

ORGANOCHLORINE INSECTICIDES RESIDUES IN RAW COW'S MILK, CREAM AND BUTTER FROM BENI-SUEF GOVERNORATE

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Received: 18 August 2016; Accepted: 30 August 2016

ABSTRACT

Organochlorine insecticides residues (α -HCH, β -HCH, Lindane, HCB, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor) were analyzed in 75 samples of raw milk, sour cream and cooking butter- collected from Beni-Suef Governorate- using gas chromatography with electron capture detector (GC-ECD). The results indicated that all the examined samples were accepted except three butter samples showed residue levels exceeded the Maximum Residues Levels (MRLs).

Key words: Organochlorine insecticides, milk, cream, butter, gas chromatography

INTRODUCTION

Milk is considered as an early complete food since it is a good source for protein, fat and major minerals. Also, milk is the main constituent of the daily diet, especially for vulnerable groups such infants, schoolage children and old age (Kampire *et al.*, 2011).

Milk and milk products are subjected to many persistent environmental contaminants such as organochlorine insecticides and their metabolites. Organochlorine insecticides are highly stable under normal environmental conditions, bio accumulate through food chain and readily excreted in the milk fat due to lipophilic property (Mukherjee and Gopal, 1993 and Aman and Bluthgen, 1997).

The environmental pollution with organochlorine insecticides has decreased due to banning of these compounds, however, several recent reports (Aksoy *et al.*, 2013; Ranganathan *et al.*, 2014; Kotinagu and Krishnaiah, 2015) indicate that pollution with organochlorines still present and may be of public and environmental health significance.

The contamination of milk and its products with organochlorine insecticides and their metabolites have been recorded in different countries. In Egypt,

although the use of organochlorine pesticides had been banned since 1985, these chemicals were found in milk and milk products from some localities (El-Talawy, 2010; Shaker and El-sharkawy, 2015).

Organochlorine insecticides are a source of several mysterious diseases through chronic toxicity and sub lethal exposure. These diseases include cancer, liver and kidney dysfunction, growth depression and neuritis (Schuh *et al.*, 2009; El Amine *et al.*, 2013 and Wilson *et al.*, 2014). Therefore, there is need for monitoring levels of OCP residues in milk and milk products so this work was carried out to investigate the extent of contamination of milk, cream and butter with 10 Organochlorine insecticides and to assess human exposure to OCPs through the consumption of these products.

MATERIALS AND METHOD

The method of Heck *et al.* (2007) was adopted.

A- Collection of samples:

A total of 75 random samples including raw cow's milk, sour cream and cooking butter (25 of each) were collected from different districts at Beni-Suef Governorate, Egypt, during the period from September 2014 to June 2015. and transported in cooling boxes containing ice bags to the laboratory of Animal Health Research Institute where they were immediately stored in a freezer at -20 °C until further analysis.

B- Sample extraction:

1) Milk samples: The samples were thawed, homogenized and mixed thoroughly. 50 ml of each

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sample centrifuged for 15 min, at 5000 rpm to obtain milk fat. Anhydrous sodium sulfate (25 g) was mixed with one gram milk fat in a 250 ml flask. To this mixture 100 ml of petroleum ether were added with vigorous shaking for 2 min. It was then allowed to settle down and the petroleum ether was filtered off into another flask through a glass funnel containing anhydrous sodium sulfate on top of glass wool to remove any traces of water. The filtered extract was evaporated to near dryness using a rotatory evaporator and concentrated to about 1 ml which was taken up in 3 ml of hexane and kept in a flask for the clean-up with 100 ml of hexane to extract organochlorine insecticides.

2) Cream samples:

100 ml of well mixed cream sample were centrifuged at 5000 rpm for 15 min. The upper layer composed of fat was removed and placed in a small clean beaker from which 1g of fat was weighed out for extraction as mentioned before.

2) Butter samples: 100 g of butter sample were warmed to about 50°C until fat separated and decanted through dry filter paper No. 10, then 1 g was weighed out for extraction as mentioned before.

C- Gas Chromatography analysis:

Agilent GC Model 6890 N equipped with an Ni⁶³-electron capture detector was used.

