compliance with the safety
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17 7 requirements for general population of processed product (peach puree) may be expected
18
19 8 only in case when peeling procedure was carried out of the fruits treated with
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21 9 procymidone. The same is valid for peaches treated with vinclozolin. Processing
22
23 10 procedures for pureeing are not effective enough to reduce the residues in the final
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25 11 product to levels at/below the general MRL of 0.01 mg.kg⁻¹ established by the EC for any
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27 12 individual pesticides in processed baby food. The results of the presented work indicate
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29 13 that the processed foods based on peaches treated with procymidone and vinclozolin are
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31 14 not suitable for feeding to infants and small children.
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52 16 The residues of chlorpyrifos-methyl and fenitrothion are significantly reduced during the
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54 17 pureeing process. They are suitable for treatment of peaches, intended for production of
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56 18 baby food. Peeling of fruits is recommend in all cases where the final product is intended
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58 19 for baby food.
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21 **Acknowledgement**

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1 Table 1. Recoveries and standard deviations of fortified pesticides from peaches versus
2 matrix-matched standard calibration (n=5)
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Fortification level, mg/kg	Mean recovery±SD, %			
	Chlorpyriphos-methyl	Fenitrothion	Procymidone	Vinclozolin
0.005	105±5.0	-	96.7±7.6	89.1±6.4
0.01	99.2±4.5	98.2±13.9	98.7±8.3	72.3±6.8
0.02	90.4±6.9	104±8.5	96.9±5.5	71.7±1.7

Table 2. Residues at crucial steps of simulated industrial processing of peaches treated with pesticides (mean of 2 samples)

Products	Mean residues, mg/kg			
	Chlorpyrifos-methyl	Fenitrothion	Procymidone	Vinclozolin
Whole, unwashed, unpeeled fruit (1)	1.1	2.7	4.5	n.d.
Whole, washed, unpeeled fruit (2)	0.9	1.6	2.3	0.8
Puree of washed, mechanically peeled fruit (6)	0.02	0.08	0.4	0.07
Peels of washed, mechanically peeled fruit (7)	10.3	12.8	13.8	6.4
Puree of washed, unpeeled fruit (3)	0.6	n.d.	1.5	n.d.
Puree of washed, unpeeled fruit, concentrated by boiling (4)	0.2	1.1	2.9	1.0
Puree of washed, unpeeled fruit, concentrated, thermally sterilized (5)	0.2	1.0	3.1	0.8
Puree of washed, chemically peeled fruit (8)	0.009	0.09	0.1	0.02
Puree of chemically peeled fruit, concentrated by boiling (9)	0.007	0.05	0.3	0.09
Puree of chemically peeled fruit, concentrated, thermally sterilized (10)	0.007	0.03	0.3	0.08

n.d. – no data

Table 3. Processing factors of intermediate and final products of peach-based baby food puree

Step	Processing Factors (P)*			
	Chlorpyrifos-methyl	Fenitrothion	Procymidone	Vinclozolin
P ₁ - Washing	0.8	0.6	0.5	
P ₂ - Mechanical peeling	0.02	0.05	0.2	0.09
P ₃ - Chemical peeling	0.01	0.06	0.06	0.02
P ₄ - Concentration	0.8	0.6	2,3	4.5
P ₅ - Sterilization	1.0	1.0	1.0	0.9
P _{final} (chemical peeling)	0.007	0.002	0.1	0.1
P _{final} (unpeeled fruit)	0.2	0.4	1.3	1.0

*Processing factors are calculated as ratio between the residues (R) in samples, taken at consecutive steps of processing shown in figure 1:

$$P_1 = R_{(2)} / R_{(1)}$$

$$P_2 = R_{(6)} / R_{(2)}$$

$$P_3 = R_{(8)} / R_{(2)}$$

$$P_4 = R_{(9)} / R_{(8)}$$

$$P_5 = R_{(10)} / R_{(9)}$$

$$P_{\text{final (chemical peeling)}} = R_{(10)} / R_{(2)}$$

$$P_{\text{final (unpeeled fruit)}} = R_{(5)} / R_{(2)}$$

Figure 1.

