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STUDIES REGARDING THE TRACEABILITY TESTING IN ORGANOCHLORINE PESTICIDES CONTAMINATION OF MILK

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ABSTRACT. The contamination of animal-based foods has a main source the food chain of that species. The paper aims at following the possible traceability of organochlorinated pesticides residues found in raw material milk from an area near the Oradea Municipality. For this purpose I tested samples of green fodders and water, gathered from the grassing area belonging to the village with the highest weight in the production of the raw material milk tested before. For comparison, the determinations have been performed using the same method, devices and chromatography conditions as in the case of milk. In respect of the 16 compounds tested, the majority of those identified positively in milk were found in the fodders and waters tested, in various concentrations.

Keywords: traceability, contaminants, milk, fodder, water

INTRODUCTION

As a concept connected to food safety, traceability allows the study of a product from the raw material until the sale, from administrative and qualitative point of view (Savu and georgestu, 2004). In the case of a food such as drinking milk, chemical contaminants such as organochlorinated pesticides from the raw material milk, another type of contamination being strictly incidental. Taking into consideration the bioaccumulable character of organochlorinated pesticides, more obvious in fat foods, the food chain is thought to be the most likely source of contamination.

From this reason the present document aims at investigating the incidence and the concentration of OCP residues that appear in some of the fodders and waters probably consumed by the animals from which the samples of raw materials tested have been gathered whose contamination has been determined previously (Chiş, 2008 a and b). The work will be continued on sour milk and cheese. The compounds for which the sanitary veterinary regulations set maximum residues level (Legis) have been studied.

MATERIALS AND METHODS

Materials

For the study that makes the object of the present document samples of raw material milk, green fodders and well waters (fountains) have been studied. Since this paper is a research study, not the result of a control activity, the name of the village from where the samples have been taken, was not revealed. They were coded by letters and numbers (Tables 2 to 7). From geographical point of view it is the area near the municipality of Oradea.

The samples of fodders and water came from the grassing area that belongs to the village with the highest weight in the production of the previously tested raw material milk. The fodders consisted of green mass gathered from the field and the water samples came from two fountains used for drinking.

Methods

The gathering of the product samples

The recipients used for the gathering were made of plastic for the samples of fodders, respectively milk (single use) and glass for water samples. All the containers are water-tight and they were cleaned properly to avoid the contamination hazard. At the end they were rinsed with light petroleum. The samples were transported with a cooling case.

The chemical determinations tied to characteristics, according to the samples tested were gathered in the period of time, respectively 24 hours but the contaminants analysis were performed both on fresh foods (fodders and waters) and of frozen products (milk), since the OCP determinations were executed at the Institute of Hygiene and Public Health from Cluj.

The gathering of the samples was made in June-August 2007 and January March 2008. The gathering of the samples of fodders and waters was made in the months of June and July.

The preparation of the samples for analysis

The preparation of the milk samples consists in the homogenization and bringing at the required temperature, 20°C (Guş and Semeniuc, 2005). In the case of frozen samples, they were defrost at room temperature then they were prepared as for fresh products (only for the fat separation for the determination of pesticides). The density of milk was determined. The measurement of the density of milk was necessary for the determinations in which we worked with test measured by volume but where, in the calculus, we used quantities. We applied the aerometric method (SR2418/2008) using a thermolactodensimeter, that allowed us to correct the read values according to the temperature of the sample for values at 20°C.

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The preparation of the fodders samples consisted in the determination of the humidity since the extraction technique of the OCP from non-fat products depends on the content of water of the samples. (SR EN 12393-2004). The used method was the reference one that is the drying in the thermo-adjustable oven, at 105°C, for 4 hours; the determination was performed immediately after the gathering in the same day in the food toxicology laboratory of the Faculty of Environment Protection from Oradea.

