Preliminary assessment of fluxes of priority pollutants in stormwater discharges in two urban catchments in Lyon, France

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Abstract: In urban catchments, diffuse stormwater discharges from both separate and combined sewers contribute significantly to the contamination of water bodies by numerous pollutants, including the priority substances listed in the European Water Framework Directive. Whereas concentrations and loads of traditional pollutants (suspended solids, BOD, COD, nutriments…) in stormwater discharges are well documented, very few information is available on the presence, the concentrations and the loads of priority substances. The ESPRIT project aims to identify, evaluate, characterise and later on model fluxes of priority pollutants in urban stormwater discharges. Two sites were chosen for measurement campaigns: Ecully (residential site, combined sewer) and Chassieu (industrial site, separate sewer). In total, 26 metals and 36 organic pollutants (in particulate and dissolved phases) are measured in stormwater discharges at the catchment outlets. Some results obtained for a set of rainfall events monitored since the beginning of 2008 are presented. Significant inter-site and inter-event variability in concentrations and specific fluxes (g/active ha) are observed for metals and organics pollutants, particularly for pesticides. **Keywords:** combined sewer system, priority pollutants, separate sewer system, stormwater, Water Framework Directive

INTRODUCTION

Stormwater has been recognised as one of the major pathways for spreading of nonpoint-source pollutants in urban environment (Scholes *et al.*, 2008). Management of stormwater pollutant loads has thus become an increasingly important environmental concern. Contaminants in sewer systems derive from a number of sources (Fig 1): materials from wet and dry atmospheric deposition, traffic-related activities (tire wear, vehicle break emissions, fluid leakages), or released from roofs and buildings, may be flushed by rainfall and transferred into sewer systems (Mason *et al.*, 1999; Davis *et al.*, 2001). In addition, sewage and in-sewer deposits can also represent a significant contribution to storm weather pollutant loads (Gromaire *et al.*, 2000). Urban storm weather discharges include waters from i) wastewater treatment plant outlets (mixture of sewage and stormwater), ii) overflow facilities (mixture of untreated sewage and stormwater) and iii) stormwater outlets (generally untreated stormwater), during rain events and during the subsequent dry weather period during which the sewer system does not function under nominal dry weather conditions.

The European Water Framework Directive (WFD) (EC, 2000) establishes objectives for chemical and ecological quality of surface and ground water bodies. In this context, reducing urban stormwater discharges is one of the major stakes for operators of urban water systems. According to the WFD, the good chemical status of a water body is reached if concentrations of a set of priority substances (41 pollutants including 4 metals, organic substances and pesticides) in water or biota (for 3 substances) are below Environmental Quality Standards (EQS) (EC, 2008). Reaching this ambitious objective will be very difficult "without major changes of ours paradigms as regards to drinking water, sanitation, agriculture and environmental policy" (IFEN, 2006).

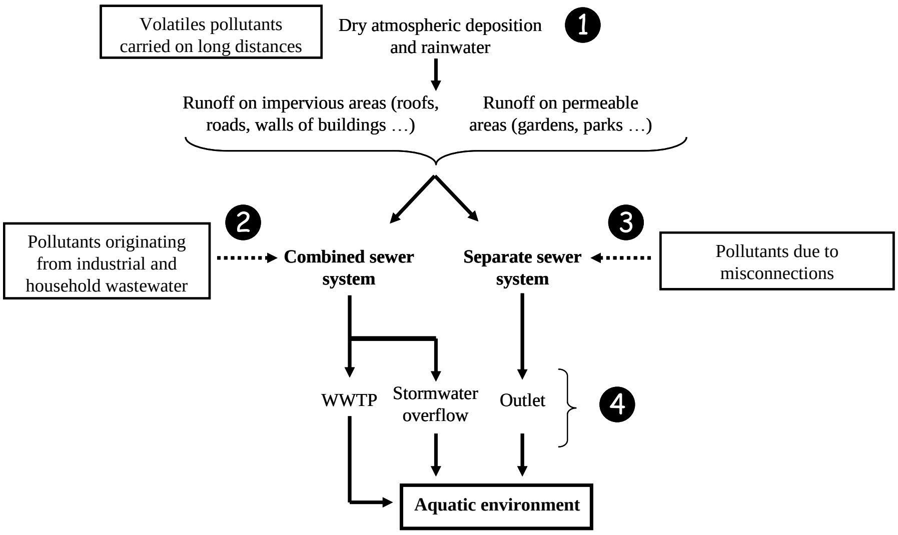

Fig 1. Contribution and major pathways of priority substances transport into surface waters in urban catchments: ❶ Atmospheric deposition on surface: dry atmospheric deposition and rainwater; ❷ Household and industrial wastewater and sediments deposited during dry weather; ❸ misconnections and ❹ during storm events, discharge from overflows and outlets into the aquatic environment.

