

REPORT ON THE ANALYSIS OF DDT AND ITS METABOLITES AND LINDANE IN SOIL AND PLANT SAMPLES FROM CONTAMINATED SITES IN DIFFERENT AREAS IN TANZANIA

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Executive Summary

Recoveries of 110% and 91% were observed for soil and plant samples respectively. The high percentage recoveries observed validate the extraction methods used. The concentrations of the OCPs are significantly high, particularly DDT and its metabolites in soil samples. Lindane was not detected in most of the plant samples indicating significantly reduced uptake of the OCP. The concentrations of DDT and its metabolites are significantly higher than those of lindane in both soil and plant samples. Lindane concentrations in soil ranged from ND-203 ng/g; while that of DDT and its metabolites ranged from 121-2,510 ng/g; 902-28,751 ng/g; 114-761 ng/g; 887-10,961 ng/g; 185-5,485 ng/g and 941-29,586 ng/g for Mitotane o,o-DDD, p,p'-DDD, o,p'-DDE, p,p'-DDE, o,p'-DDT and p,p' DDT respectively. In the case of plants samples (batch 1), the concentrations of lindane ranged from ND-0.22 ng/g. ; while that of DDT and its metabolites range from 0.03-21.59 ng/g; ND-429.60 ng/g; 0.04-67.44 ng/g; <0.01-7.29 ng/g; ND-53.32 and ND-287.71 ng/g for Mitotane o,o-DDD, p,p'-DDD, o,p'-DDE, p,p'-DDE, o,p'-DDT and p,p' DDT respectively. Similarly, the concentrations of lindane in plant samples (batch 2) ranged from ND-0.38 ng/g; while 0.14-27.05 ng/g, ND-391.56 ng/g, 0.03-18.63 ng/g, ND-2.74 ng/g, ND-74.62 ng/g and ND-402.92 ng/g respectively. The concentrations of DDT in soil and plants samples are significantly higher than the Canadian Soil Quality guidelines for protection of the environment and for human health.

ABBREVIATIONS

<i>DDT</i>	2,2-bis(chlorophenyl)-1,1,1-trichloroethane
<i>DDD</i>	2,2-bis(chlorophenyl)-1,1-dichloroethane
<i>DDE</i>	2,2-bis(chlorophenyl)-1,1-dichloroethene
<i>o,p'</i> -DDT	Ortho,para 2,2-bis(chlorophenyl)-1,1,1-trichloroethane
<i>p,p'</i> DDT	para,para 2,2-bis(chlorophenyl)-1,1,1-trichloroethane
<i>p,p'</i> -DDE	para, para 2,2-bis(chlorophenyl)-1,1-dichloroethene
<i>o,p'</i> -DDE	ortho, para ,2-bis(chlorophenyl)-1,1-dichloroethene
<i>o,o</i> -DDD	ortho, ortho 2,2-bis(chlorophenyl)-1,1-dichloroethane
<i>p,p'</i> -DDD	para,para 2,2-bis(chlorophenyl)-1,1-dichloroethane

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CHAPTER 1: INTRODUCTION AND BACKGROUND

1.1 Background information

Organochlorine pesticides (OCPs) have been widely used in growth fields in order to eliminate pests and in this way, increase crops outputs, improve quality of products and decrease the incidence of illnesses propagated by insects. Their high toxicity has made their use very restrictive reaching the point of being forbidden in most countries since last 1970s (Krauthacker et al., 2001) and some of them are also included in the Stockholm Convention.

Because of their toxic potential, their persistence and their tendency to bio-concentrate, the widespread use of pesticides in the environment generates increasing risks to human health such as cancer and disruption of hormonal functions (Sheridan and Meola, 1999; Basheer et al., 2002). Therefore, the determination of OCPs in water, plants, foodstuffs and soils is of major importance.

Organochlorine pesticides such as hexachlorocyclohexanes (HCHs) popularly known as lindane and isomers of dichlorodiphenyltrichloroethane (DDT) have been applied since 1940s for plant protection and sanitation throughout the world. Their use permitted plant protection, harvest assurance and the combat of vector-transmitted diseases. DDT was restricted to sanitary action to combat malaria and banned for agricultural uses in many counties. Lindane is a broad-spectrum organochlorine insecticide and fumigant that has been used on a wide range of soil-dwelling and plant eating insects. Technical lindane comprises the gamma isomer of hexachlorocyclohexane (HCH); other isomers of HCH (α , β , δ) occur in technical lindane but only gamma has significant insecticidal properties (Loibner et al., 1998). The common characteristic of these pesticides is their persistence in the environment and their bio-concentration in organism lipid compartments.

