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Geochemical assessment of soils in Roma Mahalla, Mitrovica, Kosovo: implications for the proposed resettlement of families presently living in the Osterode and Cesmin Lug Camps.



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Geochemical assessment of soils in Roma Mahalla, Mitrovica, Kosovo: implications for the proposed resettlement of families presently living in the Osterode and Cesmin Lug Camps

Report for the Foreign and Commonwealth Office by:

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Glossary

Element abbreviations used in this report

Pb	lead
Zn	zinc
Cu	copper
Cd	cadmium

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1 Introduction

Prior to the 1999 conflict in the former Yugoslavia, an 8,000 strong Roma community lived in Roma Mahalla, a suburb of Mitrovica, northern Kosovo. During and following the conflict, the residents of Roma Mahalla fled to the north Mitrovica region, fearing violence from Albanians who viewed them as "Serb collaborators" (Human Rights Watch, 2009). The internally displaced people (IDP) established camps at Cesmin Lug, Kablare (barracks adjacent to Cesmin Lug), and Leposavic (45 km NW of Mitrovica) (Figure 1). Unfortunately, the two camps in Mitrovica (Kablare and Cesmin Lug) were not only built on land contaminated by heavy metals, but they were also located adjacent to smelter waste heaps and downwind of the Zveçan lead mining/smelting complex and the Zharkov Potok tailings pond (800 m to the northeast) (Figure 1).

As early as 2000, concerns were raised about the health of the Roma communities living in the Cesmin Lug and Kablare camps (KMEG, 2009). Random blood testing of IDPs in August/September 2000 revealed "dangerous levels of lead poisoning. The lead levels were so high in some of the children that the Belgium lab doing the analysis asked for the tests to be redone. The lab had never seen such high lead levels. The second tests confirmed the first tests." (KMEG, 2009). Since these initial tests, numerous other health surveys have been conducted involving the analysis of blood (WHO, 2004), hair (Runow, 2005), plants (Riccobono et al., 2004; Borgna et al., 2009), water (Behrami et al., 2008) and sediment (Riccobono et al., 2004). As a result of the increasing awareness of the severity of metal contamination in the camps, the World Health Organisation (WHO, 2004) presented a report to UNMIK (United Nations Mission in Kosovo) that recommended closure of the existing camps and the establishment of new camps on more suitable land. Therefore, in 2006, and with the support of UNMIK, 150 families were moved from the Zitkovac and Kablare camps to the newly established Osterode Camp for 'decontamination' treatment. However, the Osterode camp itself proved to be too contaminated for treatment to be effective.



Figure 1: The Mitrovica study area showing the location of the camps and the principal sources of metal contamination.

In the light of the ongoing metal contamination problems at Osterode and Cesmin Lug, an urgent need arose to find a permanent and suitable home for the Roma community. Roma Mahalla has now been identified as a suitable site, but in order for reconstruction to commence the site needs to be declared, or made, safe for the IDP returns process to be successful. There is a concern that the families in the existing Osterode and Cesmin Lug camps are not relocated to an area that has the same soil contamination problems as the existing camps. Although a number of studies of soil quality in the Roma Mahalla area have been conducted to date (Riccobono et al., 2004; Directorate for Agriculture and Environment within Mitrovica

(DAEM), 2008), hitherto there has been no single study that has surveyed the entire proposed development site or synthesised all previously published/unpublished data. To tackle these issues, Fluvio was commissioned by the British Foreign and Commonwealth Office, and Post Telecommunications Kosovo, to address three specific objectives:

- 1. To prepare a report that identifies safe and unsafe areas (in terms of contaminant metal concentrations) in Roma Mahalla, and identify the possible sources of contamination.
- 2. To recommend, if appropriate, remedial action to improve soil quality.
- 3. To present the findings of the study to community groups and all interested stake holders.

This report directly addresses objectives 1 and 2; objective 3 will be completed when Fluvio presents the findings of this investigation to community groups and interested stake holders in March 2010.

2 Field sampling and laboratory analysis procedures

To address Objective 1, soil, river water, drinking water and mining/industrial waste samples were collected from Roma Mahalla and from sites in and around Mitrovica. Table 1 summarises the type and location of samples collected and the analysis methods used; all samples were collected between December 15th and 17th 2009. The UTM coordinates of all sample sites were recorded using a Garmin 60CSx GPS unit.

2.1 Soil sampling and analysis

In Roma Mahalla, a total of 171 soil samples were collected from the c. 4.6 ha plot of land designated for the housing project (Figure 2). Sample sites were located on a series of SSE-NNW orientated transects; the 15 transects were placed 30 m apart and samples on each transect were separated by c. 30 m. At each sample point up to three separate soil samples were collected (Table 1; Figure 2): (i) surface soil (0-

10 cm), (ii) shallow sub-surface soil (10-30 cm), and (iii) deep sub-surface soil (30-50 cm). The surface soil samples comprised a composite of 10 sub-samples collected using a stainless steel trowel within a 4 m² area, the 10-30 cm and 30-50 cm sub-soil samples were a composite of 2-5 subsamples collected using a stainless steel Edelman soil auger. Additional surface soil samples were collected from the Osterode (n=4) and Cesmin Lug (n=3) camps to assess if the degree of metal contamination was higher in these areas due to their close proximity to the Zharkov Potok tailings pond, the Zveçan Pb (lead) smelter and Gornje Polje waste dumps (Figure 3).

		4	Mode of analysis		
Sample type	Number of		Field	Laboratory	
	samples		XRF	AAS and /or	
				MS-ICP-MS	
0-10 cm soil	68	RM, CL, O	\checkmark	\checkmark	
10-30 cm soil	58	RM, O	×	\checkmark	
30-50 cm soil	56	RM, O	×	\checkmark	
Soil profiles (n=3)	9	RM	\checkmark	\checkmark	
Mine tailings	3	Zharkov Potok	\checkmark	\checkmark	
Smelter waste	5	Gornje Polje	\checkmark	\checkmark	
Industrial waste	4	Chemical plant	\checkmark	\checkmark	
House dust	7	RM, CL, O	×	\checkmark	
River water	1	Ibar	n/a	\checkmark	
Domestic water	6	RM, CL, O	n/a	\checkmark	

Table 1: Summary of samples collected and modes of analysis employed.

¹: RM = Roma Mahalla, CM = Cesmin Lug, O = Osterode



B)





Figure 2: Location of soil sample sites in Roma Mahalla: A) surface (0-10 cm), B) shallow sub-surface (10-30 cm), C) deep sub-surface (30-50 cm), and D) vertical soil profiles.



Figure 3: Location and number of ancillary samples (soil, river water, river channel sediment, house dust, mine tailings, industrial waste) collected in Roma Mahalla and the wider Mitrovica area.

At each soil sampling site, surface geochemistry was also determined using a NITON XIt792 XRF unit. The XRF unit was used to identify Pb concentrations and possible 'hotspots' in the field, so that the soil sampling strategy could be adjusted if necessary. The unit was operated in bulk soil mode and XRF readings were recorded for a minimum of 60 seconds; if after 60 seconds the analytical precision for Pb was less than 10% then the data recording was stopped, if the precision exceeded 10% then data recording was continued until the 10% precision level was

reached (NB data recording was stopped after 120 seconds even if the desired 10% analytical precision had not been achieved).

