PLANT PROTECTION PRODUCT RESIDUES IN AGRICULTURAL PRODUCTS OF SLOVENE ORIGIN FOUND IN 2008

OSTANKI FITOFARMACEVTSKIH SREDSTEV VKMETIJSKIH PRIDELKIH SLOVENSKEGA POREKLA V LETU 2008

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ABSTRACT

In the year 2008, 166 apple, bean, carrot, cucumber, lettuce, pear, potato and spinach samples from Slovene producers were analysed for plant protection product residues. The samples were analysed for the presence of 158 different active compounds using three analytical methods. In two samples (1.2%) exceeded maximum residue levels (MRLs) were determined which is better than the results of the monitoring of pesticide residues in the products of plant origin in the 27 European Union, Member States (EU MS) and 2 European Free Trade Association (EFTA) States: Norway and Iceland in 2008 (2.2%). The most frequently found active substance in agricultural products was dithiocarbamates. Products which contained 4 or more active substances per sample were apples and pears.

Keywords: GC/MS, LC/MS/MS, plant protection products, plant protection product residues, monitoring

IZVLEČEK

V letu 2008 smo na ostanke fitofarmacevtskih sredstev analizirali 166 vzorcev jabolk, fižola, korenja, kumar, solate, hrušk, krompirja in špinače slovenskih tržnih pridelovalcev. Vse vzorce smo analizirali s tremi analitskimi metodami na prisotnost 158 različnih aktivnih spojin. V dveh vzorcih (1,2%) smo določili presežene maksimalno dovoljene količine ostankov (MRL), kar je boljše kot rezultati monitoringa ostankov pesticidov v rastlinskih proizvodih v 27 državah članicah Evropske skupnosti in 2 državah iz Evropskega združenja za prosto trgovino (EFTA): Norveški in Islandiji v letu 2008 (2,2 %). Najpogosteje najdena aktivna snov v kmetijskih pridelkih so bili ditiokarbamati. Kmetijska proizvoda, ki sta vsebovala 4 ali več aktivnih snovi na vzorec sta bila jabolka in hruške.

Ključne besede: GC/MS, LC/MS/MS, fitofarmacevtska sredstva, ostanki fitofarmacevtskih sredstev, monitoring

RAZŠIRJENI IZVLEČEK

V letu 2008 smo na Kmetijskem inštitutu Slovenije analizirali 166 vzorcev jabolk, fižola, korenja, kumar, solate, hrušk, krompirja in špinače slovenskih tržnih pridelovalcev. Naključno vzorčenje je potekalo na osmih pridelovalnih območjih: Celje, Koper, Kranj, Nova Gorica, Novo mesto, Murska Sobota, Maribor in Ljubljana. Vzorčenje so opravili kmetijski inšpektorji. Vzorce so odvzeli na polju ali v skladiščih, po poteku karence. Seznam odvzetih vzorcev je prikazan v tabeli 1.

Od 166 analiziranih vzorcev 121 vzorcev (72,9%) ni vsebovalo ostankov, 43 vzorcev (25,9%) je vsebovalo ostanke nižje ali enake MRL in 2 vzorca (1,2%) sta vsebovala ostanke nad MRL.

Analizirali smo 38 vzorcev jabolk: ostankov, ki bi presegali MRL nismo določili, 27 vzorcev (71,1 %) je vsebovalo ostanke pod MRL, v 11 vzorcih (28,9 %) ostankov nismo določili. Analizirali smo 8 vzorcev fižola: ostankov nismo določili v nobenem vzorcu.

Analizirali smo 17 vzorcev korenja: ostankov, ki bi presegali MRL nismo določili, 2 vzorca (11,8 %) sta vsebovala ostanke pod MRL, v 15 vzorcih (88,2 %) ostankov nismo določili.

Analizirali smo 20 vzorcev kumar: ostankov, ki bi presegali MRL nismo določili, 2 vzorca (10,0 %) sta vsebovala ostanke pod MRL, v 18 vzorcih (90,0 %) ostankov nismo določili.