Within urban catchments, data on diffuse sources of priority pollutants are still limited. Previous stormwater research has focused mainly on nutrients, heavy metals, organic and particulate matter, and polycyclic aromatic hydrocarbons (PAHs) (Brown and Peake, 2006). Knowledge concerning emission, occurrence and fate of other specific organic contaminants in urban runoff remains scarce (Rule *et al*., 2006). In this context, the ESPRIT project presented in this paper aims to qualitatively and quantitatively estimate the fluxes of priority substances in stormwater discharges. Monitoring and sampling in two contrasted urban catchments (one residential combined system and one industrial separate system) enables estimating and comparing pollutant fluxes. Some preliminary results are presented in this paper.

MATERIALS AND METHODS

Study sites

Monitoring campaigns were carried out at the outlet of two experimental catchments (Ecully and Chassieu) in Greater Lyon. The main characteristics of both sites are presented in Table 1. Both sites are equipped and monitored as part of the the Field Observatory on Urban Hydrology (OTHU) (www.othu.org). At catchment outlets, sets of sensors allow continuous monitoring (2 minutes time step) of quantitative (flow velocity, water depth) and qualitative (pH, conductivity, turbidity, temperature) parameters.

Table 1. Main characteristics of the two experimental sites (Lyon, France)

	Ecully	**Chassieu**
Location	West of Lyon	East of Lyon
Total surface (ha)	245	185
Land use (% of total surface)	residential	industrial
Impervious surfaces	34	72
Urban green spaces	60	20
Agricol area	5	4
Natural area	1	4
Active surface (ha)	60	54
Land slope (%)	2	0.4
Percent of impervious area	42	75
Sewer type	combined	separated

Sampling of urban stormwater discharge

Both catchments are equipped with similar specific sampling devices. Stormwater samples are collected at the outlet by means of a Bühler 4010 refrigerated automatic sampler with volumetric vacuum pump and a set of twenty-four 1 L glass bottles. The sampler is operated with constant time step and fixed volume accounting for weather conditions. For each sampled storm event, a flow proportional event mean sample is reconstituted manually according to flow and conductivity measurements. Event mean concentrations (EMCs) of pollutants are then measured by laboratory analyses of this event mean sample.

Analytical methods

Metals

In total, 26 metals (4 metals of the WFD - Cd, Pd, Ni and Hg – and 22 other metals) are systematically analysed in both dissolved and particulate phases by inductively coupled plasma mass spectrometry (ICP-MS, Thermo X7 series II) according to standard methods (ISO, 2004, 2005). The limits of quantification LOQs for the dissolved phase vary between 0.01 µg/L (for Cd) and 5 µg/L (for Al and Ba). Typical expanded uncertainties (i.e. 95 % confidence intervals) vary between 8 and 20 % of the measured value depending on the element.

Organic pollutants

A specific multi-residue method by gas and liquid chromatography coupled to mass spectrometry has been developed and validated for the analyses of 36 priority organic substances (in the dissolved phase) listed in the WFD in various water matrices (Barrek *et al.*, 2009), including 17 pesticides, 8 PAH and 10 others substances (alkylphenols, volatile organic compounds, …). Limits of detection (LOD) vary between 0.2 ng/L (pentachlorophenol) and 3 ng/L (octylphenol) for LC-FLD-MS/MS analysis. Limits of quantification (LOQ) for GC-MS analyses are between 13 and 67 ng/L for pp DDT and endosulfan A, respectively (see Table 5). The method is presently under validation for the particulate phase: this paper gives thus only results in the dissolved phase.

Quality control

Strict protocols for equipment cleaning and checking (automatic sampler, automatic atmospheric dry deposits and rain water sampler, etc.) have been applied to avoid any risk of sample contamination. Blank tests are carried out periodically to detect any possible contamination of samples and of the sampling chain. The event mean samples are rapidly transported (i.e. in less than 12 hours) to the laboratory and then transferred into polyethylene bottle for metals analyses, into Teflon bottle (FEP) for Hg analyses and into glass bottle for organic pollutants analyses.

