Short communication

Note on the occurrence of selected trace metals and organic compounds in water, sediment and biota of the Crocodile River, Eastern Transvaal, South Africa

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Abstract

Samples of water, sediment and biota (7 species of fish and 2 species of macrophytic plant) were collected at a number of sites on the Crocodile River, E. Transvaal. These samples were analysed for the trace metals arsenic (As), cadmium (Cd), copper (Cu), lead (Pb), mercury (Hg), selenium (Se), zinc (Zn), manganese (Mn), aluminium (Al) and iron (Fe), the pesticides DDT, DDE, DDD, Dieldrin, Heptachlor, Lindane and Endosulfan, as well as the polychlorinated biphenyls Arochlor 1254 and Arochlor 1260. Acid-extractable concentrations of the detected metals are reported. Concentrations of As, Cd, Cu, Hg and Se never exceeded their respective detection limits in water samples. All the metals but Cd, Pb and Hg were detected in the sediment samples, and all but Cu, Pb and Hg were detected in muscle tissue of fish. Arsenic, Cu, Zn, Mn, Al and Fe were detected in plant tissue. The detected metals always occurred at higher concentrations in the roots than in the leaves of the sampled plants. None of the selected pesticides was detected in the sediment samples and only *p,p*-DDE was found in muscle tissue of fish.

Introduction

Pollution of South African waters by pesticides, mainly of agricultural origin, and by trace metals, mainly of industrial origin, is a cause of increasing concern. This concern has escalated rapidly in recent years following major fish kills in certain parts of the country, e.g. the E. Transvaal and the Pretoria-Witwatersrand-Vereeniging (PWV) areas (Heath, 1992). Concern has also been expressed about the inadequacy of knowledge of, and data on, pesticide and metal concentrations in South African waters (DWA, 1986).

Birds, reptiles and mammals (including man) drink water and derive food from aquatic ecosystems, and are vulnerable to contaminant exposure and accumulation. The impact of pesticides and/or trace metals on aquatic organisms can be either dramatic (e.g. acute fish kills), or insidious due to gradual accumulation in the body tissues of organisms. Biotas are not only influenced by toxicant concentrations in the water or sediment, but due to the process of bioaccumulation, may be exposed to much higher amounts. Concentrations of many toxic substances in water are magnified as they are passed up through the food web, via phytoplankton, zooplankton and forage fish, to piscivorous fish. This results in high contaminant concentrations in top predators (Swackhamer and Eisenreich, 1991).

Many of the organisms which live in, and feed from, aquatic systems are of ecological and economical value. Often the primary cause of toxification by organic and inorganic contaminants associated with the aquatic environment is consumption of fish or shellfish, rather than drinking of water (Mackay and Clark, 1991). Health risks associated with consumption of contaminated fish may be 20 to 40 times higher than those resulting from exposure to the same chemicals through drinking water (Foran, 1990). A hydrophobic chemical such as a polychlorinated biphenyl (PCB) or DDT may establish a concentration in fish 100 000 times that in water, implying a bioconcentration factor of 100 000 (Mackay and Clark, 1991). Because bioaccumulation may ultimately produce levels of contaminants in commercial species that may be toxic to humans, it is important to monitor key members of food chains (Chapman et al., 1982).

In order to allow detection of unacceptable contamination and of long-term trends, it is necessary to establish baseline levels nationally and to implement a statistically sound monitoring programme. A programme similar to the National Contaminant Biomonitoring Programme, where the US Fish and Wildlife Service periodically determines concentrations of organochlorine chemicals and selected trace metals in freshwater fish (Schmitt and Brumbaugh, 1990 and Schmitt et al., 1990), should provide useful information. The need for a national assessment of concentrations of persistent contaminants in the aquatic environment is certainly acknowledged. However, work in this field has in the past been hampered by the high costs involved and the limited availability of manpower. Currently the Department of Water Affairs and Forestry is investigating the feasibility of conducting bioaccumulation surveys in selected catchments.