For the water samples the pH was verified and no correction was necessary because it ranged in all the samples 5-7.5. Since we also wanted to test the likely presence of endosulfan, a separate sample was gathered from which an acid pH has been maintained, at value 2. The samples were kept before the extraction at 4°C, in the dark and the extraction was performed in the first 24 hours in order to prevent decomposing. The suspensions were determined because the method applied for the determination of the residues of organochlorinated pesticides is influenced by the content of suspensions from the water samples. The method consists of the separation of the suspensions through filtration, followed by their gravimetric determination. (Mănescu *et al.*, 1994).

The preparation of the samples for chromatography

All the reagents used were Merck type with chromatographic purity and no additional purifications were performed, but witness testes of the reagents on each work phase. Of the methods used and recommended in the literature and legislation, we selected those that use the same type of solvents for the extraction, respectively purification in order to obtain results with a high level of comparability.

Milk

The lyposoluble contaminants are analyzed; the phases performed for the qualitative and quantitative are (SR EN 1528, 2004):

- The separation of the fat that contains the toxic: the method used was partition with n-hexan/acetone 2:1 mixture (V/V), drying on anhydrous sodium sulphate and evaporation of the solvent;
- Extract purification: performed through repeated partitions with acetonitryl (saturated with light petroleum) and light petroleum. The dehydration of the etheric extracts reunited through passing on column of anhydrous sodium sulphate.
- The extracts dehydrated and evaporated up to about 10 ml were passed on activated Florisil column from where the OCPs were eluted with 200ml mixture of light petroleum / ethylic ether 94/6 (V:V). The maximum elution speed was 5ml/minute.
- The mixed eluates were eluted in the spinning-evaporator.

Figure 1 shows the final phase of the separation and purification phases, respectively the evaporation of the solvents with the spinning-evaporator, at low pressure induced by the water trunk.

Fig. 1 The evaporation of the solvent in the extracted fat (source: personal archive)

Fodders

The technique used is of the type of extractions with solvent, followed by liquid/liquid partition – LLE (Schenck and Hobbs, 2004). The phases carried out are (SR EN 12393-2004):

- The toxic extraction: an amount exactly weighted, as closely to 100 g as possible, from the grounded sample was homogenized with 200 ml of acetonitryl without adding water, because the humidity was over 75%. The filtrate (F) resulted is extracted through partition with 100 ml light petroleum in the separation funnel. The extract (P) goes through the purification phase; the F and P values resulted in the phase of sample preparation is noted during the determination because they will be used in the calculation of the amount of sample that goes though the purification column.
- Colum purification with Florisil: three successive elutions are performed with 200 ml mixture elution ethilic ether / petroleum ether, V/V proportion: 6.94 (A), 15:85 (B) and 50:50 (C).

Waters

For the determinations from the present paper, the extraction of the OCP pesticides through the LLE method (Gan and Bondarenko, 2008) was used. The petroleum ether is used as extraction solvent. The extract was not purified anymore because the tested samples had suspension values under 0.05 mg/l (SR EN ISO 6468/2000).

The determinations of the residues of organochlorine pesticides in the studies products

In order to have comparable results, the same method was used for all the types of samples, that is gas chromatography (Tadeo, 2008; Hura, 2006). Moreover, it presents the method set forth by current

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legislation for the calculation of pesticide residues in water, animal and vegetal products.

The same device has been used for all the samples in similar chromatography conditions, as follows:

Used Devices

We have used a GC 2010 Shimadzu gas chromatograph, with the following characteristics:

- Capillary column type RTX -CL-pesticides 30 m length an 0.25 mm diameter. The column works at a temperature between 150 ÷ 320°C with a gradient of 3°C/2 minutes
- Detector with electrons capture (ECD), nuclide ^{63}Ni – 370 MBq (10mCu)
- Autosampler injection system with 6+2 spaces for vials, type AOC-210

Chromatography conditions:

- Injection temperature (splitting) = 250°C
- Splitting temperature = 163.5 Kpa
- Splitting gas : He with a 124 ml/min flow at scavenging 30 ml/min
- Carrying gas : N_2 ultrapure 99,99%
- Detector: = 320°C, detector current of 2 nA, make-up flow = 30 ml/min

The device is connected to a computer and uses a specific program for the interpretation of the results. Chromatograms are displayed on a singular monitor for the tested sample or together with the chromatogram of the used sample. The program supplies the retention times, the height of the picks and their surface, through automatic integration.