Contaminant levels in vegetation samples are used as indicators of environmental pollution as plants can adhere particles from soil and absorb compounds through their roots, and they can absorb volatile compounds and suffer atmospheric deposition in their leaves (Krauthacker et al., 2001). As plants accumulate pollutants in their tissues, they can be employed for decontamination of polluted sites. Agriculture products such as cereals, fruit, and vegetables

are often found to be contaminated with residues of persistent pesticides and other toxic substances. The major source of entry of these compounds to food chain is the contaminated soil. Therefore, the status of the residue level of most persistent organochlorinated pesticides (OCP) in soil and agricultural crops should be monitored regularly. The frequency of occurrence and contamination levels of OCP residues in soil and plant samples such as broad leaved sweet potato (*Ipomea batatas*); Slender-leaved sweet potato variant (*Ipomea batatas*); Simsim (*sesamum indicum*); Finger millet (*Eleusine coracana*); Chinese cabbage (*Brassica rapa* subsp. *pekinensis*); pumpkin (*Cucurbita maxima*); Calabash (*Lagenaria siceraria*); castor oil (*Ricinus communis*); collard (*Brassica sp.*); Wheat (*Triticum Aestivum*) and Barley (*Hordeum vulgare*) from different geographical regions in Tanzania were determined.

The present study aims at assessing the occurrence of OCPs, organochlorine pesticides such as DDT and its metabolites and lindane in soil and plant samples.

CHAPTER 2: MATERIAL AND METHODS

2.1 Study area

Dr Hamisi Tindwa from Sokoine University of Agriculture, Tanzania supplied the soil and plant samples. DDT and its metabolites and lindane were determined in all the soil and all plant samples from Morogoro site (Chinese cabbage, sweet potato (narrow leaves) sweet potato (broad leaved), irish potato, pumpkin, calabash and finger millet and Tengeru Arusha (simsim, chilli pepper, collard, lettuce, wheat and castor oil).

2.2 Extraction of soil samples

Soil samples (ca. 2.5 g) were weighed into pre-cleaned cellulose thimbles and were thereafter transferred into cleaned Soxhlet apparatus for extraction. The samples were extracted for 16 h using a mixture of n-hexane:acetone (2:1, v/v). Upon completion, the extracts were allowed to cool down to room temperature. The extracts were carefully transferred into pre-clean round bottom flasks and were rotary evaporated to approximately 2 mL. Two laboratory reference soil and plant samples were spiked with known amounts of p,p'-DDT and aforementioned extraction procedures repeated. The spiked reference samples serve as a quality assurance measure to assess the efficiency of the extraction method by estimating the analytical recoveries of the targeted compounds.

2.3 Extraction of plant samples

The root and shoot parts of several edible plants, namely: pumpkin, castor oil, Irish potato, hot pepper, collard, wheat, amongst others, were prepared for the determination of residual levels of the targeted OCPs. Approximately 2.5 g of the plant samples were weighed into pre-cleaned amber bottles. The samples were soaked overnight with 50 mL of n-hexane:acetone (1:1, v/v) followed by ultrasonic-assisted extraction for 30 min. The set-up was allowed to cool down to room temperature and the extract was carefully transferred into a clean round bottom flask. The extraction was repeated using the same volume of the extraction solvent and time. The extracts were combined and subjected to rotary evaporation as was previously indicated for soil samples. Similarly, two laboratory reference plant samples (lettuce) were spiked with known amounts of p,p'-DDT and similarly prepared following the aforementioned procedures.

The spiked reference samples were employed for the estimation of the analytical recoveries of the targeted compounds.

