CONTAMINATION OF FOODSTUFFS AND HUMAN FATTY TISSUE BY PESTICIDE RESIDUES IN YUGOSLAVIA IN 1969—1971

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A survey is given of the studies performed on pesticide residues in food and human fatty tissue in Yugoslavia. Out of 800 samples analysed in Yugoslav laboratories during the period of 1969—1971, 578 were food products and 222 human fatty tissue samples.

The findings can be summarized as follows: (1) Food of animal origin was contaminated by organochlorine insecticides. DDT residues in milk ranged between 0.0035 to 0.025 ppm. (2) The residue levels of OC-insecticides in animal fat varied considerably in different Yugoslav laboratories, the mean value for DDT being 0.404 ppm. (3) OC residues in fruits and vegetables were found within the tolerance limits. (4) OC insecticide residues were found in all samples of human fatty tissue, with significant differences between geographical regions, as well as between age groups. (5) Organophosphorus insecticide residues were found in 19 percent of samples of plant origin. All values were within tolerance limits. (6) Arsenic was found in 52 percent of apple samples.

A more serious investigation of the contamination of foodstuffs by pesticides was started about the year 1948 (26). After in the U.S.A. the regulations governing tolerance levels of pesticide residues in foodstuffs had been made, the investigation of pesticide residues in foodstuffs was undertaken on a larger scale since 1961 (19). According to the report from the Laboratory for Pesticides in Florida (14), during the period of 1963—1966 the number of samples containing excessive amounts of pesticides increased from 4.9 percent in 1963 to 10.0 percent in 1966. Canadian reports from 1966 (22) state that 6.3 percent out of the total of 3167 samples of domestic foodstuffs were unacceptable with regard to the content of pesticides, while out of 461 samples of imported food 3.0 percent did not meet tolerance requirements. *Egan* et al. (12) and *Hammence* (16) investigated pesticide residues in meat, milk products and

vegetables marketed in Great Britain. Except in sheep tallow, in most of the other foods very small amounts of organochlorine insecticide residues were found.

In U.S.S.R. Stenberg et al. (13) and Baratov et al. (7) reported on the results of the investigation of foodstuffs for pesticide residues. DDT residues were rather high. In 1967 Engst et al. (13) reported on the results of the investigation of milk and butter in East Germany where 14 percent samples of milk and 34 percent samples of butter contained pesticide residues. Denes (11) investigated DDT residues and other organochlorine insecticides in foods marketed in Hungary. On the average the amount of DDT residues was above the tolerance limit of 1.0 ppm.

Although the investigation of pesticide residues in foods has been conducted practically since 1962 in the laboratories for pesticides at the Institute of Public Health in Zagreb and Beograd, a relatively small amount of data from this field has been published.

Adamović et al. (2) conducted investigation of pesticide residues in powdered milk, cow milk and mother milk. All the samples contained one or more organochlorine insecticides. Adamović et al. (3) proved that the amounts of DDT residues and its metabolites had been increasing since 1966, until they reached five times higher values in 1969.

Bačić (5) has conducted a small scale investigation of residues of organochlorine insecticides in milk and butter supplied by Yugoslav dairy plants. In most cases the samples contained larger amounts of pesticide residues than allowed by our legislation on the maximum tolerance levels of pesticide residues in food (21).

The use of pesticides, DDT in the first place, and other organo-chlorine insecticides, resulted inevitably in their accumulation in humans, mostly in their fatty tissue. In U.S.A. as early as in 1942 *Hayes* et al. (17) investigated 10 samples of human fatty tissue, but failed to discover storage of organochlorine insecticides. In 1950 *Lang* et al. (29) found DDT residues in 75 samples of human fatty tissue. Subsequent work in this field has shown that the amounts of DDT residues in the American population have been increasing constantly (till the year 1961 — Edits. comment). In 1958 and 1959 *Maier-Bode* (20) analysed samples of fatty tissue and discovered that an average German had stored less DDT than an American.

The largest amount of the accumulated DDT was registered in the population of India (10). The levels registered in Israel (25) and Roumania (15), though, differ from those normally found elsewhere.