Qualitative and quantitative calculations

All tested samples submitted to the verification from the point of view of the contamination with organochlorine pesticides have undergone the procedures explained in "Test preparation for chromatography" part of the paper. The extract purified, retaken in petroleum ether was submitted to the chromatography under the same conditions as the standard test, as well as the blanks-test of the used reagents, according to the separation / purification method.

For the qualitative and quantitative calculation of the contaminants possibly present in the tested products, we used a standard produces by the RESTEK Company No 32292, Lot nr A021837, type "Organochlorine pesticide Mix ~~A~~B" having a concentration of 200 ppb. Notice the fact that the standard contains, with one exception (hexachlorobenzen) all the organochlorine compounds under sanitary-veterinary surveillance in vegetal and animal foods. The standard was used at the 50 ppb dilution for milk and green fodder samples and at the 40 ppb dilution for water samples.

Even if the same sample was used for all the tested samples, the value of the retention times pertaining to each compound is not identical, which is exemplified in table 1. This value varied according to the concentration of the injected standard and the modification of any of the work conditions. Even if for the determination pertaining to the fodders samples the standard was used at the same concentration as for milk samples, the change of the catching position in time of the chromatography column, led to the slight modification of the retention time of the 20 compounds from the standard. The elution order does not suffer any modification. This observation indicated the necessity to reinject the sample before each new series of determinations, for an increased accuracy, strictly necessary in case of determination from the ppb and ppm range of concentration, In the case of change of any of the work conditions, the reinjection of the sample is compulsory.

Table 1

Retention times of the components of the complex standard, in various work conditions

Elution order	Organochlorine compound from the standard	Retention time, min		
		Raw milk	Green fodder	Water
1	α HCH	6.811	7.194	7.191
2	γ HCH	7.978	8.426	8.42
3	β HCH	8.403	8.886	8.873
4	δ HCH	9.074	9.552	9.553
5	Heptaclor	9.898	10.372	10.357
6	Aldrin	11.156	11.657	11.641
7	Heptaclor epoxid	14.036	14.652	14.63
8	γ Chlordan	14.657	15.274	15.251
9	α Chlordan	15.322	15.951	15.928
10	4,4' DDE	15.849	16.518	16.493
11	α endosulfan	16.088	16.753	16.733
12	Dieldrin	17.103	17.766	17.743
13	Endrin	18.18	18.878	18.853
14	4,4' DDD	19.105	19.853	19.831
15	β Endosulfan	19.361	20.076	20.049
16	4,4' DDT	20.564	21.305	21.280
17	Endrin aldehida	21.585	22.385	22.355
18	Metoxiclor	23.648	24.346	24.317
19	Sulfat de endosulfan	23.845	24.693	24.66
20	Endrin cetona	25.205	26.030	25.998

For the quantitative determinations, we have used the value of the surface of the drops (compounds that have been previously identified as being present in the tested sample). In order to prevent possible calculation errors, we introduced the data we obtained through the automatic integration of the unknown tests and the used standard in EXCELL calculation sheets, selecting only the surface of the picks we need, that is the compounds that were found from the qualitative point of view. According to the legislative requirements (SR 1528-2004 and SR EN ISO 6468-2000), in the quantitative calculation of the raw milk and water tests, we took into consideration the percent of recovery of the residues. They were between 80.8% - 103.2% for raw material milk and between 88% - 90% for water. In the case of non-fat products, the values obtained are not corrected with the recovery degree (SR EN 12393-2004).

