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The significance of entry routes as point and non-point sources of pesticides in small streams

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Abstract

In an agricultural catchment area in Germany we analyzed water samples from five entry routes for 2 insecticides, 5 fungicides and 13 herbicides. The sewage plant outlet and the emergency overflow of a sewage sewer contained only herbicides. In each farmyard runoff we found on average 24 g pesticides during application period, presumably caused by cleaning the spraying equipment. In comparison, the field runoff and the rainwater sewer contained less load, but also insecticides, fungicides and herbicides. The sewage plant caused 65.9% of the total herbicide load, the sewage sewer 19.8% and the farmyard runoff 12.8%. The farmyards also caused 83.7% of total insecticide and 83.8% of fungicide load. The total load of all entry routes is correlated with the amount of pesticides applied in the catchment area and the $K_{o/w}$ value for each pesticide (mult. regress. r^2: 0.82; $p<0.0001$; $n = 14$). In stream A the sewage plant caused a slight but continuous contamination by herbicides with 82% of the total load found during low-water phases. In comparison, stream B had only farmyard runoff and non-point sources, which caused high peaks of herbicide and a contamination by insecticides. Consequently, high-water phases generated 70% of the total pesticide load.

Keywords: Pesticide; Headwater streams; Surface runoff; Catchment; Farmyard seepage; Sewage plant

1. Introduction

Streams contaminated by pesticides are impaired because they can in turn pollute groundwater and the contamination can severely affect the aquatic community [1,2]. Small streams with intensively cultivated catchment areas receive non-point input of pesticides via field runoff [3–5] and field drainage pipes [6,7]. Sewage plant outlet [8], sewer overflows [9] and runoff from farmyards [10] also make a significant contribution. Less important are inputs by drift, direct spraying or from the atmosphere in precipitation.

Input by most entry routes depends on heavy precipitation, so that their contribution to stream pollution is brief and unpredictable. It is generally thought that the insecticides so introduced are mostly bound to suspended matter, whereas herbicides are transported in dissolved form [11,4]. The input of herbicides from point sources, farmyard runoff [12,13] and field drainage pipes [6,7] has been investigated. Input of insecticides in the water phase has so far been documented mainly for field runoff [3,14].

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The significance of the individual entry routes has previously been estimated by measuring the water in the streams and not by direct sampling of the entry routes themselves [13]. Our aim was to fill this gap by extensive sampling of the water flowing through all relevant entry routes in the catchment area after precipitation, and to analyze a broad spectrum of 20 pesticides. Using these data, the entry routes were compared and their contribution to pesticide contamination estimated. Another objective was to compare the contamination profiles of each stream following heavy precipitation and periods of dry weather.

2. Materials and methods

2.1. Study area

Near Viersen in the Niederrheinische Bucht in Nordrhein-Westfalen (NRW) in Germany, two small streams the Nette (stream A) and its tributary the Pletschbach (stream B), were investigated above their confluence (see Table 1).

In both catchment areas, runoff from cultivated fields and direct runoff from farmyards were identified as input sources. Additional input to stream A only originates from a sewage plant and an emergency overflow of a sewage sewer and to stream B from a rainwater sewer. Because of the sewage plant, the basic discharge in stream A is greater. Intensive agriculture prevails in both catchment areas: predominantly grain and potatoes (each 25% of the total area), then sugarbeet (19%) and maize (14%), other vegetable crops (4%) and grassland plus pasture (6%). The amount of agricultural pesticide (in the spectrum analyzed here) applied on an average during the investigation period was determined by adding up the applications by farmers from ca. 37% of the total catchment area: herbicides 1.5 kg ha^{-1}, insecticides 0.002 kg ha^{-1}, fungicide 0.18 kg ha^{-1}.