2.4 Extract clean-up

The soil and plant extracts were purified using deactivated silica gel packed into glass columns. Prior to the clean-up procedure, the packed columns were conditioned using 25 mL of n-hexane to remove trapped air and possible interfering contaminants. The concentrated extract was quantitatively transferred into the glass column and was eluted under gravity with 40 mL of n-hexane:acetone (2:1, v/v). The eluate was rotary evaporated to approximately 2 mL, and was transferred into an amber vial where it was further concentrated with high purity nitrogen gas until it reaches incipient dryness. The extracts were re-constituted with 1 mL of Toluene followed by the addition of 50 μL of 500 $\text{pg } \mu\text{L}^{-1}$ of DDT-d8 that was employed as an internal standard.

2.5 Instrumental analysis

The quantitative estimation of all the targeted compounds was performed using an Ultra-trace 2010 Shimadzu GC equipped with QP 2010 Ultra mass spectrometer operated in EI mode. The chromatographic separation of these compounds was achieved using DB-5 MS (15 m, 0.25 mm i.d., 0.10 μm film thickness) capillary column. The optimal conditions employed for the GC-EI-MS instrument are shown in Table 2-1. To enhance the sensitivity of the instrument and to overcome the inherent problems of interfering co-extractants, the MS acquisition was carried out in selected ion monitoring (SIM) mode. In this case, a target ion and two reference ions were selected for each targeted compound as well as internal standard (DDT-d8) for their identification and quantification. The details of the selected target and reference ions for both the target compounds and the internal standard are presented in Table 2-2.

The quantification of all the target compounds, which was based on peak area, was done using internal calibration techniques. This was done using eight to ten calibration levels (1, 2, 5, 10, 20, 50, 100, 200, 500 and 1000 ng mL^{-1}) for all targeted OCP compounds. In all cases, the linearity (R^2) of the resulting calibration curves was greater than 0.99.

Table 2-1: Optimised GC-EI-MS conditions employed for OCPs analysis

Parameters	Optimum conditions
GC parameters	
<i>Injection volume</i>	1 µL
<i>Carrier gas (% purity)</i>	Helium (99.999%)
<i>Injection mode</i>	Splitless
<i>Flow control mode</i>	Linear velocity
<i>Injector temperature</i>	270°C
<i>Linear velocity</i>	63.5 cm/sec
<i>Column flow</i>	1.5 ml/min.
<i>Purge flow</i>	3.0 ml/min.
<i>Equilibrium time</i>	3.0 min.
<i>Sampling time</i>	2.00 min.
<i>Oven temperature programming</i>	70°C held for 1.0 min., ramped @ 25°C/min. to 180°C, ramped @8°C/min to 300°C held for 5 min.
MS parameters	
<i>Ion source temperature</i>	270°C
<i>Interface temperature</i>	280°C
<i>Solvent cut time</i>	2.0 min.
<i>Acquisition mode</i>	SIM
<i>Ionisation method</i>	EI

Table 2-2: Details of the target and reference ions employed during SIM analysis of OCPs

Targeted compounds	Retention time (min.)	Target ion	Reference ions
Lindane	5,24	183	181, 219
Mitotane (o,p'-DDD)	6,92	235	237, 199
o,p'-DDE	7,05	246	248, 176
p,p'-DDD	7,36	235	237, 199
p,p'-DDE	7,51	246	248, 176
p,p'-DDT	7,365	235	237, 199
o,p'-DDT	7,59	235	237, 199
p,p'-DDT-d8 (Internal Standard)	7,33	243	245, 173