The first investigation of DDT and other organochlorine insecticide residues in human tissue in this country were conducted by *Adamović* et al. (1, 2, 3, 4). All the investigations in this field have been isolated and did not yield a picture of the contamination of food and human fatty tissue that would pertain to the whole of Yugoslavia. Having this in mind and wishing to join the efforts made by individual laboratories in the same period we have set as our task to perform the following studies:

- 1) to establish which pesticides and in what concentrations appear as residues on or in food that is commonly consumed by the Yugoslav population;
- 2) to evaluate the level of contamination of human fatty tisuse by pesticide residues.

MATERIAL AND METHODS

With the purpose of examining food and human fatty tissue for pesticide content 800 samples were analysed during 1969—1971, of which 70 percent were food samples and 30 percent human fat tissue. About half the samples were analysed in 1970, and the other half in 1971. The total number of analysed samples of human fatty tissue was 222. They were taken from abdomen during surgical operations on people who suffered from diseases the nature of which, it was assumed, would not affect the changes in the process of accumulation of insecticides. No preservers were used. The samples were accompanied by the data on sex, age, occupation, domicile and kind of disease that led to the operation.

Residues of organochlorine, organophosphorus insecticides and arsenic were determined by standard methods (7, 8).

Among organochlorine insecticides the following were determined: Alpha and gama HCD, pp'DDE, pp'DDD, pp'DDT, op'DDT and dieldrin. Terminal fraction of each sample in which organochlorine insecticides were determined was examined by gas and thin layer chromatography. The results applied only to those insecticides or their degradation products which were identified and proved by both methods.

The analysis for organophosphorus insecticides was limited by the equipment which was not uniform. Therefore, vegetable, fruit and bread samples were analysed only for the presence of organophosphorus insecticides which were determined by the total phosphorus content (17). In some cases both identification and determination was performed.

Arsenic in apples was determined spectrophotometrically (7).

THE RESULTS AND DISCUSSION

Residues of organochlorine insecticides in food

During the 1969—1971 a total of 139 milk samples from the Yugoslav market were analysed in our laboratories. The results of the investigation of organochlorine insecticide residues in food of animal origin, bread and fruits for the above period for Yugoslavia are shown in Table 1 indicating the mean values of organochlorine insecticide content.

The mean values of organochlorine insecticides in the food of animal origin show that in milk and animal fat samples tolerance limits were exceeded with respect to the recommendations of WHO and FAO. Gama HCH in milk was found to have a 20 times higher concentration than the maximum tolerance. The total amount of DDT in milk was below the tolerance limit (0.005 ppm), but the established values exceeded three times the tolerance limit according to our regulations. Judging by the amount of organochlorine insecticide residues in milk determined by individual laboratories, it is evident that the amount of the total DDT found in milk samples in almost all laboratories, with the exception of Zagreb, exceeded the maximum tolerance level.

A higher amount of total DDT in milk indicates that animal feed and soil were contaminated by organochlorine insecticides.

The residues of organochlorine insecticides in animal fatty tissue (swine and cattle) are shown in Table 1. The samples of swine fat contained alpha and gama HCH, para of DDT form and dieldrin in measurable amounts. The values for total DDT in the Belgrade and Skopje laboratories were 6—8 times higher than the values found in other laboratories, what can be accounted for by differences in the feeding of swine and cattle.

Animal feed produced in the country is not subject to any control of pesticide content and no tolerance limits have been prescribed for it.

Obviously urgent steps have to be taken to establish maximum tolerance limits for organochlorine and organophosphorus insecticides in animal feed, and to introduce a stricter control.

The data on organochlorine insecticides in vegetables are presented in Table 2. The largest average amount of organochlorine insecticides was found in the samples of potato and carrot. The mean value of the total DDT in potato was 0.5374 ppm and in carrot 0.1701 ppm: i.e. near the maximum tolerance (1.0 ppm), they added considerably to the mean value and pp'DDT can be accounted for primarily by the potato and carrot samples, taken from private farmers selling on the markets in Sarajevo. With the exception of these samples, the values of pp'DDE, pp'DDT and total DDT go for potato on the average far below the tolerance limit.

The amounts of organochlorine insecticide residues in fruits are shown in Table 1. Almost all average values for Yugoslavia are below maximum tolerance limits.