Relatively few studies have been undertaken in South Africa dealing with levels of pesticides and trace metals in sediment or biota, and even fewer have incorporated the 3 main compartments of the aquatic environment, namely water, sediment and biota. For the rivers of the E. Transvaal, published data include levels of organochlorine pesticides in fish from the Letaba and Crocodile Rivers (Heath, 1992), pesticide concentrations in water, sediment and fish in the Olifants River (Grobler, 1991) and concentrations of selected trace metals in the tissues and organs of tigerfish (*Hydrocynus vittatus*) in the Olifants River (Du Preez and Steyn, 1992).

The Crocodile River catchment in the E. Transvaal supports the largest irrigation area in South Africa, with some 95 000 ha of a variety of crops being irrigated (Heath, 1992). Apart from its economic importance, the ecological significance of the Crocodile River is accentuated as it forms the southern boundary of the

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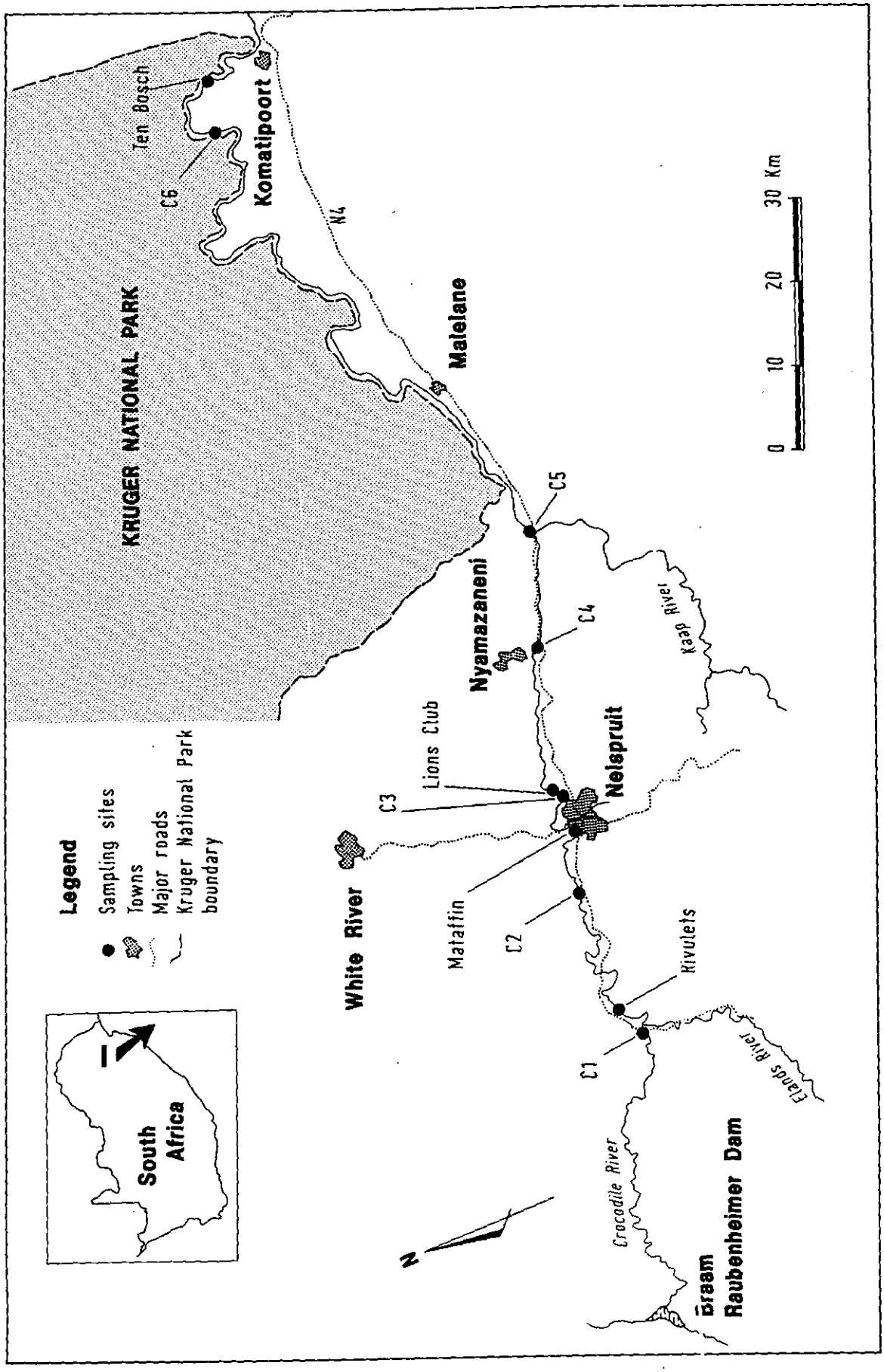