Table 1
Parameters describing the streams and their catchment area

	Stream A		Stream B	
Length (km)	4		5.7	
Flow rate (L s^{-1})	100–600		10–300	
Catchment area (ha)	1550		1080	
Surrounding soil type	Coarse loam to coarse sand		Coarse loam to coarse sand	
Slope of terrain (%)	0.8–2		0.8–4	
Land use (%)	Settlements	31	Settlements	10
	Fields	58	Fields	80
	Meadows	8	Meadows	5
	Woodland	3	Woodland	5

2.2. Sampling methods

The sampling was done in 1998 while pesticide application was most intensive, from early April to mid-July. Samples were taken from the sewage plant and its emergency overflow of the sewage sewer (only stream A), the outlet from a rainwater sewer (only stream B), farmyard runoff (3 out of 25 farmyards) and surface runoff from cultivated fields (7 out of 20 fields). Drainage pipes, as the entry route of water percolating through the ground were not investigated because they are rare in this region.

Water samples from the streams were taken during high-water and low-water phases. After dry-weather phases we used hand-sampling. When precipitation exceeded 10 mm d^{-1} we used computer-controlled water samplers described [3]. Simultaneously, passive high-water samplers were employed, in which a container filled up with water whenever the stream level rose by more than 5 cm. We assumed that the characteristic of the samples were the same for both sampler types. The 24 h precipitation was measured daily. From all available high-water-phases only those samples taken after heavy precipitation were analyzed.

In the entry routes we used various sampling methods. Runoff from cultivated fields was collected in sampling bottles at the entrance to the stream described in [14]. This sample principle was also applied to the farmyard runoff, the emergency overflow of the sewage sewer and the discharge from the rainwater sewer. From the sewage plant outlet a composite sample (100 mL h^{-1}) was taken every day by an automatic sampler.

2.3. Analysis methods

The water samples were analyzed in two different laboratories. The water samples taken from the streams after heavy precipitation and the water samples from all entry routes were analyzed at the Institute for Ecological Chemistry of the Technical University of Braunschweig. The samples were concentrated by solid-phase extraction (RP-C18) and analyzed by GC/MS similar to the method described by [3]. Two insecticides (fenvalerate and parathion-ethyl) and five fungicides (azoxystrobin (= pyroxystrobin), kresoxim-methyl, epoxiconazole, fenpropimorph, propiconazole) were analyzed. Of the 13 herbicides analyzed, atrazine and simazine are prohibited for agricultural use. Terbuthylazine, metazachlor, chloridazon, ethofumesate, metamitron, isoproturon, prosulfocarb, metribuzin, and metobromuron are actual agricultural herbicides. Bromazil (= imazalil) and diuron were also analyzed, although not used agriculturally. The detection limits reached 0.1–0.5 μg L^{-1} depending on the matrix loading. Four (5%) heavily matrix-loaded samples gave only detection limits of 0.6–

$1\,\mu g\,L^{-1}$. The detection limit for metobromuron and diuron was $1\,\mu g\,L^{-1}$.

The stream samples taken after dry-weather phases were analyzed by the Staatliches Umweltamt Düsseldorf, Germany. The samples were taken and analyzed according to the norms DIN 38407-2 (1993-02) and DIN EN ISO 11369 (1997-11). After liquid–liquid extraction the extracts were analyzed by GC/ECD. A determination limit of $0.1\,\mu g\,L^{-1}$ was reached. From the 20 agents presented in this paper this analysis included no insecticides, no fungicides and only 10 herbicidal agents (atrazine, simazine, terbuthylazine, metazachlor, chloridazon, metamitron, isoproturon, metribuzine, metobromuron and diuron).

2.4. Stream load calculation

The hydrograph curve of discharge gauges indicated that 6 h was the average duration of a flood event. Therefore the concentrations measured for a high-water phase were expressed to the respective 6 h discharge volume. Concentrations after dry-weather phases were averaged and loads were calculated using the average discharge between flood events.

2.5. Entry route load estimation

The sewage plant outlet and the emergency overflow of the sewage sewer were measured with level recorders, so that the respective pesticide loads could be estimated from the measured concentrations. The plant treats the sewage output from 23,000 inhabitants and industrial waste water equivalent of 22,000 inhabitants with multistage mechanical, biological and chemical treatments.