Finally, at Roma Mahalla the sub-surface geochemistry in three c. 1 m deep soils profiles was measured with the NITON XRF unit (Figure 2D). Measurements were taken at 10 cm depth intervals on the cleaned faces of three pits that had been dug for the disposal of domestic waste; the same precision analysis protocol was used as described above for the surface samples. In addition, soil samples (at 10 cm depth intervals) were collected from soil profile A (Figure 2D) for subsequent laboratory analysis. In this report, the XRF data are only used to show Pb concentrations in two vertical soil profiles (see Section 4.1), all other presented geochemical data are derived from either AAS or MS-ICP-MS analyses.

All soil samples were air-dried, disaggregated in an agate pestle and mortar and sieved through a stainless steel mesh to isolate the <2000 μ m soil fraction usually utilised in the analysis of soil geochemistry. Samples were then digested for 1 hour in 70 percent HNO₃ at 100°C and analysed for their Pb (lead) and Zn (zinc) content (mg kg⁻¹) using an Atomic Absorption Spectrophotometer (AAS), and their for Cd (cadmium) and Cu (copper) content (mg kg⁻¹) by MS-ICP-MS.

2.2 River sediment, mining/industrial waste and house dust sampling and analysis

A suite of ancillary samples was collected to help identify potential (i) sources (e.g. mine tailings, smelter waste, industrial waste), (ii) modes of transport (e.g. wind, water), and (iii) sites of deposition (e.g. river channel sediment, house dust) of metal contaminants, particularly Pb (Table 1, Figure 3). Source samples of tailings (from the Zharkov Potok tailings pond), smelter waste (from the Zveçan smelter), industrial waste (from the site of the former battery factory and Zn electrolysis plant) and river channel sediment (from bars on the Ibar and Trepca rivers) were collected using a stainless steel trowel from a depth of 0-10 cm and comprised 10 spot samples from a 4 m² area (aggregate sample weight c. 250 g). House dust samples (deposited on window sills) from the Cesmin Lug and Osterode camps, and from uninhabited houses in Roma Mahalla, were obtained using a nylon brush.

All samples were air-dried, disaggregated in an agate pestle and mortar and sieved through a <63 μ m stainless steel mesh to isolate the chemically active silt and clay fraction. Samples were then digested for 1 hour in 70 percent HNO₃ at 100°C and analysed for their Pb and Zn content (mg kg⁻¹) using an Atomic Absorption Spectrophotometer (AAS), and for their Cd and Cu content (mg kg⁻¹) by Magnetic Sector–Inductively Coupled Plasma–Mass Spectrometer MS-ICP-MS. Analytical precision of metal analyses ranged from 1 to 7%, with analytical accuracy versus two reference materials (ABS1 [a mid-Wales soil]) and CANMET2 [a Canadian soil]) ranging from 3 to 10 % and 2.5 to 13 %, respectively.

2.3 River water and drinking water sampling and analysis

A small number of water samples were collected from the River Ibar (n=1), along with samples taken from domestic supplies in the Cesmin Lug and Osterode camps, and Roma Mahalla (n=7). All water samples were filtered through 0.45 μ m cellulose nitrate membranes and stored in acid-washed 30 ml Nalgene bottles. All samples were acidified in the field with three drops of 50 percent HNO₃. In addition measurements of aqueous pH and EC (electrical conductivity) were made using a Hanna Instruments HI98129 meter. All water samples were analysed using an MS-ICP–MS at the Aberystwyth University and metal levels are presented in μ g l⁻¹. Analytical precision of metal analyses was between 0.8 and 6.3 %.

2.4 Human hair sampling and analysis

Finally, a number of hair samples were collected (using stainless steel scissors) from children in the Cesmin Lug and Osterode camps to facilitate the identification of potential Pb sources responsible for causing health problems in the Roma community. The analytical procedures followed, and the results from this survey, will be presented in an addendum to this report that will be published in March 2010 (fluvio report 2010/03/67).

2.5 Identification of Pb sources using isotope analysis

In order to identify the possible sources of Pb in Roma Mahalla, Pb isotopes (²⁰⁴Pb, ²⁰⁶Pb, ²⁰⁷Pb and ²⁰⁸Pb) were determined in a selection of soil, house-dust and mining/industrial waste samples using a Thermo-Finnigan Element2 MS-ICP-MS.

Samples were analysed in triplicate in batches of 5 along with blank samples and the NIST981 reference material. Analytical precision was found to be 0.13 % ($^{206/207}$ Pb) and 0.13 % ($^{208/206}$ Pb). Analytical accuracy versus the NIST981 standard was 0.3 % ($^{206/207}$ Pb) and 0.24 % ($^{208/206}$ Pb). Values for 204 Pb were corrected for the interference of 201 Hg, and isotopic ratios adjusted against the NIST981 data.

3. Environmental quality guidelines

Environmental quality guideline values for a range of metals and other potentially toxic substances have been established through legislation in many countries, in order to protect both human and ecosystem health (RCEP, 2004). Metal concentrations in river water and domestic water supplies have been compared to those set by EU directive 75/440/EEC, the World Health Organization and the US EPA (Table 2). These water quality threshold values relate to the quality required of water that is to be used for drinking water abstraction.

Guidelines	Pb	Zn	Cu	Cd
80/778/EEC ¹				
Target	-	100	100	1
Imperative	50	5000	3000	5
WHO ²				
MAC ³	10	-	2000	3
US EPA⁴				
MAC ³	15	-	1300	5

Table 2. Water quality standards for selected metals (values in $\mu g l^{-1}$).

¹Quality of water intended for human consumption

²World Health Organization

³Maximum Admissible Concentration

⁴United States Environmental Protection Agency drinking water guidelines

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Soil quality guidelines, employed particularly in Europe, have been designed to minimise human or animal health risks associated with exposure to potentially toxic substances (see Tables A1.1 and A1.2 in Appendix 1). These guidelines take into account both exposure and toxicological hazards (Ferguson and Denner, 1994; Chapman et al., 1999) and are set for specific land uses, each with their own different exposure risk. This approach results in a large range of contaminant concentration thresholds; for example, Pb guidelines for soils range from 70 mg kg⁻¹ for sensitive or high-risk land uses up to 2000 mg kg⁻¹ for lower risk land uses.

An alternative approach to assessing the hazards posed by metal contamination in soils and sediments is to calculate the naturally occurring background concentration of potentially toxic contaminants by using uncontaminated soils/sediments as a reference (e.g. Macklin and Klimek, 1992; Swennen et al., 1998; Hudson-Edwards et al., 1999a; Martin, 2004). This technique has the advantage that resulting background thresholds are specific to the area of interest, and also takes into account the likelihood of natural elevations in metal concentrations as a result of the erosion of surface exposures of metal-bearing veins.

In the light of the very wide range of metal threshold levels available, Pb concentrations in soils are evaluated using a twofold approach: first, by comparison to the latest guideline values developed by the Dutch Ministry for Housing and Spatial Planning (Department of Soil Protection, 2000). The Dutch guidelines represent a long-established (first used in 1962) and stringent set of quality criteria that are based upon extensive studies of both human and eco-toxicological effects of contaminants. Lead concentrations are also compared to the UK CLEA SGV values for residential land with plant uptake and commercial/ industrial land (Table 3).