Analizirali smo 24 vzorcev solate: 1 vzorec (4,2 %) je presegal MRL, 3 vzorci (12,5 %) so vsebovali ostanke pod MRL, v 20 vzorcih (83,3 %) ostankov nismo določili.

Analizirali smo 21 vzorcev hrušk: ostankov, ki bi presegali MRL nismo določili, 9 vzorcev (42,9 %) je vsebovalo ostanke pod MRL, v 12 vzorcih (57,1 %) ostankov nismo določili.

Analizirali smo 32 vzorcev krompirja: ostankov nismo določili v nobenem vzorcu.

Analizirali smo 6 vzorcev špinače: 1 vzorec (16,7 %) je presegal MRL, v preostalih 5 vzorcih (83,3 %) ostankov nismo določili. Rezultati so prikazani na sliki 1.

V vzorcih jabolk, korenja, kumar, solate, hrušk in špinače smo določili naslednje aktivne spojine: klofentezin, cipermetrin, fenazakvin, pendimetalin, pirimikarb, prosimidon in tiametoksam vsako v 1 vzorcu (0,6 %), difenokonazol, linuron in metalaksil vsako v 2 vzorcih (1,2 %), lufenuron, spirodiklofen in tebufenozid vsako v 3 vzorcih (1,8%), metoksifenozid v 4 vzorcih (2,4%), flukvinkonazol in pirimetanil vsako v 5 vzorcih (3,0%), kaptan, klorpirifos in tiakloprid vsako v 6 vzorcih (3,6%), acetamiprid v 11 vzorcih (6,6%), boskalid in piraklostrobin, vsako v 14 vzorcih (8,4%), ter ditiokarbamate v 21 vzorcih (12,7%). Rezultati so zbrani v tabeli 3.

Aktivni spojini, ki sta presegali MRL sta bili: ditiokarbamati v enem vzorcu špinače (0,6%) in pendimetalin v enem vzorcu solate (0,6%).

INTRODUCTION

In the production of cereals, fruit and vegetables the appropriate protection from harmful organisms, which tend to appear in inappropriate places at inappropriate times, is needed. At present plant protection is based on the use of plant protection products (PPPs) which, when properly used, assure the most economical way of producing adequate quantities of high quality food. Incorrect and uncontrolled use of PPPs may cause harm to people, animals and the environment. Agricultural experts have been constantly trying to develop new technologies of healthy food production including the development of PPPs which would be friendlier to people, animals and the environment.

The task of the government is to control the proper use of PPPs, which insures healthy food on the market [1, 2, 7, 8, 11, 18]. This is why the Agricultural Institute of Slovenia was determining pesticide residues in agricultural products of Slovene producers prior to the market in accordance to Slovenian legislation [15-17]. The samples were taken randomly in eight production areas in Slovenia: Celje, Koper, Kranj, Nova Gorica, Novo mesto, Murska Sobota, Maribor, and Ljubljana. Each year analyses of pesticide residues are performed on potato, lettuce and apple samples due to the characteristic nutrition of Slovenes (the Slovene Food Basket has not yet been demarcated). Selection of other agricultural commodities and active substances followed the guidelines given in the European Union monitoring recommendations [9]. Beside apple, lettuce and potato samples, agricultural inspectors took samples of beans, carrots, cucumbers, pears and spinach in 2008 (Table 1). Agricultural products were taken directly in the field or in storehouses after the expiration of pre-harvest interval of the plant protection products used.

Control of pesticide residues in agricultural products prior to the market allows assessment of the conformity of production with good agricultural practice and the determination of sources and/or causes of residues found. Random choice of producers enables a statistical approach to the estimation of food safety on the Slovene market.