RESULTS

Table 1: Residues of organochlorine insecticides in milk samples (mg/kg)

Insecticide	Min. (mg/kg)	(mg/kg) Max.	Mean (mg/kg) ±SE	% of +ve results	Samples exceed MRL
α -HCH	0	0.000012	0.0000005 ± 0.0000005	4	0
β - HCH	0	0.0006	0.00006 ± 0.00003	20	0
γ -HCH (Lindane)	0	0.00006	0.000007 ± 0.0000034	16	0
HCB	0	0	0	0	0
Aldrin	0	0.00016	0.000052 ± 0.00001	68	0
Heptachlor	0	0.00022	0.00001112 ± 0.000009	24	0
Chlordane	0	0.00014	0.000012 ± 0.000006	28	0
total DDT	0	0.0005	0.000055 ± 0.00003	35	0
Endrine	0	0.00012	0.000009 ± 0.000006	10	0
Methoxychlor	0	0.001	0.00004 ± 0.00004	4	0

Table 2: Residues of organochlorine insecticides in cream samples (mg/kg)

Insecticide	Min. (mg/kg)	Max. (mg/kg)	Mean (mg/kg) \pm SE	% of +ve results	Samples exceed MRL
α -HCH	0	0.0000006	0.0024E-05 \pm 0.0024E-05	4	0
β -HCH	0	0.0002	0.000012 \pm 0.000008	12	0
γ -HCH	0	0	0	0	0
HCB	0	0.00002	0.00000096 \pm 0.0000008	8	0
Aldrin	0	0.00001	0.0000017 \pm 0.000000056	36	0
Heptachlor	0	0	0	0	0
Chlordane	0	0.00004	0.000002 \pm 0.0000016	12	0
total DDT	0	0.0004	0.00003 \pm 0.000017	52	0
Endrine	0	0.00016	0.00001 \pm 0.000006	28	0
Methoxychlor	0	0.00018	0.00001 \pm 0.000007	8	0

Table 3: Residues of organochlorine insecticides in butter samples (mg/kg)

Insecticide	Min. (mg/kg)	Max. (mg/kg)	Mean (mg/kg) \pm SE	% of +ve results	Samples exceed MRL
α -HCH	0	0	0	0	0
β -HCH	0	0.001	0.00004 \pm 0.00004	4	1
γ -HCH	0	0.07	0.0049 \pm 0.0034	8	2
HCB	0	0	0	0	0
Aldrin	0	0.00016	0.000007 \pm 0.000006	20	0
Heptachlor	0	0.0002	0.00004 \pm 0.000008	4	0
Chlordane	0	0.00016	0.000006 \pm 0.000008	16	0
total DDT	0	0.0022	0.0001 \pm 0.000087	36	0
Endrine	0	0.00008	0.000005 \pm 0.000003	16	0
Methoxychlor	0	0	0	0	0

Table 4: Calculated ADI and HQ for mean values of organochlorine insecticide residues in milk samples (mg/kg b.w)

Insecticide	ADI (mg / 70 kg person)	Calculated mean value (200 ml milk)	HQ
α -HCH	0.07	0.000000003	0.00000004
β - HCH	0.021	0.000000036	0.000017
γ -HCH	0.35	0.00000004	0.0000001
HCB	0.042	0	0
Aldrin	0.007	0.0000003	0.000045
Heptachlor	0.007	0.000000067	0.00001
Chlordane	0.035	0.00000007	0.000002
total DDT	0.7	0.0000003	0.00000047
Endrine	0.014	0.00000005	0.000004
Methoxychlor	7	0.00000024	0.000000034

Table 5: Calculated ADI and HQ for mean values of organochlorine insecticide residues in cream samples (mg/kg b.w)

Insecticide	ADI mg / 70 kg person	Calculated mean value (100 gm cream)	HQ
α -HCH	0.07	0.000000001	0.000000015
β - HCH	0.021	0.000000054	0.000026
γ -HCH	0.35	0	0
HCB	0.042	0.000000043	0.000001
Aldrin	0.007	0.000000076	0.00001
Heptachlor	0.007	0	0
Chlordane	0.035	0.00000009	0.0000025
total DDT	0.7	0.00000135	0.000002
Endrine	0.014	0.00000045	0.00003
Methoxychlor	7	0.00000045	0.00000006