Secondly, a statistical approach based on that of Davies (1983) was used to quantify a 'background' Pb concentration in soils collected in the Mitrovica region (see Appendix 1 for a summary of the statistical methodology used). Using this approach the threshold Pb concentration for uncontaminated soil (i.e. background) was calculated to be 80 mg kg⁻¹ in the Mitrovica region. This value is very close to the Dutch target value (Table 3) and is in good agreement with Pb concentrations

measured at depth in soil profiles in the Mitrovica region (Riccobono et al., 2004; Section 4.1 this report) and suggests that the calculated threshold of 80 mg kg⁻¹ is indicative of soils in the Pb-mineralised region, but unaffected by anthropogenically-sourced Pb deposition (e.g. from the Zveçan smelter). A second, and higher, threshold was identified at 1140 mg kg⁻¹ Pb, which defines a lower limit for severely contaminated samples. The two thresholds can be used together to define limits for background Pb concentrations (< 80 mg kg⁻¹), and those representing moderate (80-1140 mg kg⁻¹) and severe (>1140 mg kg⁻¹) Pb contamination in the Mitrovica region.

Table 3: UK CLEA SGV and Dutch Soil remediation standards for various metals (concentrations in mg kg⁻¹ dry weight).

Guidelines	Pb	Zn	Cu	Cd
UK CLEA SGV ¹				
Residential with plant uptake	450	-	-	1-8
Residential without plant uptake	450	-	-	30
Allotments	450	-	-	1-8
Commercial/Industrial	750	-	-	1400
Dutch Soil Remediation ²				
Target	85	140	36	0.8
Intervention	530	720	190	12
Background ³				
Background threshold	80	-	-	-

 Department for Food, Environment and Rural Affairs (2002) Contaminated Land Exposure Assessment soil guideline values (collated from SGV series 1, 3, 4, 5, 7 and 10). Maximum Cd concentrations are 1 mg kg⁻¹ at pH 6, 2 mg kg⁻¹ at pH 7 and 8 mg kg⁻¹ at pH 8.

- 2. Department of Soil Protection (2000) Circular on target values and intervention values soil quality standards
- 3. Background, as calculated in this study (see Appendix 2).

4. Results (Objective 1)

The presentation of results is divided into seven sub-sections, each dealing with a separate and distinct suite of data. The principal focus of this section concerns the concentration and spatial distribution of Pb in the soils within the proposed Roma Mahalla development site; Pb concentrations are assessed against Dutch, UK CLEA SGV and calculated 'background' levels. The results section concludes with an assessment of the possible sources of Pb contamination in the soils of Roma Mahalla by employing a Pb isotope analysis procedure.

4.1 Soil quality in Roma Mahalla

Lead concentrations measured in soils at 0-10 cm (surface), 10-30 cm (shallow subsurface) and 30-50 cm (deep sub-surface) are plotted as colour-coded proportional circles relative to Dutch Target and Intervention values (Figure 4): summary statistics for the Pb data are shown in Tables 4 and 5. Pb levels in both surface and subsurface soils show widespread enrichment above Dutch target values with 49 % and 52% of samples exceeding the Intervention values for surface (0-10 cm) and shallow sub-surface soils (10-30 cm), respectively (Table 5). Furthermore, between 93% (30-50 cm) and 98% (0-10 cm) of samples exceed the less stringent Target value (85 mg kg⁻¹). The highest mean Pb levels are found in shallow sub-surface soils (10-30 cm; mean = 568 mg kg⁻¹), concentrations are slightly lower in the surfaces soils (0-10 cm; mean = 513 mg kg⁻¹) and significantly lower in the deep subsurface soils (30-50 cm; mean = 252 mg kg⁻¹) (Table 5). The relatively low Pb levels between 30-50 cm are further highlighted by the fact that only 2 % of samples exceed the 530 mg kg⁻¹ Dutch Intervention value (Table 5), compared to 49 and 52% of samples at 0-10 cm and 10-30 cm depths, respectively. Using non-parametric significant difference analysis (Mann-Whitney U Test), a statistically significant difference (at $\alpha = 0.01$) is identifiable between Pb levels at 30-50 cm and those at 0-10 cm (p = 0.000) and at 10-30 cm (p = 0.000). There is, however, no significant difference (p= 0.293) between Pb levels between 0-10 cm and between 10-30 cm.



B)



A)



Figure 4: Colour coded proportional circles showing where Pb concentrations fall either below (green circles) or above (orange circles) Dutch target values, or where they exceed (red circles) Dutch intervention values for soil remediation: A) surface soils (0-10 cm), B) shallow sub-surface soils (10-30 cm), and C) deep sub-surface soils (30-50 cm).

It is likely that the relatively high Pb concentrations in surface and shallow subsurface samples at Roma Mahalla reflect atmospheric deposition of Pb-bearing particulates and aerosols. The extensive survey of surface and sub-surface soils by Riccobono et al. (2004) and Borgna et al. (2009) in northern Kosovo demonstrated widespread contamination of soils by a range of contaminant metals, and related this to airborne pollution sourced from the Zveçan smelter in Mitrovica (see Appendix 3 for a map showing the distribution of Pb in soils). To assess the depth of Pb contamination in the soils at Roma Mahalla, Figure 5 plots XRF determined Pb concentrations obtained from the three vertical soil profiles (see Figure 2D for

locations). In addition, Figure 6 plots Pb depth profiles obtained from two sites to the SE of Mitrovica investigated by Riccobono et al. (2004). All these profiles confirm the significant surface enrichment of Pb and also show that below c. 30-40 cm Pb levels reduce to values below the CLEA SVG and Dutch Interventions values, and often to values near to, or below, the calculated natural background concentration of Pb in the Mitrovica area. Taken together, these data suggest that soils below 30-40 cm were either formed prior to the onset of large-scale industrial activity in the Mitrovica area (i.e. they have been isolated from airborne contamination), and/or that pedogenic processes / anthropogenic activity have not resulted in significant finding because it demonstrates that although the surface and shallow sub-surface soils do contain elevated levels of Pb in particular, contamination is generally restricted to the upper 30- 40 cm of soil profiles.

There is also some variation in soil Pb concentrations with depth, from east to west across the Roma Mahalla site. For example, shallow (10-30 cm) and deep (30-50 cm) sub-surface soils taken from the SW end of the Roma Mahalla site (samples 35-62; Figure 2) generally have lower Pb levels (means of 402 and 190 mg kg⁻¹, respectively) than those from the NE (samples 1-34; Figure 2), which have mean Pb levels of 630 and 290 mg kg⁻¹, respectively. This variation of pattern in Pb levels is believed to be the result of ploughing practices: the NE of the site has recently been ploughed and this will have mixed the contaminated surface soils with the slightly less contaminated shallow sub-surface soils. To the SW of the site there is no evidence of recent ploughing and, therefore, there is a greater difference between the Pb levels in the undisturbed surface and shallow sub-surface soils (Figures 4A and 4B).

Element	Depth (cm)	Minimum	Maximum	Mean
	0-10	26	1000	513
Pb	10-30	28	2500	568
	30-50	40	650	252
	0-10	57	730	214
Zn	10-30	47	1540	220
30-50 57	57	260	111	
	0-10	3	125	23
Cu	10-30	2	250	26
	30-50	5	35	14
	0-10	nd	2.7	1
Cd	10-30	nd	80	3
	30-50	nd	1	1

Table 4. Summary statistics for metal concentrations in soils collected in Roma Mahalla (all values in mg kg⁻¹). nd = non-detectable.

Table 5. Percentage of Roma Mahalla soil samples exceeding Dutch, UK CLEA and statistically derived 'background' values for Pb. See Table 2 for details of threshold values.