The results are intended to:

- Determine the conformity with the legally prescribed maximum residue levels (MRLs)

- Determine the conformity of the conventional, integrated and ecological production with good agricultural practice

- Determine the sources and/or causes of residues found

MATERIALS AND METHODS

Residues of 158 different compounds were determined using three different methods:

1. *Multiresidual GC/MS method* for the determination of 81 compounds: acephate, aldrin, azinphos-methyl, azoxystrobin, bifenthrin, boscalid, bromopropylate, bupirimate, captan, carbaryl, carbofuran, carboxin, chloridazon, chlorothalonil, chlorpropham, chlorpyriphos, chlorpyriphos-methyl, clomazone, cyhalotrin-lambda, cypermethrin, cyproconazole, cyprodinil, DDT, deltamethrin, diazinon, dichlofluanid, dichlorvos, dimethoate, diniconazole, diphenylamine, endosulfan, endrin, fenamidone, fenbuconazole, fenitrothion, fenthion, fludioxonil, folpet, HCH-a, heptachlor, heptenophos, imazalil, indoxacarb, iprodione, kresoxim-methyl, lindane, malathion, mecarbam, metalaxyl, metconazole, methacrifos, methamidophos, methidathion, metribuzin, myclobutanil, omethoate, oxydemeton-methyl, parathion, penconazole, permethrin, phorate, phosalone, pirimicarb, pirimiphos-methyl, procymidone, propargite, propyzamide, pyridaphenthion, pyrimethanil, quinalphos, quinoxyfen, spiroxamine, tebuconazole, thiabendazole, tolclofos-methyl, tolylfluanid, triadimefon, triadimenol, triazophos, trifloxystrobin and vinclozolin [3, 4].

2. *GC/MS method* for the determination of dithiocarbamate group: maneb, mankozeb, metiram, propineb and zineb, the sum is expressed as carbon disulfide [5].

3. *Multiresidual LC/MS/MS method* for the determination of 76 compounds: acetamiprid, aldicarb, aldicarb sulfon, aldicarb sulfoxid, amidosulfuron, benalaxyl, bentazon, bitertanol, buprofezin, carbendazim, clofentezine, clopyralid, clothianidin, cycloxydim, cymoxanil, cyromazine, desmedipham, difenoconazole, dimethomorph, epoxiconazole, ethofumesate, famoxadone, fenazaquin, fenhexamid, fenpropidin, fenpropimorf, fenpyroximate, flufenacet, fluroxypyr, fluquinconazole, flutriafol, foramsulfuron, hexythiazox, imidacloprid, iodosulfuron-methyl-sodium, iprovalicarb, isoxaflutole, linuron, lufenuron, malaoxon, metamitron, metazachlor, methiocarb, methiocarb sulfon, methiocarb sulfoxid, methomyl, methoxyfenozide, metosulam, napropamide, pendimethalin, phenmedipham, phoxim, prochloraz, propamocarb, propiconazole, prosulfocarb, prosulfuron, pymetrozine, pyraclostrobin, pyridate, rimsulfuron, spinosad, spirodiclofen, tebufenozide, terbuthylazine, tetraconazole, thiacloprid, thiamethoxam, thifensulfuron-methyl, thiodicarb, thiophanate-methyl, triasulfuron, trichlorfon, trifluralin, triflusulfuron-methyl, zoxamide [6, 13, 14].

The trueness of methods is verified by participation in the French inter-laboratory proficiency testing scheme BIPEA (Bureau interprofessionnel d´etudes analytiques) and CRL European Proficiency Test 10.

In January 2005, a range of analyses covering pesticide residues were accredited by the French accreditation body COFRAC.

166 samples of agricultural products presented in Table 1 were analysed in 2008.

RESULTS AND DISCUSSION

38 apple samples were analysed: residues exceeding MRLs were not determined, 27 samples (71.1%) contained residues lower than MRLs, residues were not found in 11 samples (28.9%).