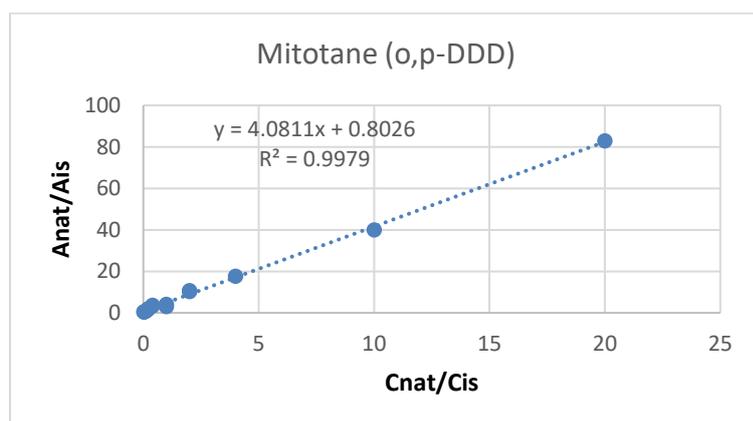
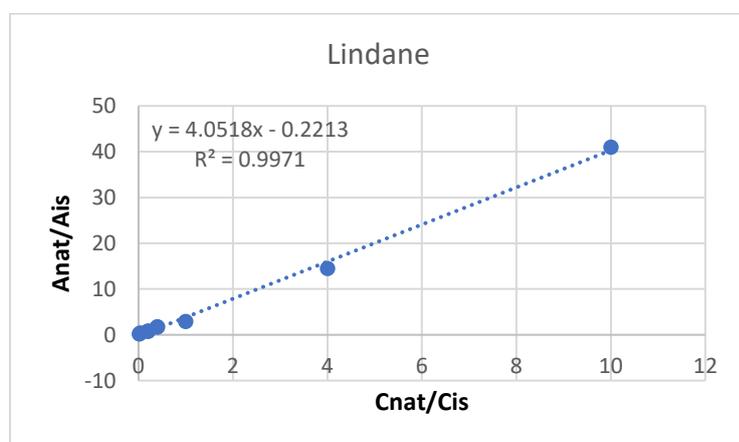
CHAPTER 3: RESULTS AND DISCUSSION

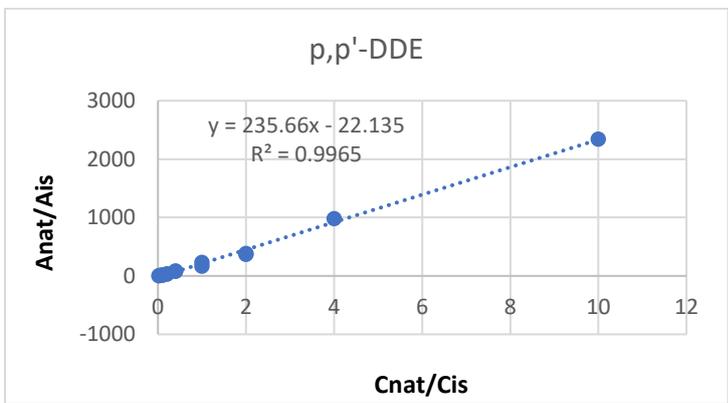
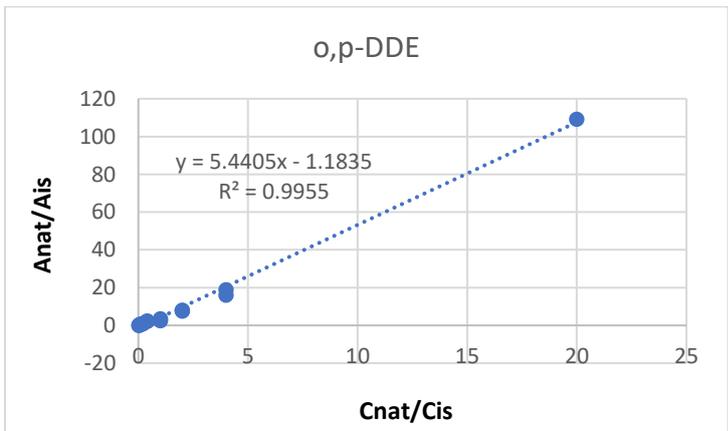
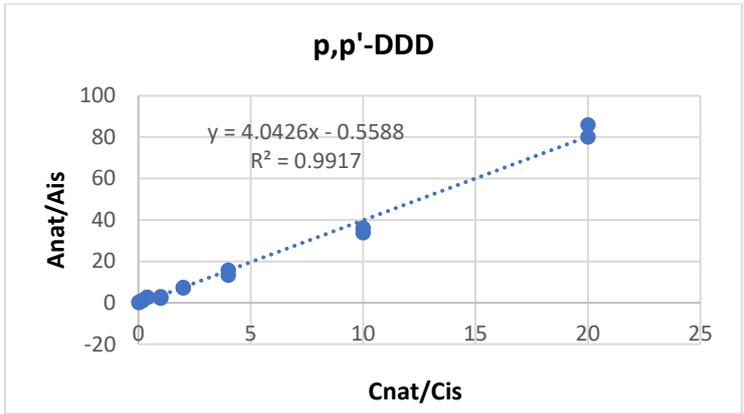
3.1 Recovery of spiked reference sample with p,p'-DDT

Recoveries of 110% and 91% were obtained for soil and plant samples respectively. The high percentage recoveries observed validate the extraction methods used.