Figure 1
Map indicating study area and sampling sites

TABLE 1
STANDARD LENGTH (SL), TOTAL LENGTH (TL), MASS, GENDER AND AGE OF SPECIES OBTAINED AT DIFFERENT LOCALITIES

Species	Fish code	SL (mm)	TL (mm)	Mass (g)	Gender	Age (years)
C. gariepinus	BR1	520	570	1 800	♀	2+
C. gariepinus	BR2	492	541	970	♀	3+
C. carpio	BR3	350	364	890	♂	5+
O. mossambicus	BR4	262	311	400	♂	4+
C. gariepinus	MW1	490	551	1 350	♂	2+
C. gariepinus	MW2	410	441	750	♀	2+
B. marequensis	MW3	151	162	50	♂	NA
B. marequensis	MW4	152	160	50	♂	NA
C. gariepinus	LC1	495	502	820	♀	2+
C. gariepinus	LC2	545	552	820	♀	2+
T. rendalli	TB1	260	320	750	♂	4+
L. rosae	TB2	370	410	1 600	♀	5+
L. congoro	TB3	270	290	550	♂	5+
B. marequensis	TB4	420	450	1 550	♀	10+

NA - Not available; BR - Braam Raubenheimer Dam; MW - Mataffin Weir; LC - Lions Club; TB - Ten Bosch Weir

Kruger National Park. During 1992 members of the Hydrological Research Institute (now the Institute for Water Quality Studies) of the Department of Water Affairs and Forestry conducted a series of biosurveys of the Crocodile River. Samples of water, sediment and biota were collected during 5 consecutive surveys, which took place at approximately 6-weekly intervals from May to October of 1992. These were analysed for the trace metals As, Cd, Cu, Pb, Hg, Se, Zn, Mn, Al and Fe, the pesticides DDT, DDE, DDD, Dieldrin, Heptachlor, Lindane and Endosulfan, as well as the polychlorinated biphenyls Arochlor 1254 and Arochlor 1260. This communication reports on, and briefly discusses, the concentrations recorded.

Materials and methods

The study area and sampling sites selected are indicated in Fig. 1. Sites C1 to C6 represent fast-flowing, well-mixed riffle areas where water samples were collected. The other sites (Braam Raubenheimer Dam, Rivulets, Mataffin Weir, Lions Club and Ten Bosch Weir) represent slow-flowing or impounded water where sediments and fish could be collected.

Sampling and preservation procedures

Water samples for trace metal analysis were collected during each of the 5 sampling surveys. These samples, except for Hg analysis, were collected unfiltered in 350 mℓ polyethylene sample bottles and preserved with 4 mℓ concentrated HNO_3. Samples for the determination of Hg were collected in glass bottles and preserved with 4 mℓ concentrated H_2SO_4.

Sediment samples were taken once at each site, during the October survey, by means of a Petersen grab. This method allowed grab collection of approximately the top 150 mm of the sediment. Samples for trace metal analysis were stored in plastic bottles at 4°C prior to analysis. Samples for organic analysis were stored in tins at -20°C prior to analysis.