Table 1: List of agricultural products, analysed in 2008, and distribution of sample locations among individual production areas.
Tabela 1: Seznam kmetijskih pridelkov slovenskega porekla, analiziranih v letu 2008 in porazdelitev lokacij vzorcev med posameznimi pridelovalnimi območji

Area	Agricultural product								Sum
	apples	beans	carrots	cucumbers	lettuce	pears	potatoes	spinach	
Celje	4	1	3	3	3	2	2	3	21
Koper	3	0	1	2	2	2	0	0	10
Kranj	1	1	2	1	2	0	12	0	19
Ljubljana	2	3	5	4	7	1	4	3	29
Maribor	14	3	3	4	5	5	5	0	39
Murska Sobota	2	0	0	2	2	1	3	0	10
Nova Gorica	1	0	1	1	1	3	0	0	7
Novo mesto	11	0	2	3	2	7	6	0	31
Sum	38	8	17	20	24	21	32	6	166

8 bean samples were analysed: residues were not found in 8 samples (100.0%).

17 carrot samples were analysed: residues exceeding MRLs were not determined, 2 samples (11.8%) contained residues lower than MRLs, residues were not found in 15 samples (88.2%).

20 cucumber samples were analysed: residues exceeding MRLs were not determined, 2 samples (10.0%) contained residues lower than MRLs, residues were not found in 18 samples (90.0%).

24 lettuce samples were analysed: one sample (4.2%) exceeded MRL, 3 samples (12.5%) contained residues lower than MRLs, residues were not found in 20 samples (83.3%).

21 pear samples were analysed: residues exceeding MRLs were not determined, 9 samples (42.9%) contained residues lower than MRLs, residues were not found in 12 samples (57.1%).

32 potato samples were analysed: residues were not found in 32 samples (100.0%).

6 spinach samples were analysed: one sample (16.7%) exceeded MRL, residues were not found in 5 samples (83.3%).

The results are presented in Figure 1.

29 samples (17.5%) out of 166 samples contained multiple residues. Residues of two active substances were determined in 7 out of 38 apple samples (18.4%) and 2 out of 21 pear samples (9.5%). Residues of three active substances were determined in 3 out of 38 apple samples (7.9%), 1 out of 20 cucumber samples (5.0%) and 4 out of 21 pear samples (19.0%). Four or more active substances were found in 10 out of 38 apple samples (26.3%) and 2 out of 21 pear samples (9.5%). The contribution of samples with multiple residues was 52.6% (20 samples) for apples, 5.0% (1 out of 20