Table 6: Calculated ADI and HQ for mean values of organochlorine insecticide residues in butter samples (mg/kg b.w)

Insecticide	ADI (mg / 70 kg person)	Calculated mean value (8 gm butter)	HQ
α -HCH	0.07	0	0
β -HCH	0.021	0.00000032	0.00002
γ -HCH	0.35	0.00004	0.0001
HCB	0.042	0	0
Aldrin	0.007	0.00000006	0.000009
Heptachlor	0.007	0.0000003	0.00004
Chlordane	0.035	0.00000006	0.0000018
total DDT	0.7	0.0000008	0.000001
Endrine	0.014	0.00000004	0.000003
Methoxychlor	7	0	0

DISCUSSION

From the results recorded in Table (1) it is cleared that 9 organochlorine insecticides were detected including α -HCH, β -HCH, γ -HCH, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor at percentages of 4, 20, 16, 68, 24, 28, 35, 10 and 4% respectively, and with mean values of 0.0000005 ± 0.0000005 , 0.00006 ± 0.00003 , 0.000007 ± 0.0000034 , 0.000052 ± 0.00001 , 0.00001112 ± 0.000009 , 0.000012 ± 0.000006 , 0.000055 ± 0.00003 , 0.000009 ± 0.000006 and 0.00004 ± 0.00004 (mg/kg) on fat basis, respectively, while HCB could not be detected. 21 (84%) samples were contaminated with one or more of the residues. Aldrin was the most frequently detected insecticide, while α -HCH and Methoxychlor had the least incidence.

The obtained data indicated the presence of organochlorine insecticides although stop use of such compounds since 1985. It may be due to their environmental persistence that induced by the Chlorine content of these insecticides which makes the molecules more resistant to enzymatic degradation by bacteria, insects, and mammals and this property has the effect of reducing biodegradability and prolonging the residence time of these compounds in the environment (Ruth Stringer and Paul Johnston, 2002). The carbon-chlorine bond is very stable towards hydrolysis and, the greater the number of chlorinesubstitutions and/or functional groups, the greater the resistance to biological and

photolyticdegradation. Chlorine attached to an aromatic (benzene) ring is more stable to hydrolysis than chlorine in aliphatic structures. As a result, organochlorine insecticides are typically ring structures with a chain or a branched chain framework (Joe Thornton, 2000) which prolonged their environmental persistence.

The presence of organochlorine insecticides at such low levels indicates the effectiveness of regulatory actions adopted for organochlorine insecticides in Egypt. However, the resistance of such compounds to breakdown leads to their presence in the environment. In the body of the animals, the metabolism and excretion of organochlorine insecticides residues are slow and finally deposited in the body fat or excreted in the milk.

Higher levels of organochlorine insecticide residues were obtained by Darko and Acquaah (2008); Salem *et al.* (2009); Kampire *et al.* (2011); Bulut *et al.* (2011); Avancini *et al.* (2012) and Gutterrez *et al.* (2013), while local studies in Egypt applied by Ahmed and Zaki (2009) detected α -HCH, β -HCH, γ -HCH, Heptachlor, Aldrin, Chlordane, Endrin and DDT at mean values of 0.003 ± 0.0001 , 0.003 ± 0.0002 , 0.002 ± 0.000005 , 0.003 ± 0.0001 , 0.004 ± 0.000009 , 0.001 ± 0.0001 , 0.0003 ± 0.000005 and 0.002 ± 0.0007 mg/kg, respectively. While Abd El-Aziz (2005) detected only DDT at mean value of 0.0027 ± 0.0001 mg/kg.