RESULTS

For the qualitative determination, we compared the retention times for the significant picks that appear on the chromatogram of the unknown tests with the ones of the compounds from the used standard sample. This way, we can determine which of the compounds that are present in the standard can also be found in the tested sample (Gocan, 1998). The results are written in tables 2, 3 and 4 for milk samples, green fodder samples, respectively water samples. For the green fodders the results include the signals from the three elutions performed.

Table 2

Qualitative calculation of OCP residues in raw material milk

A	B	C	D - 06-08.2007			D - 01-03-2008		
			LMA	LMB	LMC	LMA	LMB	LMC
1	α HCH	6.811	6.951	6.798	6.799		6.801	6.815
2	γ HCH	7.978	7.956	7.963	7.963	7.966	7.956	7.944
3	β HCH	8.403	8.380	8.387	8.380	8.384		8.378
6	Aldrin	11.156	11.163					
9	α Chlordan	15.322						
11	α endosulfan	16.088	16.050	16.060	16.060	16.068	16.081	16.068
12	Dieldrin	17.103	17.057					
13	Endrin	18.18	18.159	18.168	18.169	18.176	18.164	18.161
14	4,4' DDD	19.105	19.064			20.529	20.522	
16	4,4' DDT	20.564	20.520	20.531	20.533		23.831	20.533

Table 3

Qualitative calculation of the OCP residues, green fodder

A	B	C	D		
			FV1	FV2	FV3
1	α HCH	7.194	7.179	7.183	7.188
2	γ HCH	8.426	8.409	8.410	8.418
3	β HCH	8.886	8.791		8.818
11	α endosulfan	16.753	16.730	16.744	16.751
13	Endrin	18.878	18.831	18.848	18.838
14	4,4’ DDD	19.853	19.809	19.814	19.822
16	4,4’ DDT	21.305	21.273	21.288	21.398
19	Sulfat endosulfan	24.693	24.798		

Table 4

Qualitative calculation of the OCP residues, well water

A	B	C	D			
			Source 1		Source 2	
			Test 1	Test 2	Test 1	Test 2
1	α HCH	7.191		7.173		
2	γ HCH	8.420	8.412	8.402	8.390	8.398
3	β HCH	8.873			8.920	8.913
10	4,4' DDE	16.493	16.523			
11	α endosulfan	16.733	16.743	16.714	16.697	16.679
12	Dieldrin	17.743	17.731			
13	Endrin	18.853	18.861	18.828	18.838	18.795
14	4,4' DDD	19.831	19.844			
16	4,4'DDT	21.280	21.295	21.260	21.268	21.244

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Legend for tables 2, 3 and 4:

A - Elutions order

B - Organochlorine compound type from the sample

C - Retention time in the standard of the respective compound

D - Retention time in the sample of that respective compound

Figures 2, 3 and 4 show an example of scanned chromatogram for each of the tested materials: raw material milk, green fodder, water.