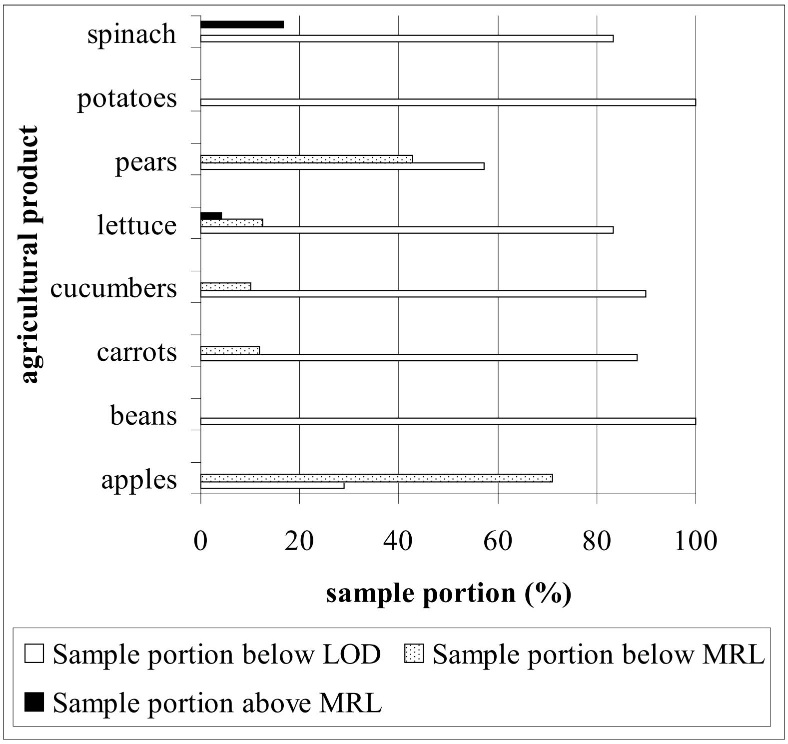
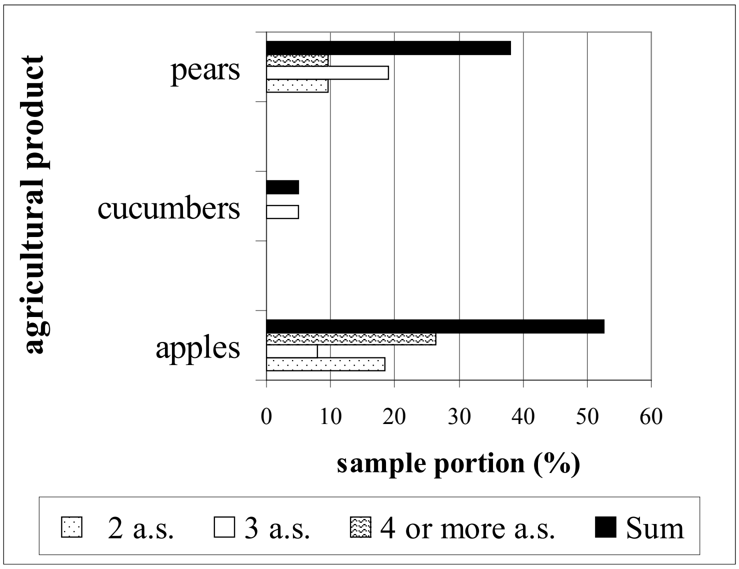


Figure 1: Plant protection product residues in agricultural products of Slovene origin in 2008
Slika 1: Ostanki fitofarmacevtskih sredstev v kmetijskih pridelkih slovenskega porekla v letu 2008

samples) for cucumbers and 38.1% (8 out of 21 samples) for pears. The results are shown in Figure 2. Residues of one active substance were found in carrot, lettuce and spinach samples while no residues were found in beans and potatoes.

Apples and pears were the matrices that contained the highest number of residues per sample. In Table 2, samples with 4 or more active substances found in one sample are presented. Boscalid and pyraclostrobin were the most frequent ones. They were both found in 8 samples of apples and pears. The highest number of different residues in one pear sample was 7.

Apple, bean, carrot, cucumber, lettuce, pear, potato and spinach samples contained the following active substances: clofentezine, cypermethrin, fenazaquin,



a.s. stands for active substances

a.s. pomeni aktivna substanca

Figure 2: Distribution of samples of Slovene origin with multiple plant protection product residues in 2008

Slika 2: Porazdelitev vzorcev slovenskega porekla z dvema ali več ostanki fitofarmacevtskih sredstev v letu 2008

pendimethalin, pirimicarb, procymidone and thiamethoxam each in one sample (0.6%), difenoconazole, linuron and metalaxyl each in 2 samples (1.2%), lufenuron, spirodiclofen and tebufenozide each in 3 samples (1.8%), methoxyfenozide in 4

Table 2: Agricultural products containing 4 or more residues per sample in 2008

Tabela 2: Kmetijski pridelki z vsebnostjo 4 ali več ostankov na vzorec v letu 2008