Field runoff could only be observed when precipitation exceeded $10\,mm\,d^{-1}$. According to models calculating the amount of effective precipitation [15] this means that at least 2% of the precipitation becomes field runoff. For load estimation we used 2% as a fixed value for all amounts of precipitation because only the first surge was sampled and is considered to be contaminated. The catchment area of each field runoff sample was estimated from a 1 : 5000 map. For the runoff from farmyards the estimated area and only the first millimetre of precipitation (first-surge approach) were used for discharge-volume calculation. For the rainwater sewer (stream B) a modified first-surge approach was used. Given the width (800 mm) and gradient (1 : 500) of the concrete tube, the level signalled by the sampler (16 cm) according to the standard formula indicated a discharge of $3600\,L\,min^{-1}$. This rate was applied to a 20-min surge to calculate the volume of contaminated water.

3. Results and discussion

3.1. Pesticides in the entry routes

The five investigated entry routes differ widely in the degree of pesticide contamination. Fig. 1 shows an overview of their contamination profiles, based on a total of 57 water samples. The mean values for the positive readings are shown together with the maximal concentration and the percentage of contaminated samples.

3.1.1. Field runoff

In the water phase of the field runoff, 19 pesticides were detected with a total load of 66.2 g during the investigation period. Samples from April were found to be either still uncontaminated or contaminated only by metribuzine. As the period of pesticide application progressed, the spectrum expanded and several pesticides were present simultaneously. The herbicides were detected at highest concentrations, with greatest frequencies. Remarkably, even herbicides not permitted in Germany were detected: atrazine and simazine as well as Diuron twice at an asparagus field. The herbicide Metazachlor was detected at a very high concentration shortly after application. The corresponding stream water sample had a low concentration, which can be explained by dilution. Insecticides and fungicides were rarely present, because the number and the amount of applications are low. Overall, 82% of the samples were contaminated with pesticide, though only small amounts were present.

High herbicide concentrations in the runoff from agricultural areas and the input to streams of insecticides bound to suspended particles have been described in depth [4,5]. Less is known about insecticides and fungicides carried in the water phase of field runoff [11,3,14].

3.1.2. Farmyard runoff

In the runoff from farmyards 17 pesticides were found, and again the herbicides dominated, occurring at extremely high concentrations and frequencies. In April, only a scattered contamination was present, e.g. isoproturon ($115\,\mu g\,L^{-1}$) or metribuzine. In June, several pesticides were detected at high concentrations: prosulfocarb ($1451\,\mu g\,L^{-1}$), metamitron ($846\,\mu g\,L^{-1}$) and ethofumesate ($266\,\mu g\,L^{-1}$). It is notable that diuron, which is not permitted as an agricultural herbicide, was present ($9.5\,\mu g\,L^{-1}$), as was atrazine in 58% of the samples, although it has been prohibited since the mid-1980's. Altogether 95% of the water samples were contaminated with at least one pesticide.

Farmyard runoff, carrying an estimated average of 24 g per farm (total: 604 g during investigation period), clearly contributes to the contamination of the streams.

