

## Researches concerning HCH residues in feed and beef meat in a west Romanian county area

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### Abstract

The use of HCH as a pesticide was banned in the EU since several years, but residues could be found even in present due to bioaccumulation and biomagnification in the environment. The goal of this paper is to emphasize HCH residues levels in feed and beef meat from a west Romanian county area. Conclusions: the highest pesticide residues levels in feed were those of  $\gamma$ -HCH, which are situated, as medium values, in national guidelines values, but not the Europeans ones; in beef meat, medium  $\alpha$ ,  $\beta$  and  $\gamma$ -HCH concentrations are situated both in national and European guidelines levels, excepted  $\gamma$ -HCH concentrations which exceed twice European standards; by reason of HCH isomers persistence in environment, pesticide feed residues have to be surveyed in order not to become contamination sources for animals and its, on their turn, considering bioaccumulation and biomagnification properties of organochlorine pesticides, contamination sources for man.

**Keywords:** HCH, residues, feed, beef meat

### 1. Introduction

It is Hexachlorocyclohexanes (HCHs) are a group of manufactured chemicals that do not occur naturally in the environment. Technical HCH is a mixture of various HCH isomers; alpha ( $\alpha$ ), beta ( $\beta$ ), delta ( $\delta$ ) and gamma ( $\gamma$ ) (also known as lindane). The compound with the highest pesticide activity of these is  $\gamma$ -HCH, more commonly known as lindane[1,2].

It has been used as a spray for foliage, in soil applications, for seed treatment and in baits for rodent control. Furthermore, it has been applied on a variety of fruits, seed grains, and vegetable crops, in forestry and for poultry and other livestock, and also in human medicine for treatment of head lice [3, 4]. The use of plant protection products, containing  $\gamma$ -HCH as an active substance, has been banned in the EU since June 2002[5].

$\gamma$ -HCH has an acute toxic effect in mammals, which is much greater than that of any of the other isomers.

On the other hand,  $\beta$ -HCH is the isomer most strongly retained in the body fat, and may therefore cause chronic toxicity at low doses due to continuous accumulation [6, 7, 8]. Because of the lipophilic properties and persistence in the environment,  $\beta$ -HCH followed by  $\alpha$ -HCH and to a less extent  $\gamma$ -HCH may give rise to bioaccumulation and biomagnification through the food chain [9].

Food is the main source of exposure to HCHs for the general population. In order to avoid any environment misbalance or health problems is essential to make a surveillance of residues levels through the food chain. The goal of this paper is to emphasize some organochlorine pesticide ( $\alpha$ ,  $\beta$ ,  $\gamma$  HCH) residues levels in feed and beef meat from a west Romanian county area.

## 2. Material and methods

There were sampled five feeds, meat and fat beef samples in each of second, third and fourth trimester of last year (2008) from a west county of our country. Meat and fat samples were taken after the slaughtering in the slaughter house, "X" of animals issued from population.

Animal and feeds samples were homogenized, extracted and according to standardized methods. 10-30 g of sample was homogenized with a triple quantity of anhydrous sodium sulphate, 1-2 minutes. The homogenized sample was extracted over 8 hours in SOXHLET device with n-hexane.

An aliquot containing 200 mg fat was transferred into a glass cylinder and purified with concentrated sulphuric acid (for fat disintegration) by active shaking over 10 minutes. The extraction was purified and discolored. A known quantity of purified extraction was evaporated in a nitrogen flow and then diluted by 1ml of hexane. Approximate 2-5 µl were injected in gas-chromatograph.

The analyze of organochlorine compounds was done by Perkin Elmer Sigma 300 gas-chromatograph with <sup>63</sup>Ni detector of electrons capture and 2.5 m. capillary channel, 0.22 mm internal diameter and 0.12 µm film thick. Operating conditions were: column height 200°C, injector temperature 250°C, detector temperature 300°C, transporter gas flow (argon) 0.92 ml/min.

Sample pesticide residues were identified based on their retention time, quantified based on correspondent peak height, related to sample weight and expressed on mg/kg. Since recover percent was optimal, there were no necessary corrections during calculations of samples. The obtained data were statistical analyzed.