Fish were collected, during the surveys in June (Braam Raubenheimer Dam and Mataffin Weir) and October 1992 (Lions Club and Ten Bosch Weir), using gill nets. The following species were selected for analyses: *Clarias gariepinus* (African sharptooth catfish), *Cyprinus carpio* (carp), *Oreochromis mossambicus* (Mozambique tilapia), *Barbus marequensis* (large-mouth yellowfish), *Tilapia rendalli* (red-breasted tilapia), *Labeo rosae* (red-nosed labeo) and *Labeo congoro* (red-spotted mudsucker). After collection, fish were weighed, measured and gutted. Whole fish were wrapped in aluminium foil and stored at -20°C prior to preparation for analysis. Data on the length, mass, gender and age of fish used in this survey are presented in Table 1. Samples of scales or pectoral spines (in the case of *C. gariepinus*) were removed from each fish for age determination. These samples were cleaned, treated and the annuli counted with the aid of a microscope.

Aquatic macrophytic plants were collected during the survey in October by taking freely floating individuals from relatively stable colonies. Two species were collected, both of which have been declared noxious weeds (Van der Merwe, 1991), namely water hyacinth (*Eichornia crassipes*) and Kariba weed (*Salvinia molesta*). Water hyacinth was collected from Site C5, Site C6 and Ten Bosch Weir, and Kariba weed from Mataffin Weir (Fig. 1). Plants were stored in plastic bottles at 4°C prior to analysis.

Sample preparation and analyses

Trace metals

The water samples were analysed for acid-extractable metals. A portion of each sample was transferred to a glass beaker and boiling beads were added. Each beaker and its contents were weighed and the mass recorded. Concentrated HNO_3 and 50% (v/v) HCl were added to the beakers. The samples were heated in a fume cupboard to near boiling, then simmered for 30 min. After cooling the samples were made up to their original masses by the addition of deionised water before being filtered (DWAF, 1992).

Wet sediment samples were dried overnight in an oven at 30°C. Dry sediment with a mass of 0.1 g (<0.2 mm fraction) was digested in a commercial microwave oven using a Teflon digestion vessel and a 5:1 (v/v) mixture of aqua regia and concentrated HF. After digestion the samples were left to cool. Approximately 2 g of boric acid was added to each sample to complex the fluoride ions. The extracts were filtered through No. 42 Whatman filter paper before being analysed.

One gram of homogenised muscle tissue of each fish was digested in a commercial microwave oven, using a Teflon digestion vessel. The digestion mixture consisted of concentrated HNO_3 and H_2O_2. A composite sample was made from the 2 small largemouth yellowfish collected at Mataffin Weir (MW3 + MW4; Table 1). Dried plant tissue with a mass of 0.1 g of each plant sample was extracted using the method described for fish. After digestion the samples were filtered into a 50 mℓ volumetric flask, made up to the mark with deionised water and analysed.

Inductively coupled plasma emission spectrometry (ICP) was used to determine and quantify the presence of trace metals (except for Se and As) in sediment, fish tissue and plant material (Kempster, 1986). Selenium and As concentrations were determined by means of hydride generation in conjunction with ICP emission spectrometry (Pretorius et al., 1992)

Organic compounds

The sediment samples were freeze-dried and sieved to obtain the fraction < 63 µm. Twenty grams of this fraction was Soxhlet

TABLE 2
ACID-EXTRACTABLE CONCENTRATIONS (IN $mg \cdot \ell^{-1}$) FOR METALS DETECTED IN WATER FROM CROCODILE RIVER SAMPLING SITES DURING THE 5 SAMPLING PERIODS