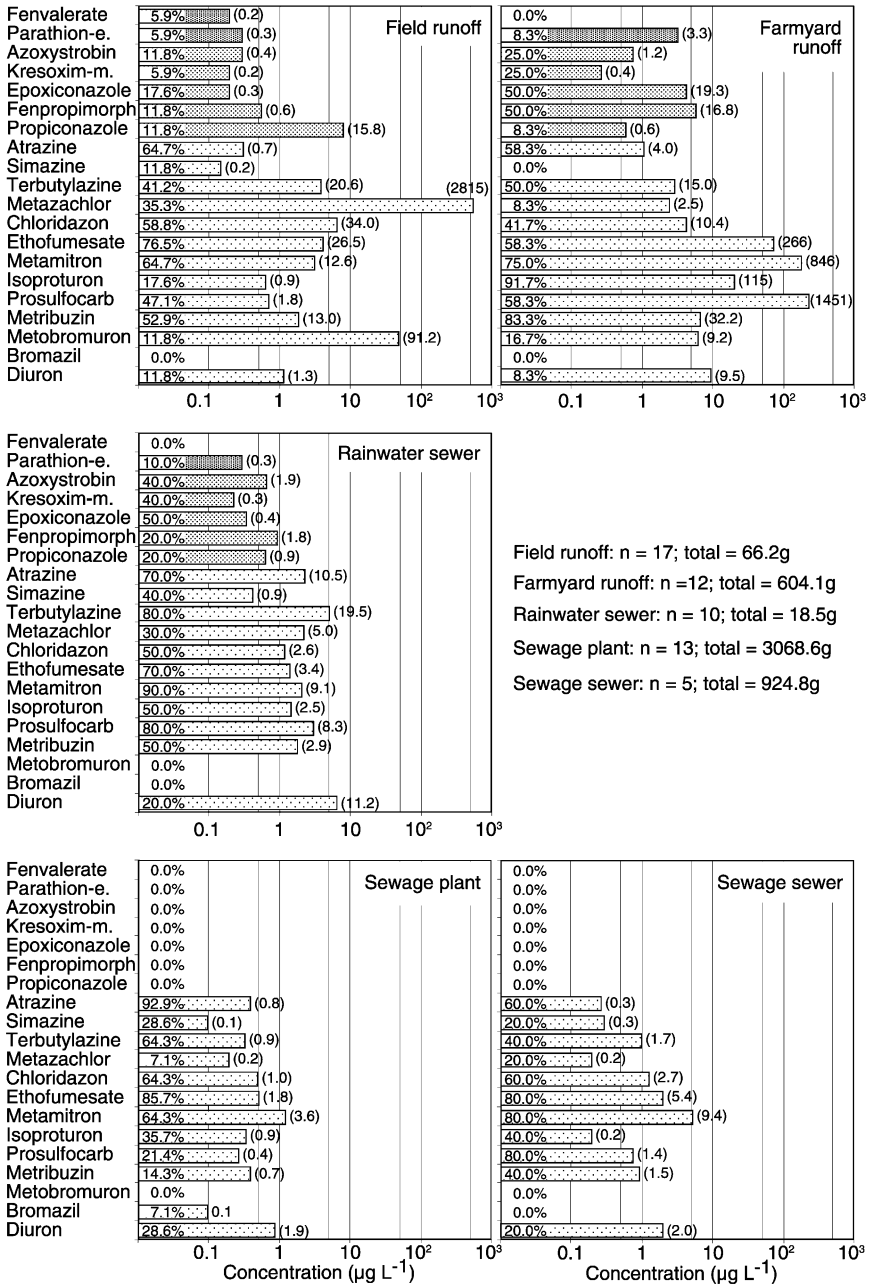

Fig. 1. Overview of the contamination of the five entry routes, showing the mean concentration for the positive readings of the pesticides (bars), maximal concentration (brackets) and the percentage of samples contaminated.

This value is well within the published range of 11–48 g [12,13]. Crucially, insecticides and fungicides also enter the streams by this route in the water phase, which has rarely been described in the literature.

3.1.3. Rainwater sewer

In the water of this entry route 17 pesticides were found. All samples were contaminated with minimally 3 and maximally 14 pesticides. The concentrations of the herbicides were particularly high. The total loading of this route was estimated at 18.5 g during the investigation period.

In the sewage systems of many small communities an effort is made to separate sewage and rainwater, so that the rainwater need not be treated. The rainwater sewer studied here collects the water drained from the eaves of buildings, the streets and the paved surfaces of a small village. Our analyses show that even such rainwater sewers can carry pesticides into streams. The spectrum of detected substances includes insecticides and fungicides, consequently, this entry route is comparable to the runoff from fields and farmyards.

3.1.4. Sewage plant

The only contaminants detected in the sewage-plant outlet were 12 herbicides with a total load of 3068.6 g during the investigation period. All the samples were contaminated with a broad spectrum (up to 11) of pesticides, of which atrazine, ethofumesate, terbuthylazine, chloridazon and metamitron were present almost continuously. The concentrations were low. From an unpublished examination by the Staatliches Umweltamt Düsseldorf, Germany we know that Diuron is presented in this sewage plant almost continuously with concentration lower than our detection limit. No insecticides or fungicides were detected, presumably because only small amounts are applied and treatment in the plant causes marked dilution and mixing, sorption and decomposition.