3.2 Calibration curves

The calibration curves for lindane and DDT and its metabolites are shown in Figures 3-1. As can be seen the calibration curves are in order.





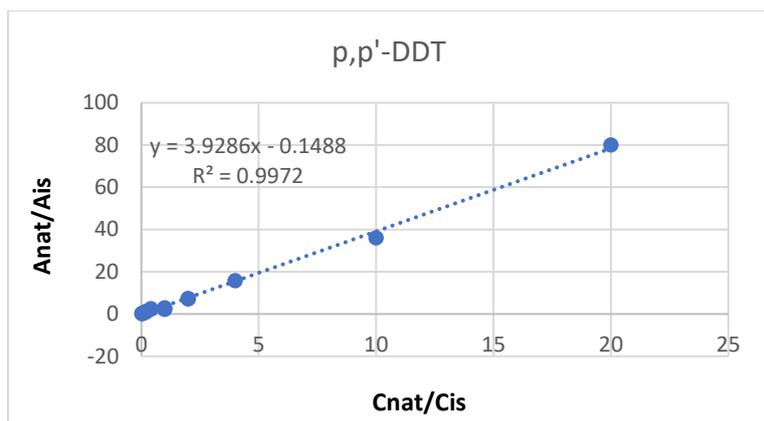


Figure 3-1 Calibration curves employed for the quantitative analysis of the targeted OCPs

3.3 Soil samples

Table 3-1 shows the concentrations of OCPs in soil samples. As can be seen in Table 3-1, lindane concentrations range from ND-203 ng/g; while that of DDT and its metabolites range from 121-2,510 ng/g; 902-28,751 ng/g; 114-761 ng/g; 887-10,961 ng/g; 185-5,485 ng/g and 941-29,586 ng/g for Mitotane o,o-DDD, p,p'-DDD, o,p'-DDE, p,p'-DDE, o,p'-DDT and p,p' DDT respectively.

Table 3-1: Mean concentrations of selected organochlorine pesticides in soil samples

	Concentration (ng/g)						
Soil sample	Lindane	Mitotane (o,p-DDD)	p,p'-DDD	o,p-DDE	p,p'-DDE	o,p-DDT	p,p-DDT
FERT_X1	13	1003	10059	357	3529	1917	10351
FERT_X2	ND	1108	12987	761	9351	2475	13364
FERT_X3	9	323	3632	114	887	692	3737
FERT_X4	ND	1152	10425	740	7435	1993	10722
FERT_X5	30	2510	28751	642	7970	5485	29586
FERT_X6	181	550	2519	443	3688	517	2706
FERT_X7	203	121	902	123	1928	195	941
FERT_X8	157	1379	5329	791	10961	1030	5473
P2_O_200 m	ND	752	6299	190	1807	1201	6484

3.4 Plant samples (Batch 1&2)

The concentrations of OCPs in plant samples (batch 1) are shown in Table 3-2. Lindane concentrations range from ND-0.22 ng/g. ; while that of DDT and its metabolites range from 0.03-21.59 ng/g;

ND-429.60 ng/g; 0.04-67.44 ng/g; <0.01-7.29 ng/g; ND-53.32 and ND-287.71 ng/g for Mitotane, o,o-DDD, p,p'-DDD, o,p'-DDE, p,p'-DDE, o,p'-DDT and p,p' DDT respectively. Table 3-3 shows OCPs concentrations in plant samples in batch 2. The range for lindane is ND-0.38 ng/g; while 0.14-27.05 ng/g, ND-391.56 ng/g, 0.03-18.63 ng/g, ND-2.74 ng/g, ND-74.62 ng/g and ND-402.92 ng/g respectively.