In grape samples the highest amount of total DDT was found in the laboratories in Skopje and Sarajevo, and the lowest in the laboratory in Belgrade. The highest values of total DDT were found in apple samples in the laboratory in Sarajevo. Although these findings were below the maximum tolerance (1,0 ppm), they added considerably to the mean value of the total DDT in apples for Yugoslavia.

Table 1
Organochlorine insecticide residues in foodstuffs of animal origin, bread and fruits in Yugoslavia in 1969—1971.

					Insecti	Insecticides in ppm	bm		
Food		alfa HCH	вата НСН	pp' DDE	pp' DDD	pp' DDT	op' DDT	Total DDT	Dieldrin
Milk	n × S	139 0,0058 0,0125	139 0,0114 0,0140	139 0,0045 0,0063	139 0,0019 0,0037	139 0,0086 0,0143	0,0020 0,0040	0,0170 0,0198	139 0,0026 0,0028
Lard	G × S	57 0,0243 0,0455	57 0,0514 0,1220	57 0,1290 0,1533	57 0,0371 0,0653	57 0,2043 0,3204	43 0,0118 0,0187	57 0,4041 0,4640	57 0,0060 0,0122
Tallow	SMB	57 0,0217 0,0449	57 0,0870 0,3350	57 0,0996 0,1156	57 0,0358 0,0239	57 0,1847 0,3905	43 0,0034 0,0166	57 0,3529 0,5310	57 0,0417 0,1315
Bread	u × ∞	0,0000	58 0,0096 0,0174	58 0,0021 0,0036	58 0,0044 0,0272	58 0,0142 0,0359	49 0,0059 0,0099	58 0,0252 0,0527	58 0,0000 0,0000
Cherries	S KI D	25 0,0000 0,0000	25 0,0006 0,0017	25 0,0008 0,0016	0,0000 0,0000	25 0,0144 0,0626	0,0007 0,0025	25 0,0158 0,0627	25 0,0000 0,0000
Apricots	S XI II	22 0,0002 0,0007	22 0,0011 0,0023	0,0007 0,0013	0,0000 0,0000	22 0,0001 0,0000	0,0000 0,0000	22 0,0008 0,0016	22 0,0000 0,0000
Apples	Z × S	0,0002 0,0012	25 0,0561 0,2540	25 0,0370 0,1565	25 0,0008 0,0027	25 0,0238 0,0972	0,0008 0,0017	25 0,0649 0,2711	25 0,0000 0,0000
Grapes	u × v	0,0000 0,0000	0,0018 0,0022	0,0038 0,0049	0,0005 0,0008	21 0,0395 0,0702	0,0053 0,0063	0,0475 0,0784	21 0,0000 0,0000
		n — Num x — Mear	Number of samples Mean value	mples					

Table 2
Organochlorine insecticide residues in vegetables in Yugoslavia, 1969—1971.

					Insec	Insecticides in	mdd	20,	
Food		alfa HCH	gama HCH	pp' DDE	pp' DDD	pp' DDT	op' DDT	Total DDT	Dieldrin
Potato	ά×Χ	36 0,0007 0,0034	36 0,0190 0,0727	36 0,2672 1,5600	36 0,0001 0,0004	36 0,2387 1,3928	30 0,0015 0,0021	36 0,5374 3,1330	36 0,0003 0,0016
Sarrot	ti ix o	32 0,0041 0,0085	32 0,1078 0,3350	32 0,0635 0,2247	32 0,0250 0,1349	32 0,0691 0,2477	26 0,0037 0,0051	32 0,1701 0,5916	32 0,0013 0,0053
Onion	Z IX S	21 0,0000 0,0000	21 0,0087 0,0236	21 0,0052 0,0068	21 0,0001 0,0002	0,0043 0,0037	0,0045 0,0037	0,0132 0,0143	21 0,0002 0,0011
Cabbage	n ix S	0,0003 0,0012	0,0097 0,0203	0,0014 0,0021	0,0001 0,0003	22 0,0098 0,0290	0,0032 0,0073	22 0,0135 0,0371	0,0000 0,0000
Spinac h	S × n	8 0,0026 0,0038	0,0451 0,0504	8 0,0083 0,0075	8 0,0119 0,0296	0,1285 0,1133	8 0,0345 0,0427	0,1847 0,1587	8 0,0001 0,0003
Lettuce	N M I	0,0014 0,0023	7 0,0080 0,0096	0,0143 0,0109	0,0000	0,0099 0,0081	7 0,0084 0,0074	0,0337 0,0233	7 0,0000 0,0000
	The state of the second								

n — Number of samples \bar{x} — Mean value S — Standard deviation

Organophosphorus insecticides in food

The food examined for the presence of organophosphorus insecticides by determining total phosphorus is shown in Table 3. Out of the total of 232 samples of vegetables, fruits, bread and oil, 44 samples were found

to contain phosphorus.