		Sampling sites					
Metal	**Date**	**C1**	**C2**	**C3**	**C4**	**C5**	**C6**
Pb	92/05/06	0.115	<0.050	<0.050	NS	<0.050	<0.050
	92/06/09	<0.050	<0.050	0.215	0.395	0.051	<0.050
	92/07/20	0.238	<0.050	0.099	0.265	<0.050	<0.050
	92/09/14	<0.050	<0.050	<0.050	<0.050	0.128	<0.050
	92/10/19	<0.050	<0.050	<0.050	<0.050	<0.050	<0.050
Zn	92/05/06	<0.004	<0.004	<0.004	NS	<0.004	<0.004
	92/06/09	0.047	<0.004	<0.004	0.015	0.005	0.019
	92/07/20	0.018	0.046	0.028	0.010	<0.004	0.017
	92/09/14	<0.004	0.012	<0.004	<0.004	<0.004	<0.004
	92/10/19	0.095	0.093	0.076	0.009	<0.004	<0.004
Mn	92/05/06	0.081	0.301	2.445	NS	0.070	0.168
	92/06/09	0.305	0.033	2.774	0.016	0.018	<0.001
	92/07/20	0.020	0.030	2.410	0.031	0.044	0.030
	92/09/14	0.083	0.050	1.461	0.146	0.086	0.099
	92/10/19	0.060	0.071	0.801	0.052	0.107	0.143
Al	92/05/06	0.469	0.168	0.354	NS	<0.100	<0.100
	92/06/09	1.206	0.438	0.487	0.108	<0.100	<0.100
	92/07/20	0.663	0.268	0.394	<0.100	<0.100	<0.100
	92/09/14	0.452	0.136	0.136	0.148	0.105	<0.100
	92/10/19	0.697	0.303	0.260	<0.100	<0.100	<0.100
Fe	92/05/06	2.713	1.623	0.886	NS	1.272	0.464
	92/06/09	2.669	1.295	1.407	0.102	<0.020	0.116
	92/07/20	1.713	0.418	0.523	<0.020	0.139	<0.020
	92/09/14	1.094	0.479	0.211	<0.020	0.125	<0.020
	92/10/19	1.456	0.848	0.704	0.284	0.218	0.203

NS - Not sampled

TABLE 3
CONCENTRATIONS OF TRACE METALS IN SEDIMENT (mg·g^{-1} DRY-MASS)

Site	As	Cd	Cu	Pb	Hg	Se	Zn	Mn	Al	Fe
BR	0.040	<0.005	0.154	<0.050	<0.020	0.014	0.092	2.103	66.550	98.270
RIV	0.067	<0.005	0.050	<0.050	<0.020	0.010	0.047	0.556	44.050	37.920
MW	0.035	<0.005	0.096	<0.050	<0.020	0.015	0.098	1.911	88.630	62.079
LC	0.038	<0.005	0.075	<0.050	<0.020	0.013	0.218	18.440	84.990	51.840
TB	0.040	<0.005	0.069	<0.050	<0.020	0.012	0.059	2.909	72.230	46.518

BR - Braam Raubenheimer Dam; RIV - Rivulets; MW - Mataffin Weir; LC - Lions Club; TB - Ten Bosch Weir

extracted with hexane for 16 h. The extract was concentrated and the sulphur removed. After sulphur removal, the extract was further purified by passing through an alumina column. Most of the muscle tissue of each fish sample was homogenised, and 20 g portions were dried with sodium sulphate. After drying the samples were Soxhlet extracted with hexane for 16 h. Extracts were concentrated and the amount of fat present was determined. Samples were purified using gel permeation chromatography followed by passage through an alumina column.

A gas chromatograph equipped with an electron capture detector was used for the identification and quantification of the chlorinated pesticides in sediment and fish samples. Detection limits for the selected pesticides that apply to this study were as follows: In sediment the limits were 0.1 $\mu g \cdot \ell^{-1}$ for 4,4'-DDT, Arochlor 1254 and Arochlor 1260; 0.2 $\mu g \cdot \ell^{-1}$ for Lindane; 0.3 $\mu g \cdot \ell^{-1}$ for 2,4'-DDE and 4,4'-DDE; 0.4 $\mu g \cdot \ell^{-1}$ for Heptachlor, Endosulfan, Dieldrin and 2,4'-DDT; and 0.7 $\mu g \cdot \ell^{-1}$ for 2,4'-DDD and 4,4'-DDD. In fish tissue the limits were 0.2 $\mu g \cdot \ell^{-1}$ for 4,4'-DDT and 0.1 $\mu g \cdot \ell^{-1}$ for all the other selected organic compounds. A gas chromatograph equipped with a mass selective detector was used for confirmation of the presence of the compounds. The methods used for determination of the organic compounds are described in detail in DWAF (1992).