Environmental quality value		0-10 cm	10-30 cm	30-50 cm
Dutch	% < T ¹	2	2	7
	% > T ¹	49	46	91
	% > I ²	49	52	2
UK CLEA	% < SGV ³	31	30	89
	% > SGV ³	69	70	11
	% < SGV ⁴	92	86	100
	% > SGV ⁴	8	14	0
Background	% < 80 mg kg ⁻¹	2	2	7
-	% 80 – 1140 mg kg ⁻¹	98	96	93
	% > 1140 mg kg	0	2	0

 1 T = Dutch target 2 I = Dutch Intervention

³ Soil Guideline values for residential land-use. No values defined for Cu and Zn.

⁴ Soil Guideline values for commercial/industrial use

Figure 5: Variations with depth in Pb concentrations (XRF-determined) from three vertical soil profiles in Roma Mahalla (see Figure 2D for profile locations).



Figure 6: Variations with depth in Pb concentrations from two vertical soil profiles to the SW of Mitrovica: A). Core MC193 (UTM: 492555 E 4745215 N), and B) Core MC447 (UTM: 490815 E 4746794 N). All data from Riccobono et al., (2004).

In the UK, the Department for Environment Food and Rural Affairs (DEFRA) Soil Guideline Values (SGVs), calculated using the Contaminated Land Exposure Assessment (CLEA) model (DEFRA, 2002), can be used to assess the risks to human health from exposure to contaminated soil. The figures represent 'intervention' values (Table 3), whereby concentration exceedence may require further investigation and/or remediation due to a potential unacceptable risk to site-users. The SGVs have been calculated for exposure pathways based upon residential, allotment and commercial/industrial land-uses, and have not been calculated for land used for agricultural or as public open space (Hudson-Edwards et al., 2008). In the case of soil Pb, the intervention value is 450 mg kg⁻¹ for residential and allotment use and 750 mg kg⁻¹ for commercial/industrial use.

Comparison of soil Pb concentrations from Roma Mahalla indicates that 69-70% of soil samples from up to 30 cm depth exceed the SGV guideline for residential and allotment land-use; suggesting that the land would require remediation prior to residential use (Table 5). Exceedence of the same SGV intervention value for soils at 30-50 cm depth is markedly lower at 11%. This suggests that the removal of the upper 30 cm of soil cover from Roma Mahalla would create compliance with SGV values; this possible site management / soil remediation approach is discussed in more detail in Section 5 of this report.

Compared to Pb, the concentrations of other metals in the Roma Mahalla soils are generally lower (Table 6). The only elements of concern are: (i) Cd, where 71% and 69% of samples exceed the UK CLEA SGV value for residential land use at 0-10 cm and 10-30 cm, respectively, (ii) Zn concentrations exceed Dutch target values in 93% and 81% of samples taken from 0-10 cm and 10-30 cm, respectively (Table 6), however Zn concentrations only exceed intervention values in 2% of samples at these depths. Mean Zn concentrations at 30-50 cm (111 mg kg⁻¹) are approximately half those in surface and shallow sub-surface soils (Table 4), with 80% of Zn concentrations at 30-50 cm depth falling below Dutch target values (Table 6).

Environmen	tal quality value		0-10	10-30	30-50
			cm	cm	cm
Zn	Dutch	% < T ¹ % > T ¹ % > I ²	5 93 2	17 81 2	80 20 0
	UK CLEA	% < SGV ³ % > SGV ³	n/a n/a	n/a n/a	n/a n/a
Cu	Dutch	% < T ¹ % > T ¹ % > I ²	93 7 0	93 5 2	100 0 0
	UK CLEA	% < SGV ³ % > SGV ³	n/a n/a	n/a n/a	n/a n/a
Cd	Dutch	% < T ¹ % > T ¹ % > I ²	21 79 0	22 76 2	80 20 0
	UK CLEA	% < SGV ³ % > SGV ³	29 71	31 69	92 8

Table 6: Percentage of Roma Mahalla soil samples exceeding Dutch and UK CLEA threshold values for Zn, Cu and Cd.

 1 T = Dutch target

 2 I = Dutch Intervention

³ Soil Guideline values for residential land-use. No values defined for Cu and Zn.

4.2 Soil Pb concentrations in the Cesmin Lug and Osterode camps

A small number of soils samples were collected from the Cesmin Lug (n=3) and Osterode (n=6) camps in north Mitrovica (Table 7). Maximum and mean concentrations in soils at both camps exceed Dutch Intervention and CLEA SGV values in surface and sub-surface samples indicating significant contamination with Pb. The mean surface soil Pb concentrations at Cesmin Lug and Osterode are on average 4.6 times and 4.7 times higher than at Roma Mahalla, respectively. Furthermore, although minimum surface soil Pb concentrations are lower at

Osterode (510 mg kg⁻¹) than at Cesmin Lug (1280 mg kg⁻¹), mean and especially maximum Pb concentrations are higher at Osterode (2412 mg kg⁻¹ & 5100 mg kg⁻¹) than at Cesmin Lug (2349 mg kg⁻¹ & 3550 mg kg⁻¹) (Table 7). This finding is significant because many NGOs have believed that the Osterode site was less contaminated than Cesmin Lug. We would strongly recommend that if Osterode remains as an inhabited camp for the foreseeable future, then a more detailed soil survey should be conducted to identify the exact severity and distribution of Pb contamination in the surface and sub-surface soils. These data could then be used to inform a soil remediation strategy for the camp.

Soil depth	Cesmin Lug	Osterode
0-10 cm	Minimum: 1280	Minimum: 510
	Maximum: 3550	Maximum: 5100
	Mean: 2349	Mean: 2412
10-30 cm	-	Minimum: 260
		Maximum: 2800
30-50 cm	-	Minimum: 125
		Maximum: 4500

Table 7. Soil Pb concentrations (mg kg⁻¹) in the Cesmin Lug and Osterode camps.

4.3 Drinking water

A small number of samples from public water supplies were collected from the Mitrovica area (Figure 3; Table 1). Measured concentrations in a majority of samples were below the limits of detection, with the exception Cu and Zn in a limited number samples (Table 8). However, the detectable Cu and Zn concentrations were below EU Target values for water intended for human consumption and WHO drinking water guidelines (Table 2). In the light of the results from this small survey, we do not believe that dissolved metals pose a significant threat to potable water quality in Roma Mahalla, or the Osterode and Cesmin Lug camps.

0						
	рН	EC	Pb	Zn	Cu	Cd
Osterode 1	7.2	290	<0.1	26	6.3	<0.1
Osterode 2	7.3	300	0.2	2.3	<1.4	<0.1
Cesmin Lug 1	7.1	340	<0.1	67	3.9	<0.1
Cesmin Lug 2	7.1	290	<0.1	1	<1.4	<0.1
Cesmin Lug 3	7.3	300	<0.1	1.6	<1.4	<0.1
Roma Mahalla	7.5	330	0.9	38	1.9	<0.1
Mitrovica	7.5	330	0.2	16	<1.4	<0.1

Table 8. Summary water geochemistry data from Roma Mahalla, Osterode, Cesmin Lug and Mitrovica.

4.4 Industrial waste

Extensive metallurgical industrial activity in the Mitrovica region has resulted in the creation of several large industrial waste dumps adjacent to the Rivers Ibar and Sitnica (e.g. Gornje Polje waste dumps, Zharkov Potok tailings pond). Metal concentrations are often very high within this material (Table 9); for example, the Zveçan waste samples contained (by weight) up to 5% Pb and up to 8% Zn. Given the relatively unconsolidated and fine-grained nature of the material, and its proximity to river channels, these waste dumps are potentially significant sources of metals that can be dispersal by rivers and especially wind. The significance of these deposits as potential sources of Pb to Osterode, Cesmin Lug and Roma Mahalla is considered in Section 4.7 of this report.