More information about materials and methods (especially sampler for dry atmospheric deposits and rain water, analytical methods, etc.) can be found in Dembélé *et al*. (2009).

RESULTS AND DISCUSSION

Rain event characteristics

In this paper, results are presented for a selection of 10 rain events with wide spread characteristics (Table 2) monitored between March 2008 and January 2009.

Table 2. Rain event characteristics (average for 10 rain events)

	Rain depth (mm)		Mean intensity (mm/h)		Maximum intensity over 6 mn (mm/h)		Rain duration (hh:mm)		Dry weather period (hh:mm)	
Sites	Chassieu	Ecully	Chassieu	Ecully	Chassieu	Ecully	Chassieu	Ecully	Chassieu	Ecully
Minimum	0.7	1.4	0.2	0.3	1.5	2.9	1:14	1:40	8:17	4:56
Maximum	38.2	37.4	1.8	2.2	74.7	48.6	1:18	23:16	12:29	16:35
Mean	3.5	4.5	0.5	0.9	3.2	5.5	12:41	8:03	12:09	3:15

Metals

Dissolved vs. particulate fractions

The distribution of metals between dissolved and particulate phases are very variable depending on both the metals and the catchment. In Chassieu, barium and strontium are mainly present in the dissolved phase, while aluminium, iron and titanium are mainly in the particulate phase. In Ecully, a similar distribution is observed, except for manganese and titanium which are more present in the particulate phase compared to Chassieu. In Ecully (combined sewer system), 75 % and 73 % respectively of copper and zinc are in particulate phase, which is similar to the results obtained by Kafi-Benyahia (2006) in the Marais catchment in Paris drained by a combined sewer system (resp. 96 % and 88 %). In both catchments, cadmium and lead are present mainly in the particulate phase (respectively 78 % and 86 % in Ecully), whereas approx. 50 % of nickel are in the particulate phase.

Site variability

Table 3 shows the average values of EMCs (in brackets) as well as minimum and maximum EMC values. A significant spatial variability is observed. For all metals, total EMCs in Chassieu are higher than in Ecully, excepted for bore. For example, nickel EMCs in Chassieu are more than two times higher than in Ecully (8 µg/L and 3 µg/L respectively). Chassieu is thus clearly more contaminated by metals than Ecully. This can be explained by the industrial activities present in Chassieu (transport, metallurgy, etc.). Kafi-Benyahia (2006) also showed a predominant role of land use in metal loads transported during wet weather in the Paris central area.

Table 3. Event mean concentrations (EMCs) (in µg/L) of several metals in Chassieu and Ecully

	Al	B	Cd*	Cu	Fe	Ni*	Pb*	Ti	Zn
Chassieu	846 - 12 354 (2 022)	21 - 327 (66)	0.1 - 0.8 (0.2)	17 - 74 (22)	784 - 7 781 (2 098)	4 - 21 (8)	2 - 37 (8)	21 - 229 (58)	171 - 667 (249)
Ecully	0 - 22 065 (767)	30 - 128 (64)	0.04 - 0.41 (0.12)	20 - 61 (40)	536 - 10 741 (724)	2 - 10 (3)	2 - 39 (5)	27 - 232 (46)	75 - 338 (138)

* : priority metals

EMCs measured in both catchments have been compared to values reported in the literature. The later values show a huge dispersion, due to multiple factors like roof material, land use, traffic, event characteristics, sampling protocols and analytical methods used. We can observe in Table 4 that EMC values in Ecully are frequently close to lowest literature values, whereas in Chassieu EMC values are higher and more comparable with literature values.

Table 4. Total Cd, Cr, Cu, Ni, Pb and Zn event mean concentrations in Ecully and Chassieu - comparison with some average values of reported in the literature (in µg/L)

	Land use	Cd	Cr	Cu	Ni	Pb	Zn
		Combined sewer overflows					
This study, Ecully	residential	0.1	2	40	3	5	138
Gasperi (2008)	Paris *intra-muros*	<	<	68	<	39	682
Gromaire (2000)	dense town center	1.5	/	117	/	211	1 530
Garnaud (1999)	dense town center	1.5	/	114	/	215	1 639
Saget (1994)	7 various sites	35	/	/	/	922	3 997
		Separate stormwater discharges					
This study, Chassieu	industrial		6			8	249
Taebi et Droste(2004)	mixed area		/			314	453
Smullen (1999)	28 various sites		/			67	162
Saget (1994)	7 various sites	21	48	/	452	994	1 685
Levy et Lara(1992)	residential		/			3100	3680
Granier (1990)	residential		15			142	733