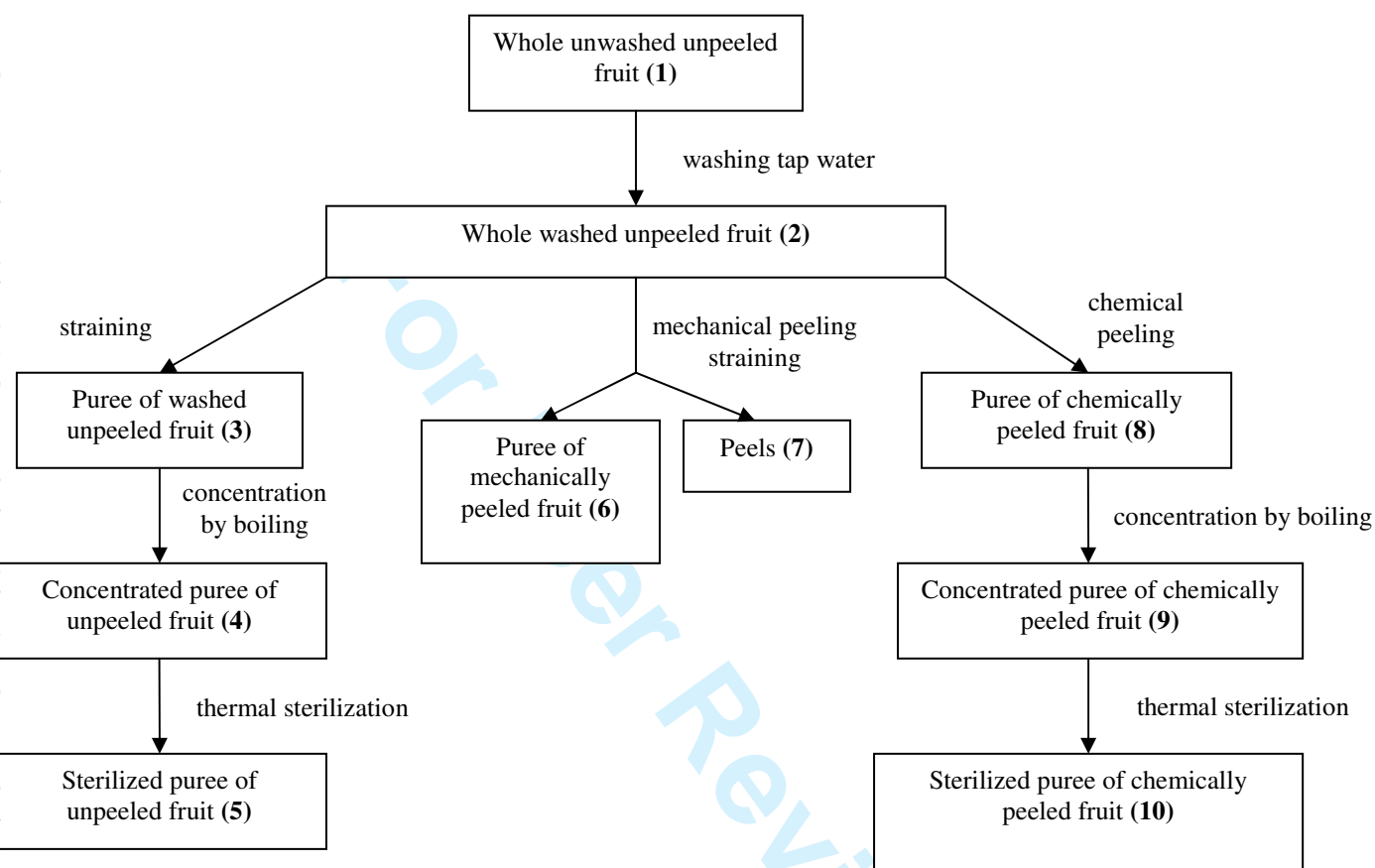
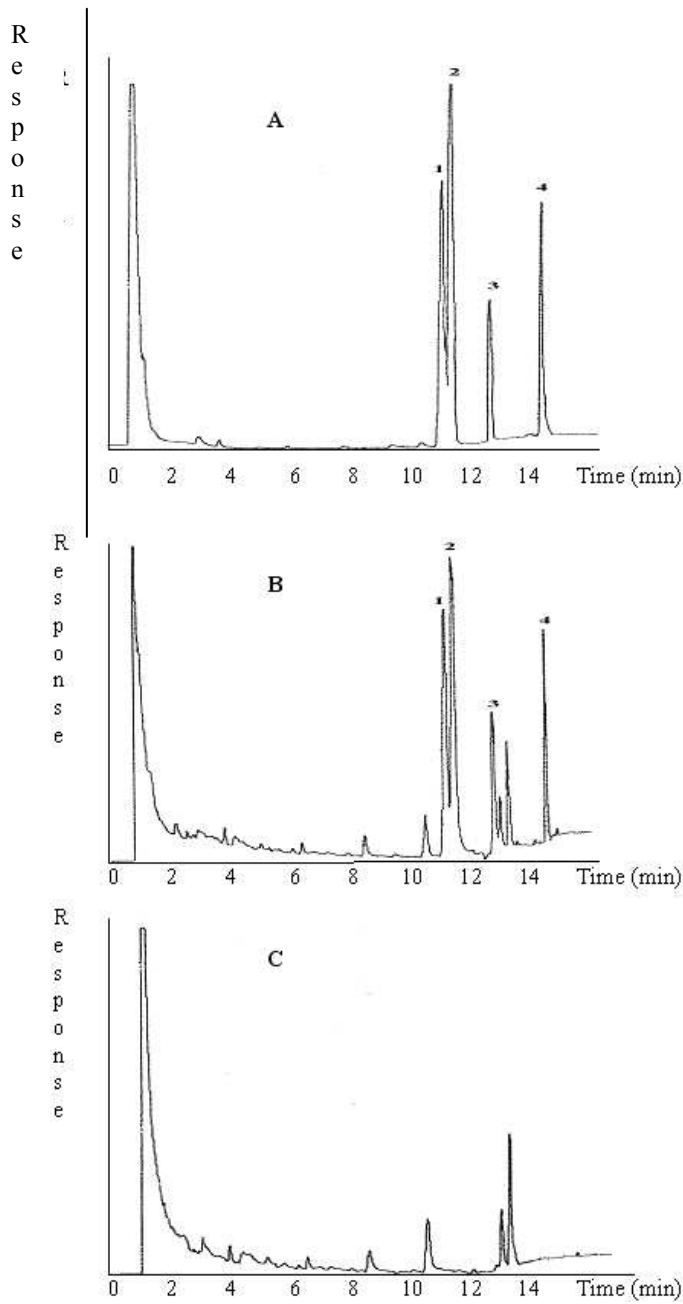


Figure 2



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Figure captions

Figure 1. Crucial steps of peach processing and samples taken for analysis.

Figure 2. Chromatograms of mixed standard solution (A) at concentration 0.1 µg/ml, extract of blank sample of peaches (B) subjected to the entire analytical procedure (matrix concentration 1g/ml), recovery sample of peaches fortified at 0.1 mg/kg (matrix concentration 1g/ml). Peak No: 1. chlorpiriphos-methyl, 2. vinclozolin, 3 fenitrothion, 4. procymidone.

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