Sewage plants are known to introduce large quantities of herbicides to bodies of water [8,13,16]. Because of the high flow rate and great dilution their final concentrations are low, but their constant presence produces a large total load.

3.1.5. Sewage sewer

The sewage sewer carries the water entering the sewage plant. During the study period heavy precipitation caused emergency overflow seven times. Five of these inputs were analyzed. All samples were contaminated with at least one herbicide and a maximum of eleven. The concentrations and frequencies of contamination generally exceeded those for the sewage plant.

Although the emergency overflow accounts for only 4.2% of the total output volume (plant plus overflow), it contains 23.2% (924.8 g) of the total amount of pesticide introduced to stream A by the two entry routes.

The introduction of unclarified sewage to a body of water as a source of agricultural pesticides has attracted little attention till now. This input route however, has proved more significant than the sewage-plant outlet. The entry from this source can only be prevented by increasing the temporary storage capacity of the plant.

3.2. Relationship between entry route load and applied amount

By regression analysis we attempted to explain the load of each pesticide found in the entry routes on the basis of certain variables. The amount of each substance applied was determined by asking the farmers. No use was reported for parathion-ethyl, atrazine, simazine, metazachlor, bromazil or diuron. The octanol/water distribution coefficient ($K_{o/w}$ value) for each pesticide was taken from [17]. For the regression calculation both quantity variables were expressed as logarithms and the normal distribution was confirmed by the Kolmogorov–Smirnov test. Both variables are correlated with the summed loads of all entry routes for each pesticide with $r^2 = 0.43$; $p<0.0106$; $n = 14$ ($K_{o/w}$ value) and with $r^2 = 0.65$; $p<0.0005$; $n = 14$ (Log (amount applied)).

Table 2
Multiple linear regression to explain the summed loads of all entry routes by the amount applied and the $K_{o/w}$ value for each pesticide. The two independent variables are not correlated with one another ($r^2 = 0.21$; p: n.s.; $n = 14$)

Dependent variable	Log (entry routes)		
mult. r^2	0.81		
P	0.0001		
N	14		
Independent variables	**Log (amount applied)**	**$K_{o/w}$**	**Constant**
B	1.57	−0.49	−4.06
SE B	0.33	0.16	1.69
Beta	0.66	−0.42	—
P	0.0006	0.0103	0.0352

In the multiple regression shown in Table 2 they correlate with r^2 of 0.81. It was confirmed that the two variables are not correlated with one another ($r^2 = 0.12$; p: n.s.; $n = 14$).

The amount applied in the catchment region and $K_{o/w}$ value both significantly influence the loads found in the entry routes. The more important factor is the amount applied. The $K_{o/w}$ value is negatively correlated with the load detected, i.e. the stronger the tendency of a substance to bind to particles, the smaller the amount found in the water phase. The same relation to $K_{o/w}$ value was found by [18]. The solubility of a substance is strongly correlated with its $K_{o/w}$ value and does not improve the multiple regression. The half-life times could not be considered, as they have not been measured with comparable methods for all substances.

It is generally thought that chemical properties (e.g., sorption) are the crucial determinants of input in the water phase of surface runoff [4,18]. Our focus on the individual entry routes in Table 3 shows however that the amounts applied are mainly responsible for the pesticide load, for all pesticide classes considered. The contribution increases the shorter the studied entry route is and with less chance for sorption or degradation.

In no case was a multiple regression with amount applied and $K_{o/w}$ value significant. A dependence on $K_{o/w}$ value was found only for the field runoff, which is

Table 3
Linear regression to explain the loads in the individual entry routes either by the amount applied or the $K_{o/w}$ value for each pesticide

Dependent variable	Independent variable	r^2	p	B	SE B	n
Log (farmyard runoff)	Log (amount applied)	0.61	0.002	1.52	0.36	13
Log (field runoff)	Log (amount applied)	0.35	0.026	0.82	0.32	14
Log (field runoff)	$K_{o/w}$ value	0.36	0.023	−0.41	0.16	14
Log (rainwater sewer)	Log (amount applied)	0.49	0.011	0.63	0.20	12
Log (sewage plant)	Log (amount applied)	—	n.s.	—	—	7
Log (sewage sewer)	Log (amount applied)	—	n.s.	—	—	7

the only place pesticides should be applied. All other entry routes can be dramatically reduced by following good agricultural practices and careful pesticide handling. This is the same for the outlet from the sewage plant and the emergency overflow of the sewage sewer although no correlation was found here. These entry routes are caused by farmyards connected to the sewage sewer and by non-agricultural use.