In the laboratories in Belgrade and Zagreb individual organophosphorus insecticides (Sevin, Lebaycid, Zolone, Gusation, Fosdrin, Diazinon, Dimetron) in vegetables and fruits and edible oil are below tolerance limits recommended by WHO and FAO, as well as by the Yugoslav regulation on maximum amounts of pesticide residues in food.

Arsenic residues in apples

Table 4 shows findings of arsenic residues in apples. Out of 25 apple samples, 13 contained arsenic $(52^{\circ}/_{\circ})$.

Storage of organochlorine insecticides in human fatty tissue

The findings of organochlorine insecticide residues in fatty tissue of the Yugoslav population are shown in Tables 5—7.

The number of samples was approximately proportionate to the population in the areas around the research laboratories.

The results of analyses performed in the laboratories are shown in Table 5, by sex in Table 6, and by age in Table 7.

Regional similarities and differences displayed by the data furnished by the laboratories indicate that:

- a) Alpha HCH was found in exceptionally small amounts in fatty tissue in the population of Croatia, and Bosnia and Herzegovina; gama HCH is relatively uniform except in the findings of the Zagreb laboratory.
- b) Accumulated DDT and its derivatives display a heterogenous picture.

The lowest level (2.22 ppm) of the total DDT was found in Croatia, while other findings range from 4.96 ppm in Bosnia and Herzegovina to 13.45 ppm in Macedonia.

The total DDT value found for the whole of Yugoslavia is 7.06 ppm.

c) Dieldrin level is considerably lower in this country than in the population of Western Europe, with a markedly low level in the population of Croatia and Bosnia and Herzegovina.

Table 6 shows the distribution of accumulation of organochlorine insecticides in fatty tissue by sex.

The level of total DDT in women (7.21 ppm) is not significantly higher than in men (6.92 ppm).

Table 7 shows the results of the analysis by sex and age.

Total DDT in both sexes, with the exception of the group of 10—19 years of age, is maintained at the level of 5.8—10.4 ppm.

Table 3
Organophosphorus insecticide residues in food in Yugoslavia, 1969—1971.

			La	boratories		
		Beograd	Zagreb	Skoplje	Sarajevo	Ljubljana
Olive oil —	n	7	6	0	0	0
Plant Oil	a	0	6	0	0	0
rant on	b	7	0	0	0	0
	n	13	6	3	7	3
Carrot	a	0	0	0	1	0
	b	13	6	3	6	0
	n	14	6	3	9	4
Potato	a	0	0	0	2 8	0
	b	14	6	3	8	4
0	n	10	6	2	3 1 2	-
Onions	a b	0 10	6	2	1	-
-	U	10	0		2	
Cabbaga	n	5	6	3 0 3	5 2 3	3
Cabbage	a	0 5	6	0	2	0
	U	J	0	.)	3	3
0 ' 1	n	5	Annahous	-	3 2 1	
Spinach	a	0 5	-		2	
	U				1	
Lettuce	n	5		_	2 2 0	
Lettuce	b	0 5			0	-
				and the Administration of the Administration		
C1	n	10	6	2	4	3
Cherries	a	2 8	0 6	0 2	0	1 2
	D	0	0		4	2
	n	10	6		3	3
Apricots	a	10	6 3 3		0	0
	b	0	3	_	3	0
	n	10	6	2	4	3
Apples	a	0	4 2	0 2	1	3 2 1
	b	10	2	2	3	1
0	n	10	6	2	3	
Grapes	a	5 5	0	2 0 2	0	
	b	5	6	2	3	-

n — Number of samples

a — Contains organophosphorus insecticides (number)

b — No organophosphorus insecticides (number)

Table 4
Arsenic residues in apples, 1969—1971.