On contrast, lower levels of some organochlorine insecticide residues were obtained by Darko and

Acquaah (2008); Bulut *et al.* (2011) and Abd El-Aziz (2005) who could not detect Lindane or Heptachlor. The low levels or undetected residues may be attributed to unuse of these insecticides or their presence was at levels less than the limit of detection. For quantitative risk assessment we calculated Hazard Quotient (HQ) (the risk to a human receptor from being exposed to a chemical via a single pathway) CCME (1996)

$$\text{HQ} = \frac{\text{Estimated Dose } (\mu\text{g/kg/day})}{\text{Tolerable Daily Intake } (\mu\text{g/kg/day})}$$

and the mean daily intake and from Table (4) we noticed that HQ and the calculated mean values (mg /70 kg person) from consumption of 200 ml milk/day for α -HCH, β -HCH, γ -HCH, HCB, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor were 0.00000004 and 0.000000003, 0.000017 and 0.00000036, 0.0000001 and 0.00000004, 0 and 0, 0.000045 and 0.0000003, 0.00001 and 0.000000067, 0.000002 and 0.00000007, 0.00000047 and 0.0000003, 0.000004 and 0.00000005 and 0.000000034 and 0.00000024 and we noticed that none of the obtained results exceeded 0.2 for HQ that indicated safety results according to CCME (1996) and also none of the obtained results exceeded the Acceptable daily Intake (ADI) of 0.07, 0.021, 0.35, 0.042, 0.007, 0.007, 0.035, 0.7, 0.014 and 7 mg/70 kg person for α -HCH, β -HCH, γ -HCH, HCB, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor, respectively, recommended by Codex Alimentarius Commission (2014).

Comparing our results with the Maximum Residues Levels (MRLs) adopted by Codex Alimentarius Commission (2014) that reported MRLs of α -HCH, β -HCH, γ -HCH, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor as 0.008, 0.0006, 0.01, 0.006, 0.006, 0.002, 0.02, 0.0008 and 0.005 mg/kg, respectively, it cleared that all the examined samples were below the MRLs and we found that these results were nearly similar to those recorded by Darko and Acquaah (2008) for (Aldrin and Lindane), Salem *et al.* (2009) for aldrine and Gutierrez *et al.* (2013) for α -HCH, β -HCH, γ -HCH, Heptachlor, Aldrin, Endrine and DDT in Mexico.

On contrast, other studies detected some organochlorine insecticide residues at levels exceeded the recommended MRL for one or more of the examined residues that may be due to illegal usage of such organochlorine insecticides during these periods or their usage were prohibited just from few years before the time of the study or excessive usage in past which with the bio accumulation and persistent nature of these

chemicals lead to presence of one or more of the examined residues at high level.

From the results recorded in Table (2) it cleared that eight organochlorine insecticides were detected in the examined cream samples which were α -HCH, β -HCH, HCB, Aldrin, Chlordane, total DDT, Endrin and Methoxychlor at percentages of 4, 12, 8, 36, 12, 52, 28 and 8%, respectively, and with mean values of $0.0024\text{E-}05 \pm 0.0024\text{E-}05$, 0.000012 ± 0.000008 , 0.00000096 ± 0.0000008 , 0.0000017 ± 0.00000056 , 0.000002 ± 0.0000016 , 0.00003 ± 0.000017 , 0.00001 ± 0.000006 and 0.00001 ± 0.000007 mg/kg, respectively. 19 (76%) samples were contaminated with one or more of these residues.

Comparing the obtained results with MRLs adopted by Codex Alimentarius Commission (2014), we found that all the detected values were below the recommended MRLs and these findings were nearly similar to the results obtained by Ranganathan Y. *et al.* (2014).

On contrast, other studies detected some organochlorine insecticide residues at levels exceeded the recommended MRL for one or more of the examined residues. In Uzbekistan Muntean *et al.* (2003) detected α -HCH and β -HCH at mean values of 0.03 and 0.045 mg/kg which exceeded the MRL for each component. In Romania Chiş *et al.* (2008) found that the HCH group (β and γ isomers) appeared in all samples except α -HCH isomer appeared in a single test, while Endosulphane, Endrin and DDT appeared randomly. All detected values of organochlorine insecticides were below MRL except one sample for Endrin (0.0966 ppm). In Gharbia, Egypt, El-Talawy (2010) found that 40% of the examined cream samples were contaminated with Endrin with average concentration of 102.07 ± 12.42 ppb and about 13.5 % of the examined sour cream samples were unaccepted as the residues exceeded the MRLs recommended by Codex Alimentarius Commission (2004).