3.3. Relative contribution of each entry route

We can now estimate the amounts of pesticides in each class and the percentage contributed by each entry route for the summed catchment area of streams A and B. This is done by direct event-controlled sampling in the entry routes. Measurements in the stream would not reveal small amounts of contamination, owing to dilution in the large volume of water.

In similar comparative studies sewage plants have been viewed as the most important source [13]. Here, however, as Table 4 shows, this applies only to herbicides and, remarkably, an additional ca. 20% is contributed by the emergency overflow of the sewage sewer. The reasons for this large contamination are farmyards connected to the sewage sewer and non-agricultural use. Insecticides and fungicides could not be detected. For these the most important input route is farmyard runoff.

For the smaller stream B, with no sewage plant or sewage sewer in its catchment area, the significance of non-point sources was confirmed. Here the farmyard runoff accounts for 89.8% and field runoff for 7.5% of the herbicide input. Farmyard runoff is the most important input to stream B for all classes of pesticides. This reflects the typical structure of the landscape, throughout which individual farms are scattered.

3.4. Pesticide concentrations in the streams

From the two streams a total of 21 water samples were taken during high-water phases and 12 after dry-weather phases. The contamination found in each stream was consistent with the findings for its individual entry routes. The profiles are typical of a stream with a sewage plant in its catchment area (A) and one with a catchment area, having mainly non-point sources (B).

Table 4
Proportion of total pesticides in the three pesticide classes contributed by each entry route, for the whole catchment area of streams A and B. Also shown are the estimated absolute amount (g) and the number of pesticides analyzed per class

	Insecticides ($n = 2$)	Fungicides ($n = 5$)	Herbicides ($n = 13$)
Total load (g)	0.32	9.82	4656.4
Field runoff (%)	3.9	7.7	1.1
Farmyard runoff (%)	83.7	83.8	12.8
Rainwater sewer (%)	12.4	8.5	0.4
Sewage plant (%)	n.d.	n.d.	65.9
Sewage sewer (%)	n.d.	n.d.	19.8

Stream A was contaminated mainly by herbicides. Every sample showed contamination. In April, isoproturon ($6.7\,\mu g\,L^{-1}$) and chloridazon ($1.2\,\mu g\,L^{-1}$) were unequivocally detected and in June the most important contaminants were metamitron ($5.1\,\mu g\,L^{-1}$) and ethofumesate ($2.1\,\mu g\,L^{-1}$). As Fig. 2 shows, no insecticides or fungicides were detected. The contamination profile of stream A reflects the input from the sewage plant and its emergency overflow, with relatively low concentrations and a dominant herbicide component. Concentration peaks from other entry routes are diminished by dilution in the large volume of water. After dry-weather phases the contamination is almost constant at a low level.

The contamination profile of stream B was different, with a greater variety of pesticides and higher concentrations resulting from precipitation-induced inputs (e.g., diuron at 4.3 and $2.4\,\mu g\,L^{-1}$ and atrazine at $2.5\,\mu g\,L^{-1}$). The peak concentrations were $31.1\,\mu g\,L^{-1}$ for terbuthylazine and $14.5\,\mu g\,L^{-1}$ for metamitron. Relatively large amounts of fungicides were present, and a high level of contamination by the insecticides fenvalerate and parathion-ethyl was observed. After dry-weather phases the pesticide load in stream B was distinctly lower, but even then all the water samples were contaminated.