	As calc	ulated into lead a	rsenate
Laboratories	Number of samples	Contain As	Mean arith.
Beograd	10	10	6,600
Zagreb	6	_	
Sarajevo	4	1	0,670
Skopje	2	2	13,70
Ljubljana	3		

Age group of 0—9 years has a considerably high average level of 8.01 ppm of total DDT, which is contrary to the data in literature.

Differences between the first two age groups are presumably due to a different number of samples within the groups and between them.

CONCLUSIONS

The investigation of the contamination of food and human fatty tissue by pesticide residues in Yugoslavia in the 1969—1971 period yielded the following results:

- 1) Food of animal origin, primarily milk, is contaminated by organochlorine insecticides. Residues of total DDT in milk samples ranged from 0.0035 ppm to 0.0250 ppm.
- 2) Animal fatty tissue used in nutrition is also contaminated by all investigated organochlorine insecticides in measurable amounts. The values of total DDT in lard samples greatly varied from laboratory to laboratory, and in Skopje and Belgrade they were 6—8 times higher than those found in the samples analysed in Zagreb. The average value of total DDT in lard in Yugoslavia is 0.4041 ppm.
- 3) In the foods of plant origin (fruits and vegetables) the values were within the tolerance limits.
- 4) 19 percent fruit, vegetable and oil samples were proved to contain organophosphorus insecticides. The found values were below maximum tolerance.
 - 5) Arsenic was discovered in $52^{\circ}/_{0}$ of apple samples.
- 6) Organochlorine insecticide residues were found in all samples of human fatty tissue. Differences by territories were significant, while those by sex were not. Values by age displayed significant variations.

Organochlorine insecticide residues in human fatty tissue in Yugoslavia, 1970 and 1971. Table 5

					Insect	Insecticides in	mdd			
Laboratories		alfa HCH	gama HCH	pp' DDE	pp' DDD	pp' DDT	op' DDT	Total DDT	Dieldrin	
All laboratories	# IX O	0,0123 0,0603	0,1199 0,2280	222 4,5492 4,4633	161 0,0926 0,2202	222 1,8395 2,3065	0,0596 0,1671	222 7,0644 6,7453	222 0,0321 0,0786	(4)
Ljubljana	E IX W	25 0,0324 0,0511	25 0,0940 0,0813	25 3,9940 2,2533	25 0,0308 0,0498	25 2,7993 1,6760	25 0,0537 0,1380	25 7,4212 3,8987	25 0,0859 0,0821	100
Zagreb	n ix o	50 0,0019 0,0137	50 1,2463 0,9871	50 0,0123 0,0348	50 0,0144 0,0549	50 0,7999 0,6270	50	50 2,2273 1,6420	50 0,0026 0,0139	
Sarajevo	n ix Q	65 0,0005 0,0041	65 0,0868 0,1357	65 3,8947 3,2093	65 0,1395 0,2660	65 0,3108 0,3422	65	65 4,9624 3,8536	65 0,0027 0,0148	
Beograd	SXIB	61 0,0107 0,0564	0,0904 0,2211	6,4562 4,1593		61 3,6400 3,0838	61 0,1000 0,2260	61 10,9259 7,5413	61 0,0629 0,1215	
Skopje	d ix S	21 0,0293 0,1276	0,3631 0,4030	21 9,5607 7,5330	21 0,2126 0,3256	2,6739 1,7370	21 0,1337 0,1906	21 13,4462 9,5900	21 0,0395 0,0478	
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n — Number of samples x̄ — Mean value S — Standard deviation — — Not found

Table 6
Organochlorine insecticide residues in human fat by sex and kind of insecticides in Yugoslavia, 1970 and 1971.