The results recorded in Table (5) revealed that HQ and the calculated mean values from consumption of 100 g cream /day for α -HCH, β -HCH, γ -HCH, HCB, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor were 0.000000015 and 0.000000001, 0.000026 and 0.00000054, 0 and 0, 0.000001 and 0.000000043, 0.00001 and 0.000000076, 0 and 0, 0.0000025 and 0.00000009, 0.000002 and 0.00000135, 0.00003 and 0.00000045 & 0.00000006 and 0.00000045, respectively, and it is cleared that none of the obtained results exceeded 0.2 for HQ that indicated safety results according to CCME(1996) and also none of the obtained results exceeded the ADI of 0.07, 0.021, 0.35, 0.042, 0.007, 0.007, 0.035, 0.7, 0.014 and 7 mg/70 kg person for

α -HCH, β -HCH, γ -HCH, HCB, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor, respectively, as recommended by Codex Alimentarius Commission (2014).

From the results recorded in Table (3), it cleared that seven organochlorine insecticides were detected in the examined butter samples including β -HCH, γ -HCH, Aldrin, Heptachlor, Chlordane, total DDT and Endrin at percentages of 4, 8, 20, 4, 16, 36 and 16%, respectively, and with mean values of 0.00004 ± 0.00004 , 0.0049 ± 0.0034 , 0.000007 ± 0.000006 , 0.00004 ± 0.000008 , 0.000008 ± 0.000006 , 0.0001 ± 0.000087 and 0.000005 ± 0.000003 mg/kg, respectively. 10 (40%) samples were contaminated with one or more of the residues. Higher levels of organochlorine insecticide residues were obtained by Waliszewski *et al.* (1997); Amr (1999); Pandit *et al.* (2002); Waliszewski *et al.* (2003); Abd El-Aziz (2005); Witczak and Abdel-Gawad (2014). In Turkey Aksoy *et al.* (2013) detected β -HCH in 3 of 88 samples at a mean level of 0.001 mg/kg. Prachar *et al.* (1995) found that the examined butter samples from Slovakia were contaminated with HCB and DDT at mean values of 0.004 and 0.01 mg/kg, respectively. Salem *et al.* (2009) in Jordan found DDT, β -HCH at incidence of 8 and 16%, respectively, and at mean values of 0.009 and 0.019 mg/kg, respectively.

On contrast, lower levels of some organochlorine insecticide residues were obtained by Aksoy *et al.* (2013) in Turkey who could not detect DDT, α -HCH, Lindane, Aldrin or HCB and Salem *et al.* (2009) in Jordan also could not detect α -HCH, γ -HCH, Aldrin, Heptachlor or HCB and this may be attributed to presence of these residues at very low values below the detection limits.

For quantitative risk assessment, the HQ and the mean daily intake from consumption of 8 g butter/day in Table (6) for α -HCH, β -HCH, γ -HCH, HCB, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor were 0 and 0, 0.00002 and 0.00000032, 0.0001 and 0.00004, 0 and 0, 0.000009 and 0.00000006, 0.00004 and 0.0000003, 0.0000018 and 0.00000006, 0.000001 and 0.0000008, 0.000003 and 0.00000004 & 0 and 0, respectively, It is cleared that none of the obtained results exceeded 0.2 for HQ that indicated safety results according to CCME (1996) and also none of the obtained results exceeded the ADI of 0.07, 0.021, 0.35, 0.042, 0.007, 0.007, 0.035, 0.7, 0.014 and 7 mg/70 kg person for α -HCH, β -HCH, γ -HCH, HCB, Aldrin, Heptachlor, Chlordane, total DDT, Endrin and Methoxychlor respectively as recommended by Codex Alimentarius Commission (2014).

By comparing the obtained results with the MRLs adopted by Codex Alimentarius Commission (2014)

we found that all the detected values were below the recommended MRLs except γ -HCH exceeded the MRL in two samples and β -HCH in one sample that may be attributed to persistent nature and bio accumulation of these compounds in the fatty foods (Aman and Bluthgen, 1997).

From the public health point of view, we observed that despite the low percentage 4% (3/75) of samples exceeding the MRLs in this study, these compounds represent a potential risk to human health because of their accumulation properties in human fat tissue and presence of different residues which may be synergize together and cause health hazards to human beings. The overall results from this study showed that milk and dairy products are an important route for OCPs since they contained high fat content and consumption of these products contribute in high degree to human exposure to organochlorine insecticides.