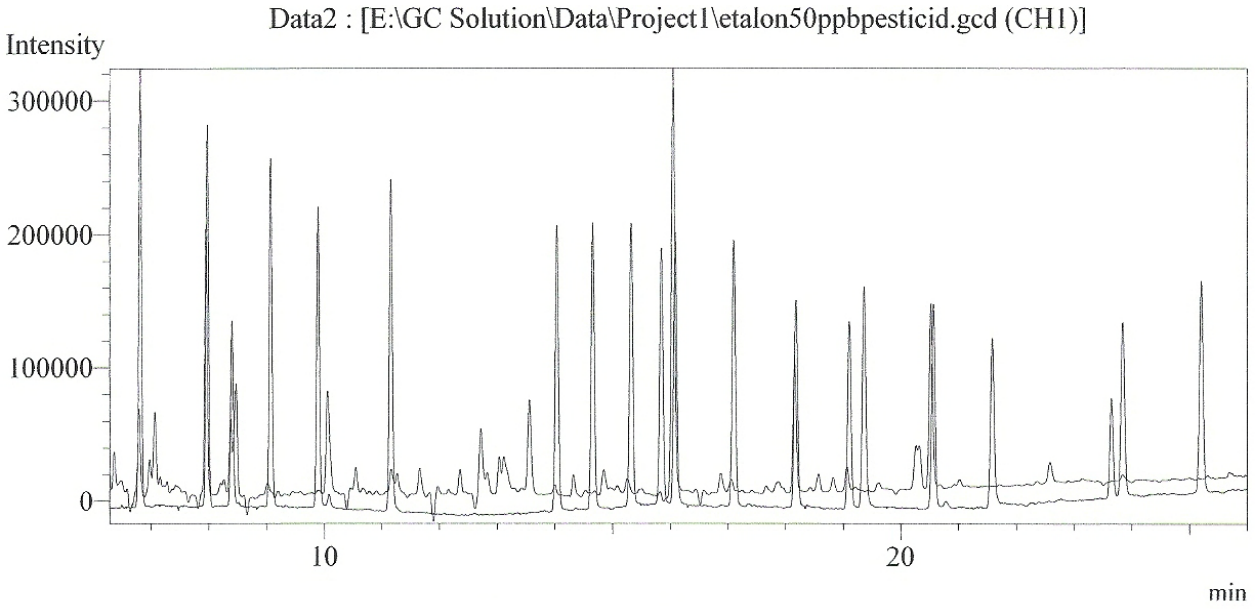

Fig. 2 Chromatogram, raw material milk (source: personal archive)

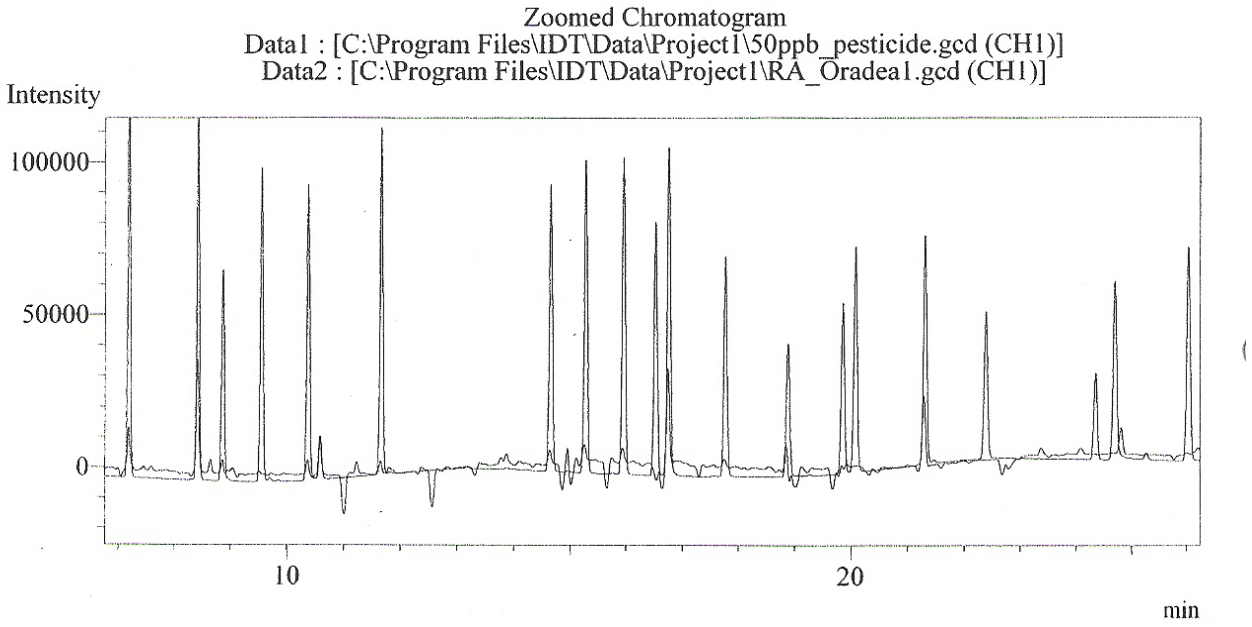

Fig. 3 Chromatogram, green fodder (source: personal archive)