Product	Multiple pesticide residues from 1 sample (mgkg-1)						
Apple	Acetamiprid (0.01)	Boscalid (0.06)	Captan (0.44)	Dithiocarbamates (0.96)	Pyraclostrobin (0.02)	Pyrimethanil (0.02)	
Apple	Boscalid (0.09)	Chlorpyriphos (0.04)	Dithiocarbamates (0.06)	Pyraclostrobin (0.04)			
Apple	Boscalid (0.12)	Captan (0.54)	Chlorpyriphos (0.05)	Dithiocarbamates (0.11)	Pyraclostrobin (0.05)		
Apple	Chlorpyriphos (0.05)	Clofentezine (0.05)	Fenazaquin (0.03)	Fluquinconazole (0.02)	Pyrimethanil (0.02)		
Apple	Acetamiprid (0.01)	Difenoconazole (0.01)	Fluquinconazole (0.02)	Tebufenozide (0.17)			
Apple	Boscalid (0.02)	Dithiocarbamates (0.12)	Methoxyfenozide (0.01)	Pyraclostrobin (0.01)	Pyrimethanil (0.02)		
Apple	Boscalid (0.05)	Chlorpyriphos (0.11)	Methoxyfenozide (0.03)	Pyraclostrobin (0.02)			
Apple	Boscalid (0.10)	Captan (0.25)	Pyraclostrobin (0.04)	Spirodiclofen (0.03)			
Apple	Acetamiprid (0.02)	Captan (0.86)	Dithiocarbamates (0.12)	Spirodiclofen (0.04)			
Apple	Acetamiprid (0.03)	Boscalid (0.08)	Pyraclostrobin (0.03)	Thiacloprid (0.01)			
Pear	Acetamiprid (0.02)	Chlorpyriphos (0.05)	Dithiocarbamates (0.18)	Lufenuron (0.03)			
Pear	Boscalid (0.36)	Difenoconazole (0.01)	Dithiocarbamates (0.36)	Fluquinconazole (0.03)	Lufenuron (0.09)	Pyraclostrobin (0.13)	Thiacloprid (0.09)

samples (2.4%), fluquinconazole and pyrimethanil each in 5 samples (3.0%), captan, chlorpyrifos and thiacloprid each in 6 samples (3.6%), acetamiprid in 11 samples (6.6%), boscalid and pyraclostrobin each in 14 samples (8.4%) and, finally, dithiocarbamates in 21 samples (12.7%). The results are given in Table 3.

Active substances exceeding MRLs were the following: dithiocarbamates in one spinach sample and pendimethanil in one lettuce sample. The content of

Table 3: Number and portion of active substances found in each matrix analysed in 2008
Tabela 3: Število in delež določenih aktivnih snovi v vsaki matriki analizirani v 2008

	No. of samples									
Active substance	Apples	Beans	Carrots	Cucumbers	Lettuce	Pears	Potatoes	Spinach	Sum	Sample portion (%)
Acetamiprid	9	0	0	0	0	2	0	0	11	6.6
Boscalid	10	0	0	0	0	4	0	0	14	8.4
Captan	5	0	0	0	0	1	0	0	6	3.6
Chlorpyrifos	5	0	0	0	0	1	0	0	6	3.6
Clofentezine	1	0	0	0	0	0	0	0	1	0.6
Cypermethrin	0	0	0	0	1	0	0	0	1	0.6
Difenoconazole	1	0	0	0	0	1	0	0	2	1.2
Dithiocarbamates	13	0	0	1	1	5	0	1	21	12.7
Fenazaquin	1	0	0	0	0	0	0	0	1	0.6
Fluquinconazole	2	0	0	0	0	3	0	0	5	3.0
Linuron	0	0	2	0	0	0	0	0	2	1.2
Lufenuron	0	0	0	0	0	3	0	0	3	1.8
Metalaxyl	0	0	0	2	0	0	0	0	2	1.2
Methoxyfenozide	4	0	0	0	0	0	0	0	4	2.4
Pendimethalin	0	0	0	0	1	0	0	0	1	0.6
Pirimicarb	1	0	0	0	0	0	0	0	1	0.6
Procymidone	0	0	0	1	0	0	0	0	1	0.6
Pyraclostrobin	10	0	0	0	0	4	0	0	14	8.4
Pyrimethanil	5	0	0	0	0	0	0	0	5	3.0
Spirodiclofen	3	0	0	0	0	0	0	0	3	1.8
Tebufenozide	3	0	0	0	0	0	0	0	3	1.8
Thiacloprid	2	0	0	0	0	4	0	0	6	3.6
Thiamethoxam	0	0	0	0	1	0	0	0	1	0.6

dithiocarbamates in spinach sample was 0.07 $mgkg^{-1}$ (MRL = 0.05 $mgkg^{-1}$). The content of pendimethanil in lettuce sample was 0.06 $mgkg^{-1}$ (MRL = 0.05 $mgkg^{-1}$).