Results and discussion

Trace metals

Reported quality criteria for metals in the aquatic environment vary from addressing total concentrations to the fraction of metals in the dissolved phase, and as yet there is no consensus as to which fraction should be used. This, together with the fact that South African quality criteria/guidelines for the protection of aquatic life are still under development, hampers the interpretation of obtained metal concentrations. For the purpose of this communication, concentrations are mainly reported for their background and archival value. A discussion on the effects of various metals on riverine ecosystems and a summary of existing criteria can, however, be found in Dallas and Day (1993).

Concentrations of As (0.003 mg·ℓ^{-1}), Cd (0.005 mg·ℓ^{-1}), Cu (0.005 mg·ℓ^{-1}), Hg (0.002 mg·ℓ^{-1}) and Se (0.002 mg·ℓ^{-1}) in the water samples never exceeded their respective detection limits, given in parentheses. Acid-extractable concentrations for Pb, Zn, Mn, Al and Fe are presented in Table 2. Since acid-extractable concentrations were determined, the metals adsorbed onto suspensoids will have contributed to the result.

Lead was sporadically detected at Sites C1, C3, C4 and C5. The occurrence of Zn was also sporadic, but this metal was detected at least once at each of the sampling sites. Manganese was regularly detected throughout the study area. The relatively high Mn concentrations at Site C3 may be a result of several point-source inputs in the vicinity of Nelspruit (Fig. 1). The higher Al and Fe concentrations at Site C1 can probably be associated with the higher suspended-solid content of the river in the upper reaches (Roux, 1994).

Concentrations of trace metals in sediment, fish muscle and plant material are given in Tables 3, 4 and 5 respectively. Cadmium, Pb and Hg did not exceed their detection limits in any of the sediment samples, whereas Cu, Pb and Hg were not detected in muscle tissue of fish. Downstream of Nelspruit (Lions Club), as with the water samples, a relatively high Mn concentration (18.440 mg·g^{-1}) occurred in the sediment sample (Table 3). Arsenic was recorded in 2 of the fish samples from Mataffin Weir, at concentrations of 1 and 2 $\mu g \cdot g^{-1}$ respectively. A Cd concentration of 2 $\mu g \cdot g^{-1}$ was detected in one of the fish sampled from Ten Bosch Weir. Aluminium was relatively high (12 $\mu g \cdot g^{-1}$) in the composite sample of the 2 large-mouth yellowfish from Mataffin Weir. Selenium and Mn were detectable in respectively 7 and 10 of the 13 fish samples, while Zn and Fe were present in all samples (Table 4). Cadmium, Pb, Hg and Se were not detected in any of the plant samples. Metals always occurred at higher concentrations in the roots than in leaves of plants sampled (Table 5).

Organic compounds

Water samples and plant material were not analysed for the selected pesticides. These hydrophobic compounds are generally not present in water in measurable concentrations and previous studies have indicated little or no uptake and translocation of highly lipophilic compounds, such as DDT and PCBs, in plant material (Fries, 1991).

None of the selected pesticides was detected in any of the sediment samples. Although concentrations of Dieldrin, Endosulfan, DDT, DDE and DDD have been reported in some organs and tissues of fish from the Crocodile River (Heath, 1992), during this