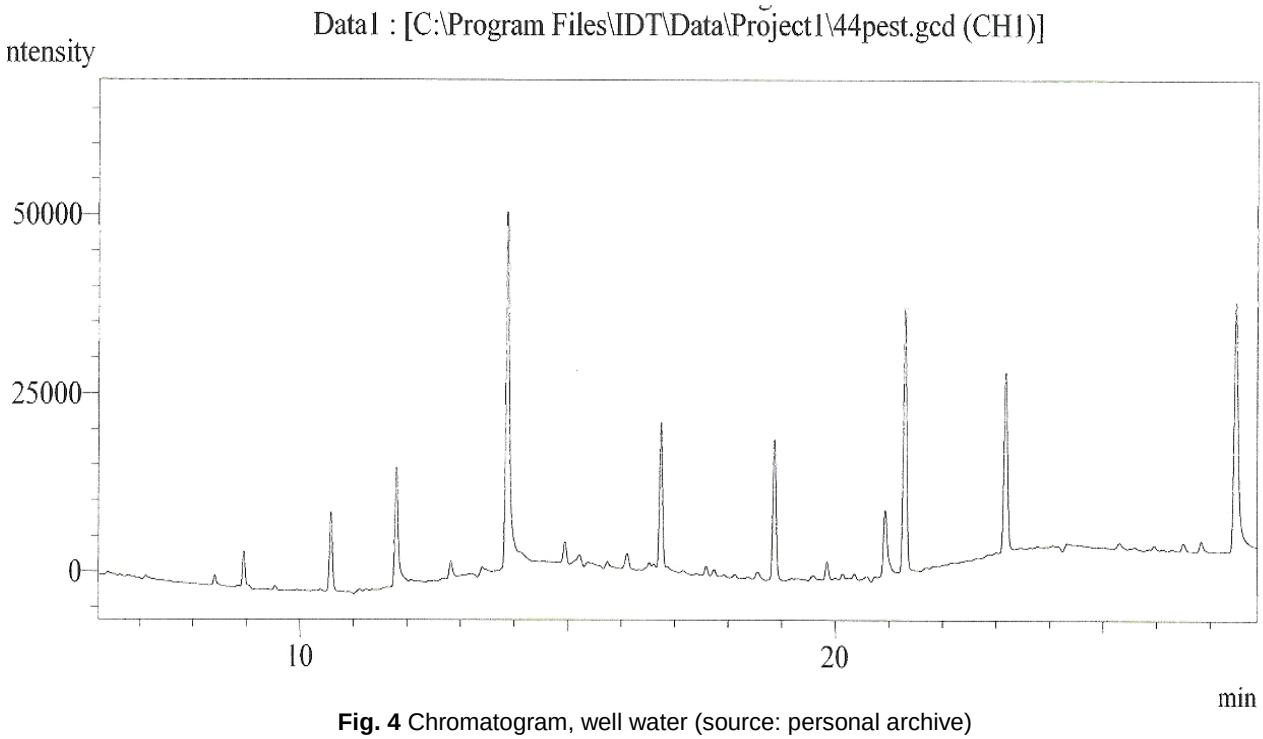

Fig. 4 Chromatogram, well water (source: personal archive)

The results of the quantitative determination are shown in tables 5, 6 and 7. The values of the concentration of the maximum resides level (MRL) is the one specific to the tested sample, according to the sanitary veterinary norms in vigour (Legis). For the green fodders the results include the sum of the values from the three elutions performed. In what regards water, the requirements referring to the pesticide contamination with OCP pesticides were taken into account, because the tested water is used for animals to drink. Therefore the value for each compound in part is limited as well as the total value of the class of contaminants tested.