In our study there are three butter samples contained α -HCH and γ -HCH above the recommended MRL that is considered highly dangerous because HCH isomers were considered as liver tumors producer according to Agency for Toxic Substances and Disease Registry (ATSDR) (1997) and U.S. Environmental Protection Agency (1997).

Periodical surveys and studies for detection of organochlorine insecticides to estimate the state of food and environmental contamination with such compounds are recommended with Application of prompt regulatory actions to prevent possible illegal use of organochlorine insecticides in Egypt.

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بقايا بعض المبيدات الحشرية في اللبن الخام والقشدة والزبد في محافظة بني سويف

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تم تجميع ٧٥ عينة (٢٥ من كلاً من اللبن البقري الخام والقشدة الفلاحى والزبد الفلاحى) من محافظة بني سويف وتم إستخلاصها وتحليلها بواسطة جهاز الكروماتوجرافى الغازي وذلك لقياس متبقيات ١٠ مبيدات حشرية كلورونية، وقد وجد أن ٤ ، ٢٠ ، ١٦ ، ٢٤ ، ٦٨ ، ٢٨ ، ٣٥ ، ١٠% من عينات اللبن ملوثة بمتبقيات ٩ مبيدات كلورونية تشمل ألفا-هكسا كلورو سيكلوهكسان، بيتا-هكسا كلورو سيكلوهكسان، جاما-هكسا كلورو سيكلوهكسان، ألدرين، هيبتاكلور، كلوردان، د.د.ت، إندرين وميثوكسي كلور بمتوسطات 0.000005 ± 0.000005 ، 0.000006 ± 0.000003 ، 0.000007 ± 0.000003 ، 0.000005 ± 0.000002 ، 0.000009 ± 0.000001 ، 0.000011 ± 0.000009 ، 0.000012 ± 0.000006 ، 0.000005 ± 0.000003 ، 0.000006 ± 0.000003 ، 0.000009 ± 0.000001 ، 0.000006 ± 0.000004 و 0.000004 ± 0.000004 مجم /كجم على التوالي وفي عينات القشدة تم إكتشاف ٨ متبقيات مبيدات كلورونية تشمل ألفا-هكسا كلورو سيكلوهكسان، بيتا-هكسا كلورو سيكلوهكسان، هكساكلوروبنزين، ألدرين، كلوردان، د.د.ت، إندرين وميثوكسي كلور بنسب تواجد ٤ ، ١٢ ، ٨ ، ٣٦ ، ١٢ ، ٥٢ ، ٢٨ ، ٨% على الترتيب و بمتوسطات 0.000002 ± 0.000002 ، 0.000008 ± 0.000008 ، 0.000009 ± 0.000008 ، 0.000017 ± 0.000006 ، 0.000002 ± 0.000002 ، 0.000016 ± 0.000003 ، 0.000017 ± 0.000001 ، 0.000006 ± 0.000001 و 0.000007 ± 0.000001 مجم /كجم على التوالي وبمتوسطات ٤ ، ٤ ، ٤ ، ٤ ، ٢٠ ، ٤ ، ١٦ ، ٣٦ و ١٦% على التوالي و بمتوسطات 0.000004 ± 0.000004 ، 0.000004 ± 0.000004 ، 0.000009 ± 0.000003 ، 0.000007 ± 0.000006 ، 0.000004 ± 0.000008 ، 0.000008 ± 0.000008 ، 0.000006 ± 0.000001 ، 0.000007 ± 0.000001 و 0.000003 ± 0.000005 مجم /كجم على التوالي. وقد وجد ان نسب المتبقيات التي تم الحصول عليها كانت أقل من الحدود المسموح بها من قبل منظمة الصحة العالمية ومنظمة الأغذية والزراعة إلا في ثلاثة عينات من الزبد إثنان منهما إحتوتا جاما - هكسا كلورو سيكلوهكسان وعينة واحدة إحتوت بيتا- هكسا كلورو سيكلوهكسان بنسب أعلى من المسموح بها.