## 3. Results and discussion

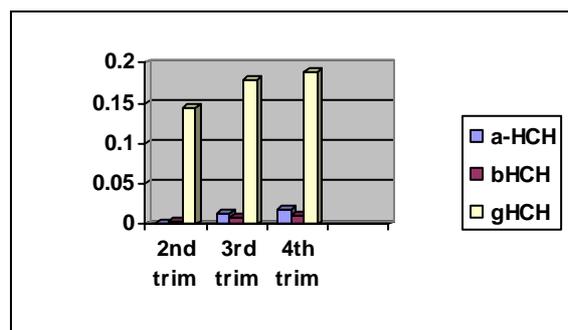
Residues levels of α, β, γ- HCH from feed samples are presented in table 1 and figure 1.

From the results presented in table and figure 1 we can observe that the highest levels of feed residues were those of γ-HCH in all sampling trimesters.

The maximum admitted α, β, γ- HCH levels in feed, according national and European legislation are presented in table 2 [5, 10].

**Table 1.** α, β, γ- HCH levels in feed (mg/kg)

Isomer	Concentration	2 <sup>nd</sup> Trim	3 <sup>rd</sup> Trim	4 <sup>th</sup> Trim
α-HCH	x ± Sx	0,0022 ± 0,0016	0,013 ± 0,001	0,019 ± 0,013
	min	0,000	0,000	0,000
	max	0,0139	0,082	0,152
β-HCH	x ± Sx	0,004 ± 0,001	0,009 ± 0,001	0,012 ± 0,006
	min	0,000	0,000	0,000
	max	0,067	0,115	0,049
γ-HCH	x ± Sx	0,145 ± 0,015	0,179 ± 0,038	0,188 ± 0,042
	min	0,000	0,124	0,014
	max	0,200	0,211	0,250



**Figure 1.** Pesticide feed residues levels in all sampling trimesters

**Table 2.** Maximum admitted α, β, γ- HCH levels in feed

Specification	Analyzed parameter (ppm)			Reference guidelines
	α-HCH	β-HCH	γ- HCH	
Feed	0,02	0,01	0,2	ANSVSA Order 18/1.02.2007
	0,02	0,005	0,01	CE Directive 32/2002

As we can observe from the obtained data, medium levels of α-HCH in all analyzing periods (0.002 – 0.019 mg/kg) are between maximum admitted limits, both by national and European guidelines, but maximum concentrations registered in the 3<sup>rd</sup> (0.082 mg/kg) and, respectively 4<sup>th</sup> trimesters (0.152 mg/kg) exceed these guidelines by 4.1, respectively 7.6 times.

Medium β-HCH concentrations (0.004 – 0.012 mg/kg) are situated between national guidelines levels, but not in the European's ones, which are exceeded by 1.25, respectively 2.4 times. Maximum concentrations levels registered on this parameter (0.067; 0.115; 0.045 mg/kg) are not situated nor in national guidelines that are exceeded by 6.7, 11.5 respectively 4.5 times, neither in those Europeans that are exceeded by 13.4, 23, respectively 9 times.

Medium  $\gamma$ - HCH concentrations levels (0.145 – 0.188 mg/kg) are situated between national guidelines levels, but not in the European’s ones, which are exceeded by 14.5 respectively 18.8 times. Maximum concentrations levels registered on this parameter (0.200 – 0.250 mg/kg) slightly exceed national guidelines (by 1.25 times), but much more the European one (by 25 times).

The obtained data after these analyze are comparable with those registered in other EU countries [5]. Because HCH use was continuous restricted lately, identification frequency in feed and vegetables of these compounds and detected levels are, generally low and will continuously lower. The  $\alpha$ ,  $\beta$ ,  $\gamma$ - HCH concentrations in beef meat come from the same region and same time periods are presented in table 3:

**Table 3.**  $\alpha$ ,  $\beta$ ,  $\gamma$ - HCH concentrations in beef meat (mg/kg)

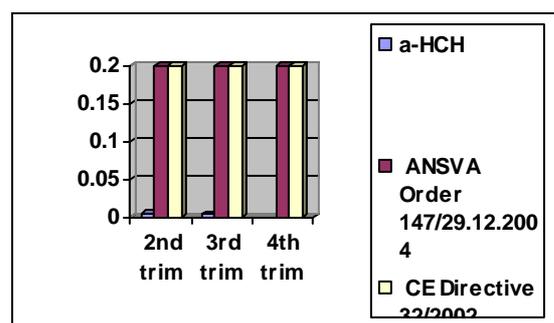
Isomer	Concentration	2 <sup>nd</sup> Trim	3 <sup>rd</sup> Trim	4 <sup>th</sup> Trim
$\alpha$ -HCH	x ± Sx min max	0,005 ± 0,0018 0,000 0,0079	0,004 ± 0,000 0,000 0,067	Undetectable
$\beta$ -HCH	x ± Sx min max	0,034 ± 0,039 0,000 0,079	Undetectable	0,049 ± 0,018 0,000 0,560
$\gamma$ - HCH	x ± Sx min max	0,088 ± 0,0016 0,000 0,1698	Undetectable	0,042 ± 0,015 0,000 0,265