Active substances not registered in the Republic of Slovenia were found in cucumbers (procymidone), lettuce (cypermethrin, pendimethanil), pears (chlorpyrifos) and spinach (dithiocarbamates) [12].

Active substances not allowed in the integrated production in the Republic of Slovenia [19-21] and active substances not allowed in the ecological production in the Republic of Slovenia were not found.

In 121 samples (72.9%) out of 166 samples residues were not detected, 43 samples (25.9%) contained residues lower or equal to MRLs and 2 samples (1.2%) contained residues above MRLs (Fig. 3).

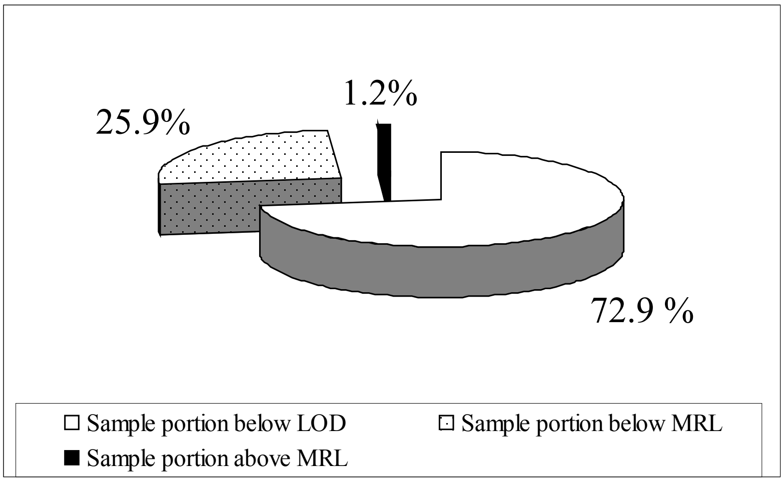


Figure 3: Results of monitoring in Slovenia in 2008
Slika 3: Rezultati monitoringa v Sloveniji v letu 2008

The results of monitoring obtained from 2001 to 2008 in Slovenia are slightly different than the results obtained in 2008. From 2001 to 2008, 1334 samples were analysed: in 804 samples (60.3%) residues were not detected, 465 samples (34.9%) contained residues lower or equal to MRLs and 65 samples (4.9%) contained residues above MRLs. In 2008 we can observe lower percentage of exceedances and higher percentage of samples where residues were not detected according to average from 2001 to 2008.

In the 2008 monitoring of pesticide residues in the products of plant origin in the 27 European union Member States (EU MS) and 2 European Free Trade Association (EFTA) States (Norway and Iceland) was found that in 62.1% of all examined fruit (oranges, mandarins, pears), vegetable (potatoes, carrots, cucumbers, spinach, beans without pods) and cereal (rice) samples PPP residues were not detected, 35.7% of samples contained residues lower or equal to MRLs and 2.2% of the examined samples contained residues above MRLs [10].

CONCLUSIONS

Levels of pesticide residues in agricultural products in Slovenia in 2008 do not give any cause for alarm. Only 2 samples of the 166 analysed exceeded MRLs (1.2%). The risk assessment showed that the acute exposure of the consumers to both exceeded samples was acceptable.

23 (14.6%) out of 158 active substances sought were found. The most frequently found active substance was dithiocarbamates. Matrices with high number of active substances in one sample were mainly apples and pears (fruit). The highest number of active substances in one pear sample was 7.

There were some violations of unregistered active substances used, but there was no violation of integrated or ecological production.

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