The volume flow rate after dry-weather phases is 7-fold higher in stream A than in stream B and the total

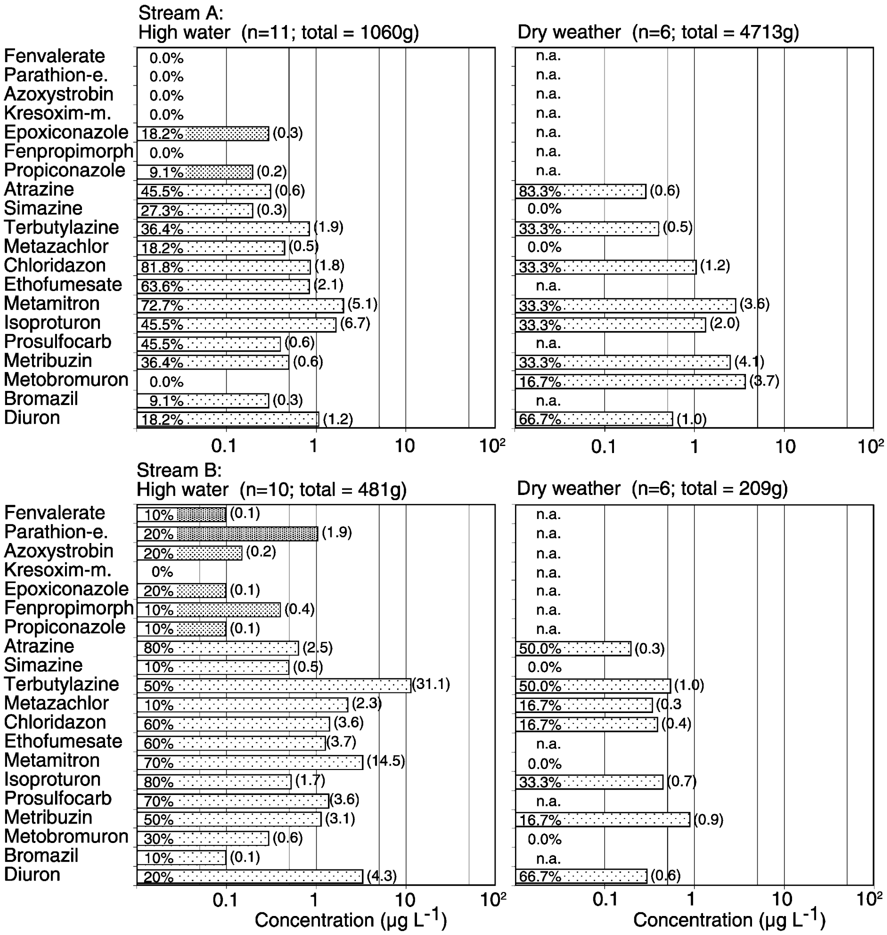

Fig. 2. Overview of the contamination of the water samples from the two streams during high water and after dry weather, showing the mean concentration for the positive readings of the pesticides (bars), the maximal concentration (brackets) and the percentage of samples contaminated.

contaminant load is also higher, by a factor of 8.4. On average, therefore, the concentrations are higher in stream A. In high-water phases, however, the contaminant load in stream A is only 2.2-fold higher than in stream B, which therefore contains distinctly higher concentrations.

In stream B 70% of the estimated load is introduced during brief events with a high contamination rate. In stream A continuous contamination of the steady volume flow during dry weather accounts for 82% of the estimated contaminant load.

The comparison shows that a stream with a sewage plant in its catchment area is continuously contaminated by herbicides, and any inputs of insecticides and fungicides are masked by dilution in the large volume of water. Streams with catchment areas including non-point sources are characterized by marked contamination peaks. During these events insecticides can be detected.

4. Conclusion

- A sewage plant can be the quantitatively most important source of herbicides, contaminating a stream almost continuously. In such streams the low-water phase accounts for most of the contaminant load and non-point sources are insignificant.
- Farmyard runoff can be the second most important source of herbicides. After heavy precipitation farmyard runoff and non-point sources (field runoff) produce contamination peaks and account for most of the contaminant load of small streams.
- Both the emergency overflow of a sewage sewer and the outflow from a rainwater sewer contaminate streams with pesticides.
- The amount of a pesticide in the precipitation-induced entry routes is determined primarily by the amount applied to the catchment area. In the runoff from farmyards the pesticides in current use are

detectable. Hence cleaning of the spraying equipment should be done only on the fields or near the manure collection pit.

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