Table 5

Quantitative calculation of the OCP residues in raw material milk

Nr (*)	Organochlorine compound found in the sample	MRL, ppm	Concentration found in the sample, ppm 06-08.2007			01-03.2008		
			LMA	LMB	LMC	LA	LB	LC
1	α HCH	0,004	0.0002	0.0002	0.0006		0.0004	Sld
2	γ HCH	0.003	0.0007	0.0007	0.0005	0.0008	0.0003	0.0002
3	β HCH	0.008	0.0004	0.0009	0.0002	0.0001		0.0004
6	Aldrin[1]	0.006	0.0001					
12	Dieldrin	0.006	sld					
11,15,19	Endosulfan (sum of α and β and endosulfan sulphate)	0.004	0.0015	0.0016	0.0013	0.0042	0.0024	0.0024
13	Endrin	0.0008	0.0006	0.0008	0.0006	0.0004	0.0006	0.0004
10,14,16	DDT (sum of DDT, DDE and DDD isomers)	0.04	0.0009	0.0011	0.0008	0.0007	0.0007	00007

Table 6

Quantitative calculation of the OCP residues, green fodder

Nr. (*)	Organochlorine compound found in the sample	MRL ppm	Concentration in the sample, ppm		
			FV1	FV2	FV3
1,3	HCH (sum of izomer α şi β)	0.01	0.0011	0.0005	0.0013
2	γ HCH	0.01	0.0009	0.0012	0.0034
11.15.19	Sum of α and β and endosulfan sulphate expressed in Endosulfan	0.05	0.0021	0.0021	0.0038
13	Endrin	0.01	0.0008	0.0013	0.0016
10.14.16	Sum of DDT. DDE and DDT isomers exressed in DDT	0.05	0.0007	0.0002	0.0013

Table 7

Quantitative calculation of the OCP residues. well water source

Nr. (*)	Organochlorine compound found in the sample	MRL, Ppb	Concentration in the sample. ppb			
			Source 1		Source 2	
			Test 1	Test 2	Test 1	Test 2
1	α HCH	0.1		0.0012		
2	γ HCH	0.1	0.0015	0.0144	0.0021	0.0013
3	β HCH	0.1			0.0121	0.0028
10	4.4' DDE	0.1	0.0015			
11	alfa endosulfan	0.1	0.0459	0.0466	0.0186	0.0250
12	Dieldrin	0.1	0.0026			
13	Endrin	0.1	0.0837	0.0460	0.0153	0.0228
14	4.4' DDD	0.1	0.0074			
16	4.4' DDT	0.1	0.2400	0.0578	0.0174	0.0248
	TOTAL POC	0.5	0.3634	0.1660	0.0655	0.0767

Legend for tables 5, 6 and 7:

(*) –refers to the elution order of the compounds in the samples, respectively in the standard

MRL - Maximum admitted level

DISCUSSIONS

Referring to the types of organochlorinated pesticides tested followed from the sanitary veterinary point of view and whose residues appear in the samples tested

Raw material milk (Table 2)

The pesticides that appear in all the tested samples are γHCH, α Endosulfan, Endrin and DDT.

The pesticide that appear in most samples are αHCH and βHCH (83% of the samples).

The pesticides that appear randomly are Aldrin and Dieldrin (17% of the samples).

Green fodders (Table 3)

The pesticides that appear in all the tested samples are αHCH, γHCH, Endosulfan (sum of isomers), Endrin and DDT isomers.

The pesticide that appears in most samples is βHCH (66% of the samples).

Source water (Table 4)

The pesticides that appear in all the tested samples are γHCH, α Endosulfan, Endrin and 4,4' DDT.

The pesticide that appears in most samples is βHCH (50% of the samples).

The pesticides that appear randomly are αHCH, 4,4'DDE, Dieldrin, 4,4'DDD (25% of the samples).

Note: in the case of water DDT isomers are studied separately, for this reason the discution present them this way.