4.5 Pb levels in house-dust

A number of published research studies have shown that house dust can contain appreciable levels of contaminant metals (e.g. Diemel et al., 1981; Jabeen et al., 2001; Spalinger et al., 2007). Mining and smelting activity have often been identified as key industrial sources of contaminated house dust (Hwang et al., 1997; Bosso and Enzweiler, 2008), but wind-blown transfer of metals from contaminated soils may also be a significant source (Layton and Beamer, 2009). Given the believed significance of airborne Pb dispersal from the Zveçan smelter, the Gornje Polje

waste dump, the Zharkov Potok Tailings pond and the disused chemical factory, house dust samples were collected from the Osterode (n = 2) and Cesmin Lug (n = 2) camps and from Roma Mahalla (n = 3).

	Pb	Zn	Cu	Cd
Trepca Mine tailings 1	1300	260	350	1
Trepca Mine tailings 2	1400	230	400	0.4
Trepca Mine tailings 3	1300	1400	180	4.9
Gornje Polje waste 1	41300	69000	1600	1.3
Gornje Polje waste 2	18500	20200	1200	0.4
Gornje Polje waste 3	4100	3300	340	0.7
Gornje Polje waste 4	49800	82500	1500	2.1
Gornje Polje waste 5	3400	1700	160	0.6
Zn electrolysis waste 1	26800	88200	1500	940
Zn electrolysis waste 2	4100	135000	11700	580
Zn electrolysis waste 3	150	115000	12000	60
Disused chemical factory waste	670	1685	250	9

Table 9. Metal concentrations (mg kg⁻¹) in mine tailings and smelter/ industrial waste in the Mitrovica region.

The data reveal a significant difference between the Pb concentrations in the house dust at Roma Mahalla and the Cesmin Lug and Osterode camps (Table 10). At Roma Mahalla, the house dust samples have Pb concentrations that broadly match the surface soil Pb concentrations (Table 2), suggesting a probable single source of Pb for both soils and house dust. However, at Cesmin Lug and Osterode Pb concentrations are an order of magnitude higher than at Roma Mahalla and they are equivalent to (Osterode), or higher than (Cesmin Lug), the maximum Pb concentrations found in the surface soils at these locations. To achieve Pb levels in household dust between 5500 and 5900 mg kg⁻¹, the source material supplying the dust must have equivalent or higher concentrations. Although Pb-based paint is one possible local source, Table 9 suggests that the Gornje Polje waste is the most likely source. Establishing the severity of contamination in household dust is very significant for the residents of the Osterode and Cesmin Lug camps, especially given the fact that contaminated airborne dust that can be readily inhaled, particularly during periods of dry weather.

	Pb	Zn	Cu	Cd
Osterode 1 (occupied)	5900	2200	280	8
Osterode 2 (occupied)	5500	2160	220	6
Cesmin Lug 1 (occupied)	5900	4630	315	5
Cesmin Lug 2 (occupied)	5800	3330	360	14
Roma Mahalla 1 (unoccupied)	610	460	60	6
Roma Mahalla 2 (unoccupied)	650	350	55	5
Roma Mahalla 3 (unoccupied)	60	60	13	1

Table10: Metal concentrations (mg kg⁻¹) in house dust samples collected from Osterode, Cesmin Lug and Roma Mahalla.

4.6 Previously published data for Roma Mahalla and Mitrovica

Lead levels in surface soils collected in the NE region of Roma Mahalla by DAEM (2008) are generally similar to those presented in this report (compare Figure 4A with Figure 7). Minimum (23 mg kg⁻¹), mean (477 mg kg⁻¹) and maximum (1692 mg kg⁻¹) Pb concentrations determined by DAEM (2008) broadly correspond with those determined by this study (Table 4), and there is no statistically significant difference between the two datasets (p = 0.0848). This suggests that the Pb levels in surface soils collected and analysed by Fluvio are representative of those in Roma Mahalla; and together the two datasets confirm the widespread occurrence of Pb enrichment in surface soils in the area.



Figure 7: Colour coded proportional circles showing where surface soil Pb concentrations fall either below (green circles) or above (orange circles) Dutch target values, or where they exceed (red circles) Dutch intervention values for soil remediation (data from DAEM, 2008).

A comparison of surface soil Pb concentrations in Roma Mahalla with those measured elsewhere in north Kosovo and the Mitrovica region (Riccobono et al., 2004; DAEM, 2008; Borgna et al., 2009), indicates that maximum Pb levels, especially in Roma Mahalla, are often much lower than those measured elsewhere in the region (see Appendix 3). For example, peak Pb levels in the Cesmin Lug and Osterode camps (Table 7) exceed those measured in Roma Mahalla, whilst Riccobono et al. (2004) have reported seven occurrences of Pb levels in surface soils in the Mitrovica region in excess of 10,000 mg kg⁻¹, with a maximum of over 37,000 mg kg⁻¹. Indeed, only 2 % of soil samples collected in Roma Mahalla exceed the statistically-derived 'background' threshold of 1140 mg kg⁻¹, seen as representing significant Pb enrichment (Table 5), with 93-98% of samples falling between 80 and

1140 mg kg⁻¹. Taken together, the soil data collected by Fluvio, and the data obtained from other reports, show that whilst the soils at Roma Mahalla do have elevated levels of Pb, and often exceed Dutch Intervention and UK CLEA SGV values, the concentrations are generally lower than elsewhere in Mitrovica, and are significantly lower than in the Cesmin Lug and Osterode camps.

4.7 Possible sources of Pb contamination in the soils of Roma Mahalla, Osterode and Cesmin Lug

Lead within the environment is present as four main isotopes: ²⁰⁴Pb, which is a stable, and the long-lived radiogenic isotopes ²⁰⁶Pb, ²⁰⁷Pb and ²⁰⁸Pb, that are the daughter products of the decay of ²³⁸U, ²³⁵U and ¹³²Th, respectively. Lead in the environment may be sourced from both natural weathering of bedrock and/or from anthropogenic sources related to the exploitation of Pb ores (e.g. mining, smelting, Pb additives in petrol), giving rise to a range of Pb isotopic signatures depending upon the source of Pb (Figure 8). The relative abundance of each of the four Pb isotopes within bedrock and Pb ores will vary according to the primordial concentrations of ²³⁸U, ²³⁵U and ¹³²Th, and the length of the decay processes (i.e. the age of the ore deposit). Isotopic signatures for are commonly expressed as the ratios ²⁰⁶Pb/²⁰⁷Pb and ²⁰⁸Pb/²⁰⁶Pb; graphing these Pb isotope ratios on bivariate plots can help identify sample populations each with a different Pb isotopic signature or 'fingerprint'. Each isotope population is then potentially indicative of the Pb originating from a distinct source (Figure 8).

Lead isotopic signatures for metallurgical waste associated with the Trepca industrial complex (Gornje Polje Pb smelter waste, Zharkov Potok Tailings and Zn electrolysis / industrial waste) show that two distinct populations can be identified based upon ²⁰⁶Pb/²⁰⁷Pb and ²⁰⁸Pb/²⁰⁶Pb ratios (Figure 9). Tailings material contained within the Zharkov Potok tailings pond has a similar Pb isotopic signature to industrial waste from the Zn electrolysis and former battery factories. In addition, this material is also similar in isotopic composition to material that forms the bulk of the Gornje Polje waste dumps adjacent to the Zvecan Pb smelter (Gornje Polje waste 2, Figure 9). This material is generally characterized by relatively low ²⁰⁶Pb/²⁰⁷Pb and ²⁰⁸Pb/²⁰⁶Pb isotope ratios and is isotopically distinct from other material deposited on the Gornje

Polje waste dump (Gornje Polje waste 1, Figure 9). Gornje Polje waste 1 is isotopically distinct from Gornje Polje waste 2; it is characterised by higher ²⁰⁶Pb/²⁰⁷Pb and ²⁰⁸Pb/²⁰⁶Pb isotope ratios and comprises black, partially-vitreous, coarse-grained material that has been deposited on top of the dump. It is probable that this material originates from a different stage of the smelting process, and is likely to be representative of particulate material emitted from the Zveçan smelter stack during its period of operation.