Maximum admitted  $\alpha$ ,  $\beta$ ,  $\gamma$ - HCH concentrations in beef meat, according national and Europeans guidelines are presented in table 4[5, 11]:

**Table 4.** Maximum admitted  $\alpha$ ,  $\beta$ ,  $\gamma$ - HCH concentrations in beef meat (mg/kg)

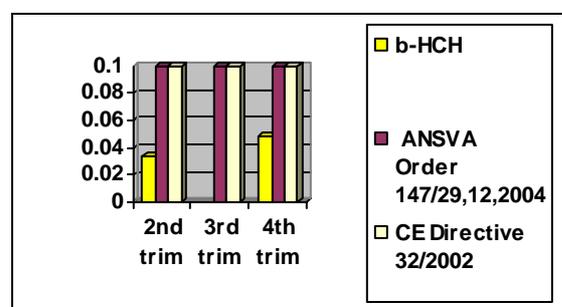
Specification	Analyzed parameter (ppm)			Reference guidelines
	$\alpha$ -HCH	$\beta$ -HCH	$\gamma$ - HCH	
Beef meat	0,2	0,1	1,0	ANSVA Order 147/29.12.2004
	0,2	0,1	0,02	CE Directive 32/2002

From the obtained data it can be observed that both medium (0.005 – 0.04 mg/kg) and maximum  $\alpha$ -HCH concentrations (0.067 – 0.072 mg/kg) are situated both in national and European guidelines levels (0.2 mg/kg) (figure 2).



**Figure 2.**  $\alpha$ -HCH concentrations in all three trimesters comparative to national and European guidelines

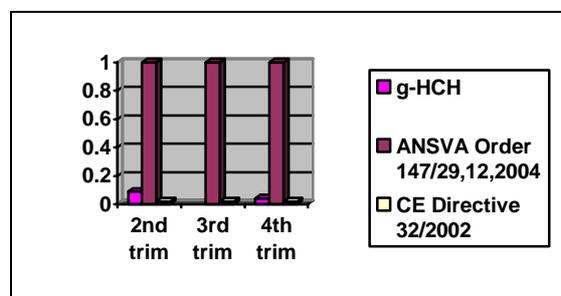
Regarding  $\beta$ -HCH, medium concentrations (0.034 – 0.049 mg/kg) are situated both in national and European guidelines levels (0.1 mg/kg) (figure 3).



**Figure 3.**  $\beta$ -HCH concentrations in all three trimesters comparative to national and European guidelines

However, maximum registered values (0.560mg/kg) exceed by 5.6 times both national and European guidelines.

Medium  $\gamma$ - HCH concentrations (0.088 – 0.042 mg/kg) do not exceed maximum national recommended (1 mg/kg), but exceed by 4.4, respectively 2.1 times Europeans guidelines (0.02 mg/kg) (figure 4).



**Figure 4.**  $\gamma$ -HCH concentrations in all three trimesters comparative to national and European guidelines

Maximum registered concentrations on this parameter (0.169 – 0.265 mg/kg) exceed Europeans guidelines by 8.45, respectively 13.25 times.

Although the obtained values are comparable to those obtained by other authors [5, 12, 13, 14], considering that food are the main pesticide source for man (excepting professional exposures), it is recommended forward monitoring of HCH residues, both in feed and animals meat, in order not to exceed acceptable daily intake -0.005 mg/HCH/kg body weight [1, 2, 15].

#### 4. Conclusions

1. The highest pesticide residues levels in feed were those of  $\gamma$ -HCH, which are situated, as medium values, in national guidelines values, but not the Europeans ones, which are exceeded by 14.5 respectively 18.8 times.
2. In beef meat, medium  $\alpha$  and  $\beta$ -HCH concentrations are situated both in national and European guidelines levels.
3. Medium  $\gamma$ -HCH concentrations in beef meat are situated in maximum national admitted limits, but exceed twice European standards.
4. By reason of HCH isomers persistence in environment, pesticide feed residues have to be surveyed in order not to become contamination sources for animals and its, on their turn, considering bioaccumulation and biomagnification properties of organochlorine pesticides, contamination sources for man.

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