Table 3-2: Measured concentrations of selected organochlorine pesticides in plant samples (batch 1)

Sample code	Sample name	Concentration (ng/g)						
		Lindane	Mitotane (o,p-DDD)	p,p'-DDD	o,p-DDE	p,p'-DDE	o,p-DDT	p,p-DDT
1	TNR OAT ROOT R1	0.22	0.03	0.40	0.15	0.02	ND	0.01
2	TNR C.OIL ROOT R1	0.10	0.10	0.30	0.24	0.04	9.63	51.38
3	TNR COLLARD SHOOT R2	ND	1.01	ND	0.18	<0.01	ND	0.80
4	TNR COLLARD SHOOT R3	ND	0.85	0.33	0.13	0.01	ND	0.23
5	TNR HOT PEPPER SHOOT R3	ND	0.14	8.82	0.46	0.01	2.54	8.99
6	TNR C.OIL ROOT R3	ND	0.55	34.60	1.06	0.09	6.54	35.42
7	TNR H. PEPPER R1	ND	0.33	9.25	1.13	0.30	9.65	9.40
8	TNR SIM SIM SHOOT R3	ND	21.59	279.61	67.44	7.29	53.32	287.71
9	TNR SIM SIM ROOT R1	ND	1.64	ND	4.21	0.61	ND	1.92
10	TNR COLLARD ROOT R1	ND	2.39	9.81	0.83	0.01	1.54	8.13
11	TNR C. OIL ROOT R3	ND	0.95	12.53	0.34	0.01	2.55	12.85
12	TNR WHEAT SHOOT R2	ND	7.56	37.09	0.60	0.11	8.14	37.82
13	TNR SIM SIM ROOT R2	ND	0.04	ND	0.01	<0.01	0.00	ND
14	TNR WHEAT ROOT R2	ND	1.26	ND	1.14	1.57	ND	2.85
15	TNR OAT ROOT R2	ND	2.11	6.93	2.19	0.14	1.30	6.93
16	TNR WHEAT ROOT R1	ND	0.79	ND	1.40	0.10	5.22	ND
17	TNR C.OIL SHOOT R1	ND	0.40	ND	0.20	0.01	ND	ND
18	TNR HOT PEPPER SHOOT R2	ND	1.33	11.03	0.45	0.01	2.16	11.28
19	TNR C.OIL SHOOT R1	ND	1.00	ND	0.38	0.02	ND	ND
20	TNR WHEAT SHOOT R1	ND	0.07	26.73	1.40	0.01	5.81	27.37
21	TNR OAT ROOT R3	ND	7.25	15.84	2.58	0.05	3.46	16.09
22	TNR HOT PEPPER ROOT R2	ND	0.66	99.03	0.67	0.65	ND	ND
23	TNR COLLARD SHOOT R1	ND	1.16	1.71	0.43	<0.01	0.33	1.64
24	TNR HOT PEPPER SHOOT R3	ND	0.02	ND	0.01	<0.01	ND	ND
25	TNR C.OIL SHOOT R2	ND	0.04	ND	0.04	0.01	ND	ND
26	TNR OAT SHOOT R3	ND	0.26	0.73	0.13	<0.01	ND	ND
27	TNR COLLARD ROOT R2	ND	4.09	ND	1.45	0.04	ND	ND
28	TNR COLLARD ROOT R3	ND	2.85	12.40	2.56	0.88	ND	12.58
29	TNR HOT PEPPER SHOOT R1	ND	0.04	0.01	0.16	<0.01	ND	ND
30	TNR SIM SIM SHOOT R1	ND	0.05	7.32	0.10	0.01	ND	ND
31	TNR WHEAT SHOOT R3	ND	0.40	429.60	2.33	0.19	7.70	14.76
32	TNR C.OIL ROOT R2	ND	0.07	0.03	0.07	0.06	ND	0.03

Table 3-3: Measured concentrations of selected organochlorine pesticides in plant samples (batch 2)