Figure 8: Bivariate plot of ²⁰⁸Pb/²⁰⁶Pb ratios and ²⁰⁶Pb/²⁰⁷Pb ratios measured in anthropogenic and natural Pb sources. Data for UK and French petrol from Monna et al. (1997), South Bulgaria tailings from Bird et al. (in press); Russian petrol from Mukai et al. (2001), Czech fly ash, Pb ore, slag and natural Pb from Ettler et al. (2004), American petrol from Erel et al. (1997) and French Pb ore from Baron et al. (2006).



Figure 9: Bivariate plot of ²⁰⁸Pb/²⁰⁶Pb ratios and ²⁰⁶Pb/²⁰⁷Pb ratios for various potential Pb sources (solid symbols) and Pb sinks (open symbols).

A significant feature of Figure 9 is the close association between the soil and house dust isotopic signatures from the Osterode and Cesmin Lug camps and the isotopic signatures from the black coarse-grained Gornje Polje waste 1 material. This close isotopic association strongly suggests that the dominant source of Pb within soils of the two camps is the atmospheric deposition of Pb-rich particulates emitted during the operation of the Zveçan smelter. This finding re-enforces the conclusion in Section 4.5 of this report that, based on metal concentration data alone, the Gornje

Polje waste is the most likely source of Pb in household dust in the Osterode and Cesmin Lug camps.

Ratios for ²⁰⁶Pb/²⁰⁷Pb and ²⁰⁸Pb/²⁰⁶Pb in surface soils and at depth (> 70 cm) in Roma Mahalla show a greater degree of variation. In Roma Mahalla, ²⁰⁶Pb/²⁰⁷Pb ratios are generally higher than in Osterode and Cesmin Lug, whilst ²⁰⁸Pb/²⁰⁶Pb ratios are generally higher than in waste material at Zharkov Potok and the former battery factory. The relationship between the isotope signatures from deep soils not exposed to recent atmospheric deposition (and indicative of 'background' Pb in a mineralised region) and the signatures in surface soils and metallurgical waste, suggests that Pb in Roma Mahalla surface soils is derived from both atmospheric deposition (anthropogenically-sourced Pb) and from natural bedrock weathering. Roma Mahalla's location further from the Zveçan smelter, compared to the Osterode and Cesmin Lug camps, probably accounts for its relatively lower soil Pb concentrations and its greater variability in Pb isotope ratios.

Although it is probable that wind-blown dispersal of metal-rich particulates is occurring from the Zharkov Potok tailings pond and from the industrial waste at the former battery factory, particularly during dry summer months, Pb isotopic evidence suggests that these two sites are not acting as the primary source of Pb for the soils and house dust in Roma Mahalla, Osterode and Cesmin Lug.

A final, but significant, point is the proximity to the Sitnica and Ibar Rivers of the Zharkov Potok tailings pond, the waste dumps at Gornje Polje, and the waste dumps surrounding the disused chemical/battery factory. Several major studies undertaken by Fluvio in Romania, Bulgaria, Hungary and Spain (Macklin et al., 2006) have investigated the serious impacts on river systems that can occur as the result of poorly regulated mining activity (ore processing and waste storage) and tailings dam failures. Whilst we do not believe that the Zharkov Potok tailings pond is a significant source of airborne Pb to the Osterode and Cesmin Lug camps, if the c. 50 m high dam failed, tailings could temporarily block the Sitnica River and possibly inundate the north eastern sector of the city (including the two camps). There would also be large-scale downstream dispersal of tailings over 10s of kilometres by the Ibar River into Serbia. During our site visit in December 2009 it was also evident that

surface runoff is causing significant erosion of the Gornje Polje waste dumps adjacent to the Zveçan smelter. We strongly recommend that detailed environmental and geotechnical surveys of the Zharkov Potok tailings pond and Gornje Polje waste dumps are undertaken so that appropriate management/remediation plans can be developed and implemented.

5. Potential for, and appropriateness of, remedial action to improve soil quality in Roma Mahalla (Objective 2)

The soil element concentration data presented in this report, from both Fluvio's sampling and from information gathered from other reports, has demonstrated that surface and shallow sub-surface soils have Pb levels significantly above the calculated natural background value and they often exceed both Dutch Intervention and UK CLEA SGV values (Table 5). However, deeper sub-surface soils (>40 cm) have much lower Pb levels, often at or below background reflecting their development prior to the onset of large-scale mining/industrial activity in the area. When set in the context of Pb concentrations in the Cesmin and Osterode camps, and elsewhere in Mitrovica, the Roma Mahalla Pb levels are relatively low. Furthermore, in a broader European context, Pb levels in Roma Mahalla are not especially high when compared to a large database of similar floodplain soils collected by Fluvio from mining affected river basins in the UK, Romania, Bulgaria, Hungary and Spain (Figure 10).

Based on the evidence presented in this report, the Pb present in the Mitrovice soils is atmospherically derived, and although mean Pb levels in surface soils at Roma Mahalla are just below the Dutch intervention value, they are (i) significantly lower than in the Osterode and Cesmin Lug camps, (ii) lower than many surface soils in northern Kosovo, and (iii) significantly lower than in similar floodplain soils in the UK where large-scale mining operations ceased over 100 years ago (Figure 10). So, although the soils in Roma Mahalla are by no means pristine, they are probably some of the better quality soils (in terms of Pb concentrations) in the Mitrovica area.



Figure 10: Range (minimum and maximum) and mean Pb levels in floodplain soils from various mining affected river systems in Europe. Equivalent data are also shown for Roma Mahalla, Osterode and Cesmin Lug. The green and red horizontal lines show the Dutch target and intervention values, respectively. Data from: ¹Hudson-Edwards et al. (2008), ²Brewer et al. (2005), ³Macklin et al. (2003), ⁴Bird (2004), ⁵Bird et al. (2003), ⁶Bird et al. (2010), ⁷Bird et al. (in press), ⁸Riccobono et al. (2004).

In the light of these findings, what is the potential for, and appropriateness of, remedial action to improve soil quality in Roma Mahalla?

5.1 Soil removal

One solution would be to remove the top 30-40 cm of soil from the proposed development site at Roma Mahalla. This might appear to be a very attractive form of remedial action because the underlying uncontaminated soil would be exposed and this would not present a hazard to the Roma community. Furthermore, the removed contaminated soil could be used to cover areas of the Zharkov Potok tailings dam/pond, the Gornje Polje waste dumps, and the waste dumps surrounding the disused chemical factory. Although the removed soil would be contaminated, it is not so contaminated that it cannot support the development of vegetation. Therefore, the soil/vegetation could provide a suitable barrier to the erosion and dispersal of Pb by wind from these highly contaminated areas.