TABLE4
CONCENTRATIONS OF SELECTED TRACE METALS (µg·g^{-1} WET-MASS) RECORDED IN MUSCLE TISSUE OF FISH

Fish code	As	Cd	Cu	Pb	Hg	Se	Zn	Mn	Al	Fe
BR 1	<0.100	<0.250	<0.250	<2.500	<1.000	0.300	2.000	<0.050	<5.000	150.0
BR 2	<0.100	<0.250	<0.250	<2.500	<1.000	0.300	3.000	<0.050	<5.000	56.00
BR 3	<0.100	<0.250	<0.250	<2.500	<1.000	<0.150	18.000	1.000	<5.000	18.00
BR 4	<0.100	<0.250	<0.250	<2.500	<1.000	0.200	5.000	2.000	<5.000	24.00
MW 1	<0.100	<0.250	<0.250	<2.500	<1.000	<0.150	3.000	<0.050	<5.000	7.00
MW 2	2.000	<0.250	<0.250	<2.500	<1.000	3.000	5.000	1.000	<5.000	10.00
MW 3+4	1.000	<0.250	<0.250	<2.500	<1.000	0.600	15.000	12.000	12.000	94.00
LC 1	<0.100	<0.250	<0.250	<2.500	<1.000	<0.150	4.700	1.500	<5.000	5.50
LC 2	<0.100	<0.250	<0.250	<2.500	<1.000	<0.150	5.300	1.100	<5.000	7.70
TB 1	<0.100	2.000	<0.250	<2.500	<1.000	<0.150	4.000	3.000	<5.000	9.00
TB 2	<0.100	<0.250	<0.250	<2.500	<1.000	0.300	5.000	1.000	<5.000	7.00
TB 3	<0.100	<0.250	<0.250	<2.500	<1.000	<0.150	11.000	3.000	<5.000	3.00
TB 4	<0.100	<0.250	<0.250	<2.500	<1.000	0.200	6.000	1.000	<5.000	10.00

BR - Braam Raubenheimer Dam; MW - Mataffin Weir; LC - Lions Club; TB - Ten Bosch Weir

TABLE 5
CONCENTRATIONS OF TRACE METALS IN ROOT AND LEAF TISSUE OF PLANTS (mg·g^{-1} DRY-MASS)

Site	As	Cd	Cu	Pb	Hg	Se	Zn	Mn	Al	Fe
MW										
roots	0.023	<0.005	0.042	<0.050	<0.020	<0.003	0.058	6.581	13.21	42.52
leaves	<0.002	<0.005	0.032	<0.050	<0.020	<0.003	0.043	4.497	8.185	27.86
C5										
roots	0.008	<0.005	0.020	<0.050	<0.020	<0.003	0.060	9.848	5.895	15.73
leaves	<0.002	<0.005	0.014	<0.050	<0.020	<0.003	0.042	4.101	0.989	3.136
C6										
roots	0.055	<0.005	0.015	<0.050	<0.020	<0.003	0.017	6.688	2.484	15.03
leaves	<0.002	<0.005	<0.005	<0.050	<0.020	<0.003	0.015	0.953	<0.100	0.736
TB										
roots	0.032	<0.005	0.028	<0.050	<0.020	<0.003	0.034	5.607	5.934	17.19
leaves	<0.002	<0.005	<0.005	<0.050	<0.020	<0.003	0.015	0.837	0.120	0.761

MW - Mataffin Weir
TB - Ten Bosch Weir

survey, of the selected pesticides, only *p,p*-DDE was found in muscle tissue of fish. These concentrations of *p,p*-DDE, as well as the percentage fat in the muscle tissue of each fish, are given in Table 6. Accumulated lipophilic pesticides are generally stored in fatty tissue, as reflected by the results of Heath (1992). This study only made use of muscle tissue, which is more relevant when estimating risk associated with human consumption. The fact that DDE, one of the more stable metabolites of DDT, was detected does not necessarily indicate a recent contamination.

TABLE 6
CONCENTRATIONS OF *p,p*-DDE (µg·kg^{-1} WET-MASS) AND % FAT RECORDED IN MUSCLE TISSUE OF FISH

Fish	*p,p*-DDE	%fat
BR1	96.7	5.6
BR2	64.7	1.3
BR3	3.9	0.11
BR4	14.9	0.48
MW1	8.9	0.39
MW2	9.4	0.21
MW3	56.8	1.1
LC1	27.3	1.8
LC2	144.0	3.0
TB1	<0.1	0.12
TB2	0.9	3.7
TB3	<0.1	1.6
TB4	181.0	7.5

BR - Braam Raubenheimer Dam;
MW - Mataffin Weir;
LC - Lions Club;
TB - Ten Bosch Weir

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