Sample code	Sample name	Concentration (ng/g)						
		Lindane	Mitotane (o,p-DDD)	p,p'-DDD	o,p-DDE	p,p'-DDE	o,p-DDT	p,p-DDT
1b	NHC CHINESE ROOT R2	ND	7.24	69.91	18.11	1.77	0.45	71.94
2b	NHC PUMPKIN ROOT R3	ND	2.41	26.44	4.29	0.82	0.18	27.20
3b	NHC PUMPKIN SHOOT R2	ND	27.05	261.27	51.96	6.19	48.34	259.47
4b	NHC PUMPKIN ROOT R1	ND	3.85	19.89	1.94	0.35	0.07	20.46
5b	NHC PUMPKIN ROOT R2	ND	6.95	27.73	5.47	0.75	0.14	28.53
6b	NHC F.MILLET ROOT R3	ND	25.09	64.46	12.97	0.04	2.26	66.54
7b	NHC CHINESE SHOOT R3	ND	0.14	ND	0.03	ND	ND	ND
8b	NHC CALABASH ROOT R2	ND	8.82	19.28	5.92	0.60	0.36	19.84
9b	NHC PUMPKIN SHOOT R1	ND	10.39	91.64	6.60	2.21	17.46	94.29
10b	TNR OAT SHOOT R2	0.38	0.36	1.51	0.26	0.05	0.29	1.54
11b	TNR SIM SIM ROOT R3	ND	2.10	1.44	0.97	0.11	ND	1.41
12b	NHC TEMBELE R2	ND	13.07	391.56	9.21	1.94	74.62	402.92
13b	NHC CHINESE ROOT R2	ND	17.09	70.36	18.63	1.80	13.41	72.38
14b	NHC TEMBELE SHOOT R1	0.36	3.69	51.73	3.17	0.01	9.86	53.22
15b	NHC IRISH P.SHOOT R2	ND	9.22	42.61	2.78	0.94	8.12	43.84
16b	NHC VIAZI SHOOT R3	0.27	9.18	55.75	4.88	1.28	10.78	57.36
17b	NHC CHINESE SHOOT R1	ND	10.71	55.76	10.66	1.33	0.27	57.38
18b	NHC VIAZI SHOOT R3	0.13	9.02	56.48	4.60	1.24	10.76	58.12
19b	NHC VIAZI SHOOT R2	0.33	10.22	66.33	8.85	1.63	12.64	68.24
20b	NHC PUMPKIN FRUIT R3	ND	1.17	4.37	1.11	0.18	0.83	4.49
21b	NHC CHINESE ROOT R1	ND	1.31	4.91	1.28	0.21	0.94	5.05
22b	NHC VIAZI SHOOT R1	ND	6.35	41.59	7.38	1.50	7.92	42.79
23b	NHC IRISH P.SHOOT R1	ND	4.42	22.27	1.82	0.42	4.24	22.91
24b	NHC PUMPKIN ROOT R3	ND	3.75	18.06	2.47	0.71	3.45	18.57
25b	NHC PUMPKIN FRUIT R2	ND	1.25	10.43	1.20	0.35	1.99	10.73
26b	NHC IRISH P.SHOOT R1	ND	7.97	41.57	3.36	1.28	8.06	42.74
29b	NHC CHINESE SHOOT R2	ND	13.69	46.17	11.51	1.42	8.80	47.50
30b	NHC F.MILLET ROOT R1	0.03	25.76	37.68	12.29	0.70	7.18	38.77
31b	NHC PUMPKIN SHOOT R3	ND	3.80	15.21	3.42	0.25	2.90	15.65
32b	NHC CALABASH SHOOT R2	ND	8.46	40.50	6.36	0.54	7.72	41.67
33b	NHC CALABASH ROOT R1	ND	5.18	12.26	2.57	0.20	2.34	12.62
34b	NHC F.MILLET ROOT R2	ND	14.41	32.10	9.29	0.42	6.12	33.03
35b	NHC IRISH P.SHOOT R3	ND	3.28	13.37	1.21	0.20	2.55	13.76
36b	NHC TEMBELE SHOOT R3	0.08	12.62	72.03	6.76	1.23	13.84	74.06
37b	NHC CALABASH SHOOT R3	ND	32.80	83.96	39.07	2.74	16.00	86.38

3.5 Conclusion

The concentrations of the OCPs are significantly high, particularly DDT and its metabolites in soil samples. Lindane was not detected in most of the plant samples indicating significantly reduced uptake of the OCP. The concentrations of DDT and its metabolites are significantly higher than those of lindane in both soil and plant samples. The concentrations of DDT in soil and plants samples are significantly higher than the Canadian Soil Quality guidelines for protection of the environment and for human health.

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