However, a cautionary note should be made concerning this possible form of remedial action. If the top 30-40 cm of soil is removed, the ground level will also be lowered by the same amount. This will lead to an increase in flooding at the site from groundwater because the water table will be nearer to the ground surface. Furthermore, because the Roma Mahalla development site is located on the Ibar floodplain, which at this point is only 2-3 m above low-flow river level, during times of overbank flooding water will 'pond' in the lower lying area where the soil has been removed.

These flooding problems could be mitigated by replacing the removed contaminated soil with uncontaminated soil from outside of the Mitrovica area. However, this would add significant cost to the resettlement project. Furthermore, data from Riccobono et al. (2004) (see Appendix 3) indicates that soil quality in Roma Mahalla is no worse than that found in the Mitrovica hinterland to the south and west. It would, therefore, be a challenge to find soils nearby that were significantly less contaminated than those already in Roma Mahalla.

5.2 Soil covering

A second option would be to cover the existing soil at Roma Mahalla with a geotextile membrane, and then coat the membrane with c. 10 cm of topsoil and then seed or turf it. Although this technique could be used to isolate the Roma Mahalla residents from direct contact with contaminated soils within the redevelopment site, it might prove to be a relatively expensive procedure, and it would not protect the community from atmospheric Pb fallout or from soil contaminated with Pb in areas not covered by the membrane.

5.3 Soil ploughing

A third soil remediation strategy would be to deep plough the site to a depth of a least 50 cm, thereby mixing contaminated surface (0-10 cm) and shallow subsurface (10-30 cm) soils with relatively uncontaminated deep sub-surface soils (30-50 cm). Deep ploughing would be to reduce contamination levels in surface soils from over 500 mg kg⁻¹ to a value perhaps closer to 450 mg kg⁻¹ (the UK SGV threshold for residential use with plant uptake) or 400 mg kg⁻¹ (EPA soil criterion for bare-earth play areas). Although deep ploughing would only result in a modest reduction in the mean soil Pb concentration, it will probably bring the Pb level below one or both of the environmental quality values. This may be viewed as significant achievement by the site developers, and ultimately lead to the Roma Mahalla site being approved for development. However, it should be noted that deep ploughing will not achieve the removal of Pb from the soil, but rather will distribute it throughout a deeper section of the soil profile.

5.4 Summary and recommendations

In the light of all the field and published data collected in this study, we believe that the primary management issue at the proposed Roma Mahalla development site is not the level of Pb in the underlying soils (they are elevated, but not sufficiently so to affect human health), but the legacy of Pb contamination in the Mitrovica region as a whole, especially in areas close to the Gornje Polje smelter waste dumps. A significant proportion of the c. 4.6 ha Roma Mahalla development site will be covered by impermeable surfaces: approximately 0.76 ha (17%) by roads, 0.72 ha (16%) by houses, 2.6 ha (57%) by gardens, and the remainder will be a 'buffer zone'. Given

that the houses and roads will effectively isolate the Pb-contaminated soil from the Roma Mahalla residents, the outstanding issue is the proximity of the gardens to the houses.

5.4.1 Specific recommendations

- Our data clearly show that the soil and air quality (the latter based on house dust analysis) is very poor at both the Cesmin Lug and Osterode camps. We strongly recommend that in the interest of public health that both of these camps are closed/relocated as a matter of urgency.
- 2) The soil data collected by Fluvio, and the data obtained from other reports, show that whilst the soils at Roma Mahalla do have elevated levels of Pb, and often exceed Dutch Intervention and UK CLEA SGV values, the concentrations are generally lower than elsewhere in Mitrovica, and are significantly lower than in the Cesmin Lug and Osterode camps. On the basis of this finding, we believe that the Roma Mahalla site would be suitable for a housing development, with the caveat that the gardens should not be used for growing fruit / vegetables for human/animal consumption.
- 3) If the Roma Mahalla development proceeds, we recommend that all gardens and areas not covered by buildings, roads or pavements, should be landscaped with paving slabs. Alternatively, these areas could be covered with a geotextile membrane and then turfed with material sourced from an uncontaminated area.
- 4) To ensure that Roma Mahalla residents are not exposed to new sources of Pb contamination, no metal smelting activity should be permitted at Roma Mahalla or in its environs. In relation to this, given that we have established that the primary source of Pb found in the house dust and soils at the Cesmin Lug and Osterode camps is sourced from the Zvecan Pb smelter, we also strongly recommend that this smelter is not reopened.

5) Finally, we have also shown that the Gornje Polje smelter waste dumps, and to a lesser extent the Zharkov Potok tailings pond, are a major source of atmospheric contamination and responsible for the Pb found in the soils and house dust at Cesmin Lug and Osterode. Therefore, we recommend that these sites are covered with soil and seeded as a matter of urgency. These actions would cut-off the primary source of contaminated airborne particulates presently being deposited in the Cesmin Lug and Osterode camps, and would also improve the air quality across Mitrovica as a whole. Furthermore, we recommend that the waste dumps adjacent to the discussed chemical factory, battery and Zn electrolysis plant are also covered with soil and seeded, as these are also a source of contaminated dust particularly to the southern part of Mitrovica city.

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Appendix 1

Table A2.1: European soils and sediment quality criteria (concentrations in mg kg⁻¹ dry weight).

Guidelines	As	Cd	Cu	Pb	Hg	Zn
UK ICRCL 70/90 ¹						
Threshold	50	3	250	300		1000
Maximum for grazing livestock	500	30	500	1000		3000
Maximum for crop growth	1000	50	250			1000
UK MAFF ²						
pH > 5.0	50	3		300	1	
pH 5.0-5.5			80			200
pH 5.5-6.0			100			200
pH 6.0-7.0			135			200
pH > 7.0			200			300
UK CLEA SGV ³						
Residential with plant uptake	20	1-8		450	8	
Residential without plant uptake	20	30		450	15	
Allotments	20	1-8		450	8	
Commercial/Industrial	500	1400		750	480	
UK EA freshwater SQGs ⁴						
TEL	5.9	0.596	36.7	35	0.174	123
PEL	17	3.53	197	91.3	0.486	315
Dutch soil remediation ⁵						
Target	29	0.8	36	85	0.3	140
Intervention	55	12	190	530	10	720
French BRGM ⁶						
SSDV	19	10	95	200	3.5	4500
FIV sensitive use	37	20	190	400	7	9000
FIV non-sensitive use	120	60	950	2000	600	pvl
Cormon PMIL ⁷						
Soil-human pathway						
Plavarounds	25	10		200	10	
Residential	50	20		400	20	
Parks	125	50		1000	50	
Industrial/commercial	140	60		2000	80	
Soil-plant pathway						
Agriculture/vegetable garden	200	0.1		0.1	5	
Grassland	50	20	1300	1200	2	
Swedish FPA ⁸						
Levels in polluted soils	15	0.4	100	80	1	350
Deniah EDA 9						
	20	F	500	400	2	1000
	20	Э	500	400	ు	1000

Table A2.2: Australian and North American soils and sediment quality criteria (concentrations in mg kg⁻¹ dry weight).