Inter-event variability

Fig 2 shows total loads (dissolved + particulate) of 3 priority metals (Cd, Ni and Pb). Inter-event variability appears very important. In comparison to Gromaire-Mertz (1998), total Pb loads measured in the Marais catchment vary from 5 to 38 g/active hectare; whereas in Ecully they vary from 0.32 to 10.77 g/active hectare. This difference can be mainly explained by the corrosion of metallic roofing materials in the Marais catchment where Zn and Pb are predominant and used for half of the roof area in the catchment (Gromaire *et al*., 2000). Moreover, loads measured for two events in October appear significantly higher than the other ones, without any obvious explanation.

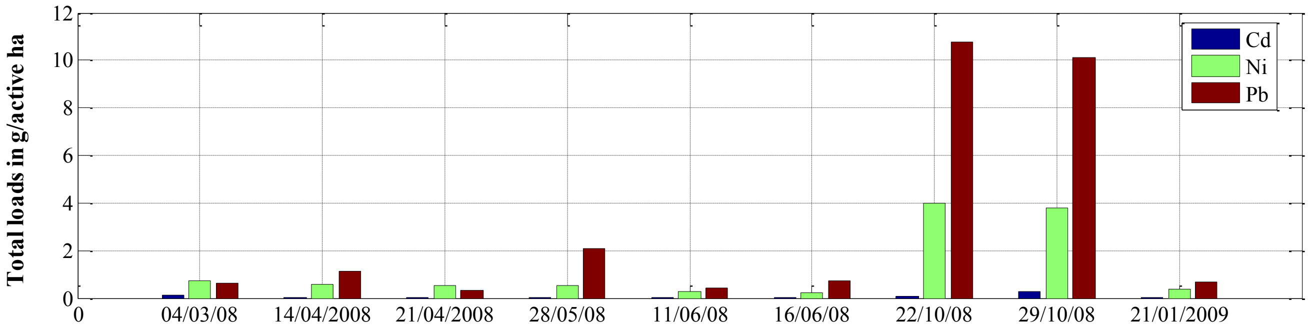

Fig 2. Total loads (dissolved + particulate) of cadmium, nickel and lead (in g/active ha) in Ecully for 9 events

Organic pollutants

Pesticides

Table 5 presents the mean EMC values measured in both sites. Most pesticides went undetected in the dissolved phase in most samples. Among the 10 detectable pesticides, only diuron and simazine were detected in all samples for both catchments. From Table 5, we can clearly observe that the mean EMCs of atrazine (forbidden since 2003), chlorfenvinphos and diuron in Ecully are significantly greater than those measured in Chassieu. For example, diuron mean EMC in Ecully is 79 ng/L and 23 ng/L in Chassieu. Diuron is commonly used for road and railway maintenance, municipal and private parks. These results are in agreement with literature values. Gasperi *et al.* (2008) measured high total (dissolved + particulate) diuron concentrations (1 400 ng/L) during wet weather in a combined sewer system in Paris *intra muros*. Blanchoud *et al.* (2004) observed that 80 % to 100 % of the diuron applied on impervious surfaces could potentially be remobilized during storm events.

Agricultural pesticides such as isoproturon or simazine (which is forbidden since October 2003) are detected in Chassieu. The quantification of simazine illustrates the persistence of this substance in the environment. The sources of other pesticides like isoproturon are probably agricultural areas neighbouring the catchment.

Table 5. Event mean concentration of dissolved pesticides (in ng/L) in urban stormwater discharges in Chassieu and Ecully