Referring to the concentration of organochlorinated pesticides found in the tested samples

Raw material milk (Table 5)

Endosulfan and Endrin are the compounds for which we calculated a concentration value equal to the MRL, in different samples (17%). In the same time one can observe the fact that in the rest of the samples, the calculated values range between 50% and 75% towards the MRL and between 32% and 60% for Endosulfan, which is relevant in the ranger order we refer to. The other compounds identified qualitatively have concentration values much under the MRL, even by two range orders.

Green fodders (Table 6)

All the compounds identified from the quality point of view, have concentration valued under the MRL, between 10^{-1} and 10^{-2} but different between the two tested sources. This situation can be due to the difference of depth and type of soil corresponding to the two sources of water testes. The concentration calculated for Endrin and for Endosulfan have the same range order as for milk and for HCH and DDT are lower than for milk.

Well water (Table 7)

In what regards the indicator "total OCP class", all the tested samples range under the MRL value, but for each compound identified positively the situation is different. The 4'4 DDT isomer exceeds the MRL value for 24% of the samples and for the other – the values fall under 46% and 84% towards the MRL. The rest of the compounds have concentrations clearly different from the MRL values. Considering the fact that for water the calculations are expressed in ppb and in milk they are expressed in ppm, in the case of water all the values are much under than milk.

The reasons of the situation acknowledged are different. Therefore, the α, β, γ isomers appear in most samples tested in a different combination of isomers. This aspect is due to the use of this product for over 30 years which led to its strong diffusion in the environment factors. On the other side, the γHCH isomer as a sole form of Lindan or as a component of some combined insectofungicide has been admitted for use in the EU until 2002 and in our country until the adhesion to the European Union in 2007. Hence, it is likely to be found in the environment and in foods for a long period of time from now on.

Endosulfan is the only OCP that was still in use in the first period of tests because it has been prohibited

only starting with 01.01.2008, so it is likely to be found in the tested samples.

Endrin is a stereoisomer form of Aldrin and Dieldrin and it has been prohibited in the same time as Dieldrin. However, its incidence in the tested samples is much bigger that the one of its isomers (being identified positively in all the samples) because it has a higher capacity of persistence and accumulation in the soil where it can be absorbed by the fodders. This way it can get in the animals' food. It is part of the OCP group with a big capacity of bioaccumulation (Kegley, 2007);

DDT (alone or as sum of isomers) is a pesticide that has been used massively and, despite the fact that it has not been used since the same period as HCH, practically it can be found in all the environment factors, therefore in foods too, because of its bioaccumulation capacity. Moreover, it is used in Asia for the control of tropical diseases.

CONCLUSIONS

Analysing the situation resulted after the determinations performed we can observe that those compounds that appear in all the raw material milk samples, can also be found in fodders and waters. The differences appear only at the level of the compounds that are considered as sum of isomers for milk and fodders, unlike for waters. So the idea according which the food chain is responsible for the contamination of milk with organochlorinated pesticides stays valid.

Anyway, in any case the concentration is higher in milk then in fodders or water due to the liposolubility and bioaccumulation. The situation is due to the short period of life of the green mass and, implicitly, the reduced period of contact with soil and water through which they can be impurified. These values have the same range order with the ones found in a study on the vegetation at altitude between 4700 – 5620 m of the Asian mountains (Wang et al., 2006). For waters the responsibility falls on the difference of solubility of the compounds tested towards milk.

An incidence similar to the OCP in milk was observed in the Asian countries where these compounds are still in used or have been recently restricted (Nag and Raikwar 2008, Sharma *et al.* 2006). But in the studies, the concentrations calculated are much above the ones found in the samples tested in the present study, from 17 up to 82 times higher. The conclusion is that the use duration and the scale of use influences the concentration of the residues because of the liposolubility but less on the incidence, because of the bioaccumulation characteristic of these contaminants.

The global traceability is for now only a concept but the studies tied to the sources and ways of contamination have an important role on food safety, therefore it is necessary for them to be continued and perfected.

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