Guidelines	As	Cd	Cu	Pb	Hg	Zn
Australian NEPC ¹⁰						
Health Investigation Levels						
A	100	20	1000	300	15	7000
В						
C						
D	400	80	4000	1200	60	28000
	200	40	2000	600	30	14000
F Ecological Investigation Levels	500	100	5000	1500	75	35000
	00	4	100	COO	4	000
Interim urban	20	1	100	600	1	200
Australian ISQGs ¹¹						
ISQG low	20	1.5	65	50	0.15	200
ISQG high	70	10	270	220	1	410
12						
Canadian EQGs '						
Freshwater sediments	5.0	0.6	25.7	25	0.17	100
	5.9 17	0.0	30.7 107	ათ 01 ვ	0.17	123
Soil	17	5.5	197	91.5	0.400	515
Agricultural	12	1.4	63	70	6.6	200
Residential/parkland	12	10	63	140	6.6	200
Commercial	12	22	91	260	24	360
Industrial	12	22	91	600	50	360
US EPA GLNPO ¹³						
PEL	17	3.53	197	91.3	0.486	315
SEL	33	10	110	250	2	820
TET	17	3	86	170	1	540
ERM	85	9	390	110	1.3	270
PEL-HA28	48	3.2	100	82	NG	540
Consensus-based PEC	33	4.98	149	128	1.06	459
US NOAA SQuiRTs ¹⁴						
TEL	5.9	0.596	35.7	35	0.174	123.1
PEL	17	3.53	197	91.3	0.486	315
UET	17	3	86	127	0.56	520
<u>AE</u>						
US NOAA NS&T ¹⁹						
ERL	8.2	1.2	34	46.7	0.15	150
EKM	70	9.6	270	218	0.71	410

Notes for Tables A2.1 and A2.2.

- 1. Interdepartmental Committee for the Redevelopment of Contaminated Land (1990) *Notes on the restoration and aftercare of metalliferous mining sites for pasture and grazing.* Guidance Note 70/90.
- 2. Ministry of Agriculture, Fisheries and Food (1998) Code of good agricultural practice for the protection of soil.
- 3. Department for Food, Environment and Rural Affairs (2002) Contaminated Land Exposure Assessment soil guideline values (collated from SGV series 1, 3, 4, 5, 7 and 10). Maximum Cd concentrations are 1 mg kg⁻¹ at pH 6, 2 mg kg⁻¹ at pH 7 and 8 mg kg⁻¹ at pH 8.
- Environment Agency Habitats Directive Technical Advisory Group on Water Quality Sediment Subgroup (2004) Assessment of sediment contaminants in estuaries. WQTAG078K. TEL: Threshold Effects Level; PEL: Predicted Effects Level.
- 5. Department of Soil Protection (2000) Circular on target values and intervention values soil quality standards.
- 6. Darmendrail, D. (2003) *The French approach to contaminated-land management* (Revision 1). SSDV: Source/Soil Definition Values (define if soil is a pollution source); FIV: Fixed Impact Values; Sensitive: residential with vegetable garden; Non-sensitive: industrial or commercial; pvl: planned value limit.
- German Federal Ministry for the Environment, Nature Conservation and Nuclear Safety (1999) *Federal Soil Protection and Contaminated Soils Ordinance.* Cd agriculture/vegetable garden: 0.04 mg kg⁻¹ when bread wheat or strongly accumulation vegetables are grown; Cu grassland: 200 mg kg⁻¹ when sheep are grazed.
- 8. Swedish Environmental Protection Agency (2002) Swedish Environmental Quality Guidelines.
- 9. Danish Environmental Protection Agency (2002) *Guidelines on remediation of contaminated sites.* Environmental Guidelines 7.
- 10. National Environment Protection (Assessment of Soil Contamination) Measure (1999). Schedule B (1) *Guideline on the Investigation Levels for Soil and Groundwater.*
 - A: Standard residential with garden/accessible soil;
 - D: Residential with minimal opportunities for soil access;
 - E: Parks, recreational open space and playing fields;
 - F: Commercial/Industrial.
- 11. ANZECC and ARMCANZ (2000) Australian and New Zealand Guidelines for Fresh and Marine Water Quality. ISQG: Interim Sediment Quality Guideline.
- 12. Summary of Existing Canadian Environmental Quality Guidelines (2003). ISQG: Interim sediment quality guideline; PEL: Probable effect level.
- 13. Ingersoll *et al.* (2000) *Prediction of sediment toxicity using consensus-based freshwater sediment quality guidelines.* USGS final report for the USEPA Great Lakes National Program Office.

PEL: Probable effect level; SEL: Severe effect level; TET: Toxic effect threshold; ERM: Effects range median; PEL-HA28: Probable effect level for *Hyalella azteca*, 28-day test; PEC: Probable effect concentration, above which harmful effects are likely to be observed.

- Buchman (1999) NOAA Screening Quick Reference Tables. NOAA HAZMAT Report 99-1. Freshwater sediment screening values. TEL: Threshold Effects Level; PEL: Probable Effects Level; UET: Upper Effects Threshold.
- 15. NOAA (1999) Sediment Quality Guidelines developed for the National Status and Trends Program. ERL: Effects Range Low; ERM: Effects Range Medium.

Appendix 2

Statistical estimation of background soil-Pb concentrations.

Following the work of Davies (1983), background or normal Pb concentrations can be estimated using regression analysis of % cumulative frequency curves of log₁₀ metal concentrations from a sample population comprising both contaminated and uncontaminated samples. The lower, linear portion of the curves is seen as representing a lognormal population derived from uncontaminated samples, and a regression equation can be derived in order to estimate background concentration based on a number of percentile values (16th, 50th, 84th). Complex, non-linear portions of the frequency curves can be further sub-divided into increasingly contaminated sample populations and threshold concentrations identified.

This study gathered all published soil-Pb data for the Mitrovica area to generate a sample group of 819 soil-Pb values (Table A2).

	Minimum	Maximum	Mean	Standard Deviation
Soil Pb	22	37,100	788	2,002

Table A2: Summary statistics of the soil Pb dataset (all values in mg kg⁻¹)

Soil Pb concentrations were converted to their log_{10} equivalents and the frequency distribution established for 50 class widths where optimum class width (log int.) is given by:

Log int. = <u>log(maximum concentration/minimum concentration)</u> Number of class intervals (50)

The percentage cumulative frequency distribution (*F*) was calculated and plotted with the X-axis representing accumulated frequency and the Y-axis the class widths (Figure A2). Sinclair's (1974) method was used to resolve the complex curve into its constituent populations. The resulting curves comprise a linear lower portion, representing the 'background' population (B), and a complex curve, derived from contaminated soils (A). For soil Pb in the Mitrovica region this lies at 98 % implying that 98 % of the total sample population is derived from contaminated soils (A) and 2 % from normal soils (B). For each point along the linear relationship (B) *F* was recalculated as F' (Figure A2), where:

$$F' = (100 - F) \times (100/B)$$

Where *F* is the % cumulative frequency distribution and B is the percentage of the total sample number derived from normal soils (i.e. 2 %). The mean and standard deviation of *F*' are readily derived, with the mean \log_{10} Pb value corresponding to 50% cumulative frequency and the anti-log is the geometric mean (*XM*) of the arithmetic data. For a normally distributed population, 68% lies within ±1 standard deviation so that the standard deviation derived from:

0.5 x (16th percentile – 84th percentile)

The antilog is the geometric deviation (*SM*) of the arithmetic data. The highest probably soil-Pb value for an uncontaminated soil is derived from the statistical inference that only 0.14% of the population is likely to lie beyond the range given by $XM \times SM^3$.



Figure A2 – Cumulative frequency curve used to determine the background populations for Pb: Curve 1 is the frequency plot of log10 Pb values, Curve 2 is the frequency curve for F' at the lower 'background' threshold, and Curve 3 is the frequency curve for F' at the upper 'severely contaminated' threshold.





Figure A3: Schematic geochemical map showing the distribution of Pb in soils in the Mitrovica area (from Riccobono et al., 2004).