)					
					Insec	Insecticides in	mdd		
Sex		alfa HCH	gama HCH	pp' DDE	pp' DDD	pp' DDT	op' DDT	Total DDT	Dieldrin
Total	n ix S	0,0123 0,0604	0,1199 0,2280	222 4,5492 4,4633	161 0,0926 0,2202	222 1,8395 2,3065	0,0596 0,1671	222 7,0644 6,7453	222 0,0321 0,0886
Male	d ix Q	78 0,0081 0,0304	78 0,1211 0,2061	111 4,3082 5,1030	84 0,0790 0,2095	111 1,7451 2,4839	78 0,0490 0,1328	111 6,9191 7,6681	0,0328 0,0856
Female	C IX S	94 0,0157 0,0767	99 0,1190 0,2439	4,5903 3,7147	77 0,1085 0,2303	111 1,9339 2,1111	94 0,0684 0,1905	7,2097 5,6745	0,0313 0,0709

Table 7
Organochlorine residues in human fat with respect to sex and age, 1970 and 1971 in Yugoslavia (values expressed in ppm)

					Age	e group			
Insecticides		6-0	10—19	20—29	30—39	40—49	50—59	69-09	above 70
Alpha HCH	Total Male Female	0,0119	0,0033	0,0266 0,0105 0,0444	0,0009	0,0196 0,0057 0,0300	0,0003	0,0220 0,0184 0,0250	0,0099
Сата НСН	Total Male Female	0,1711 0,2008 0,0560	0,0457	0,0963 0,0723 0,1229	0,0723 0,0877 0,0624	0,1769 0,8790 0,2511	0,0985 0,0570 0,1249	0,1492 0,1799 0,1228	0,1168 0,1660 0,0552
pp' DDE	Total Male Female	5,4265 6,5373 1,8162	1,7220 0,2900 1,8811	4,6302 5,0907 4,0859	3,8598 3,5741 4,1113	4,5386 3,5087 5,8627	5,3241 5,5874 5,1595	4,4077 3,9453 4,9568	6,3145 7,4583 4,9420
pp' DDD	Total Male Female	0,1865 0,2328 0,0125	0,1361	0,0689 0,0055 0,1390	0,0778 0,0818 0,0743	0,0854 0,0378 0,1683	0,0678 0,1483 0,0547	0,1021 0,0647 0,1423	0,0700
pp' DDT	Total Male Female	0,0739	0,0056	0,0335 0,0136 0,0556	0,0104 0,0107 0,0102	0,1477 0,0435 0,2259	0,0844 0,0857 0,0836	0,0347 0,0725 0,0022	0,0239 0,0428 0,0002
Total DDT	Total Male Female	8,0582 9,5489 3,2132	3,5766 0,5430 3,9137	6,9255 7,3422 6,4330	5,7932 5,2695 6,2540	7,2395 5,3271 9,6983	8,3306 9,7665 7,4332	6,5906 6,0823 7,1942	10,416 12,485 7,9326
Dieldrin	Total Male Female	0,0297	0,0001	0,0191 0,0154 0,0183	0,0225 0,0302 0,0104	0,0414 0,0138 0,0533	0,0228 0,0102 0,0306	0,0559 0,0553 0,0566	0,1136 0,1250 0,1000

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Sažetak

KONTAMINACIJA HRANE I MASNOG TKIVA LJUDI REZIDUIMA PESTICIDA U JUGOSLAVIJI (1969—1971)

Prikazani su rezultati analiza pesticidnih rezidua u hrani i masnom tkivu ljudi izvršenih u pojedinim laboratorijima Jugoslavije u vremenu od 1961. do 1971. godine. Od 800 uzoraka, 578 odnosilo se na hranu a 222 na masno tkivo ljudi dobiveno biopsijom.

Nalazi bi se mogli sažeti ovako:

1) Hrana animalnog porijekla kontaminirana je organoklornim insekticidima. Rezidua DDT-ja u mlijeku bila su od 0,0035 do 0,025 ppm.

2) Razina rezidua organoklornih insekticida izmjerenih u životinjskoj masti znatno je varirala od jednog do drugog laboratorija, sa srednjom vrijednošću za DDT od 0,404 ppm.

3) Rezidue organoklornih insekticida u voću i povrću bile su u dopuštenim

granicama.

4) Organoklorni insekticidi nađeni su u svim uzorcima masnog tkiva ljudi, sa značajnim razlikama zavisno od kraja i životne dobi.

5) U 19% uzoraka hrane biljnog porijekla nađeni su organofosforni insekticidi, no sve su vrijednosti bile unutar maksimalno dopuštenih koncentracija.

6) Arsenik je nađen u $52^{6}/_{0}$ uzoraka jabuka.

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