	LOQ (ng/L)	Chassieu (n=10)			Ecully (n=10)			Gasperi et al (2008) - CSO
			Range			Range		
		Mean	Min	Max	Mean	Min	Max	Median
Alachlor	9	0	0	<	0	0	<	<
Atrazine	1	1.2	0	2.8	2.1	0.9	3.1	<
Chlorfenvinphos	0.5	0.1	0	1.0	0.6	0	1.3	<
Chlorpyrifos	53	ND	ND	ND	ND	ND	ND	<
Diuron	6	23	11	43	79	18	257	1400
Endosulfan A	67	0	0	<	ND	ND	ND	<
Hexachlorobenzene	36	ND	ND	ND	ND	ND	ND	
α Hexachlorocyclohexane	35	ND	ND	ND	ND	ND	ND	<
β Hexachlorocyclohexane	35	ND	ND	ND	ND	ND	ND	<
γ Hexachlorocyclohexane	35	ND	ND	ND	ND	ND	ND	<
δ Hexachlorocyclohexane	35	0	0	<	ND	ND	ND	
Isoproturon	2	34	2	134	1.0	0	5.8	<
Simazine	0.2	2.3	0.4	5.1	2.8	1.7	4.8	
Trifluraline	45	ND	ND	ND	ND	ND	ND	
Aldrine	32	0	0	<	0	0	<	
Dieldrine	28	ND	ND	ND	ND	ND	ND	
Endrine	42	ND	ND	ND	ND	ND	ND	
Isodrine	49	0	0	<	ND	ND	ND	
op DDT	30	ND	ND	ND	ND	ND	ND	
pp DDT	13	ND	ND	ND	ND	ND	ND	

< : below the limit of quantification (LOQ)
ND : no detected
CSO : Combines Sewer Overflows

Organic pollutants loads measured for 9 storm events are presented on Fig 3. Like for metals, inter-event variability is very important. Variability for pesticides, e.g. diuron (applied from April to October) or isoproturon (applied from October to January), is likely related to application periods: their detection in samples occurs during of just after application periods (for example isoproturon is used between October and January (GRAP, 2004) on winter cereals: EMC = 46.51 mg/active hectare on 22/10/2008). The presence of agricultural pesticides (atrazine, isoproturon or simazine) in the Chassieu industrial area could be due to a contribution, via dry atmospheric deposition and rain water, from the agricultural areas neighbouring the catchment. The non quantification of many pesticides, such as pp DDT or γ hexachlorohexane (lindane), is not surprising since most of them are now banned or have been removed from the French market.

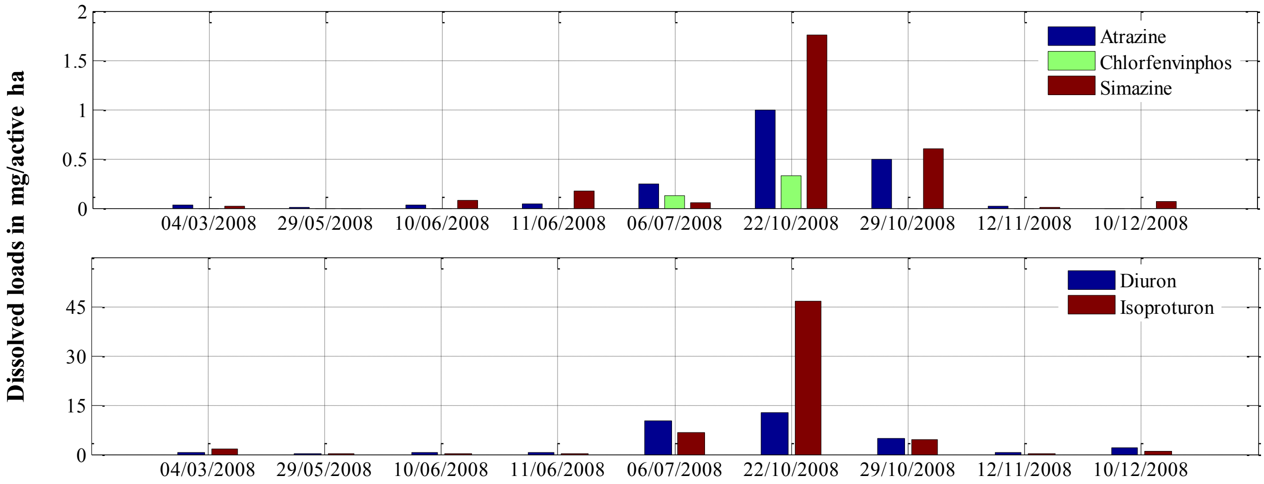

Fig 3. Loads of dissolved pesticides (in mg/active ha) in Chassieu for 9 storm events

Alkylphenols

The presence of these substances in wastewater may primarily result from the degradation of alkylphenol polyethoxylates, which are widely used as non-ionic surfactants. In Europe, alkylphenols polyethoxylates are indeed used in numerous industrial, commercial and laboratory detergents, some pesticide formulations and household applications such as detergents, emulsifiers and solubilisers. Their presence might also be linked to their direct use as plasticizers in plastics (Gasperi *et al.*, 2008). Nonylphenol is banned since 2005 in household and industrial detergents, cosmetic products and pesticide formulations.

However, no data could be found in the literature for these alkylphenols in wet weather discharges, except only in the recent study of Gasperi *et al.* (2008). Table 6 shows the EMCs of alkylphenols measured in both catchments. No difference between the two sites is observed.

Table 6. Event mean concentration of dissolved alkylphenols (in ng/L) in urban stormwater discharges in Chassieu and Ecully for 10 storm events

	LOQ (ng/L)	Chassieu (n=10)			Ecully (n=10)			Gasperi et al (2008) - CSO
			Range			Range		
		Mean	Min	Max	Mean	Min	Max	Median
Nonylphenol	30	272.8	132	460	283	69	743	/
para-ter-octylphénol	15	125	<	232	124	<	186	200

< : below the limit of quantification (LOQ)
ND : no detected
CSO : Combines Sewer Overflows

Fig 4 presents the inter-event variability for the dissolved loads of two alkylphenols: nonylphenol and para-ter-octylphenol. We observe larger flows of nonylphenol from July to December, with a mean flow of 45.61 mg/active hectare; whereas during the period of January to June, measured mean load is 8.21 mg/active hectare. Para-ter-octylphenol flows vary from 0.41 to 12 mg/active ha. A study over a longer period will allow a better interpretation of these results.

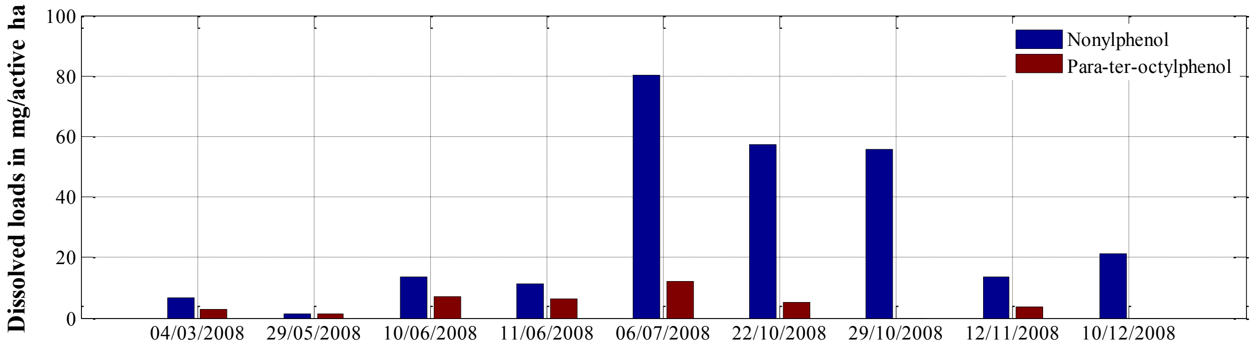

Fig 4. Loads of dissolved alklyphenols (in mg/active hectare) in Chassieu from March to December 2008 for 9storm events

CONCLUSION

The ESPRIT project started in March 2007. All sampling equipments are installed since February 2008 on two experimental sites in Ecully (residential area; combined sewer system) and Chassieu (industrial area; separate sewer system) (Lyon, France) to evaluate WFD priority pollutants event mean concentrations and loads. In total, about twenty stormwater samples for both sites have been collected. The following main trends are observed:

- A very significant variability between sites of mean total loads (dissolved + particulate) of metals, Chassieu being more contaminated by metals than Ecully;
- A similar variability is observed for organic pollutants in the dissolved phase, especially for pesticides. The difference between Ecully and Chassieu can be explained by :
    - the utilization of pesticides like diuron which is commonly used in road and railway maintenance, municipal and private parks;
    - the contribution of agricultural areas neighbouring the Chassieu catchment for agricultural pesticides like isoproturon;
- The inter-event variability is also very significant in each site for both metals and pesticides concentrations and loads per active hectare.

Long term campaigns are necessary to reliably evaluate annual pollutant loads. Moreover, i) an assessment of the respective contributions of dry atmospheric deposition, rain water, catchments surfaces and wastewater (only in combined sewers) to wet weather discharges and ii) a detailed characterisation of the particulate phase (grain-size distribution, settling velocities and X-ray microscopic analyses) will be made in the project.

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