Heavy Metals in Mosses and Soils on Six Transects Along the Red Dog Mine Haul Road Alaska

Western Arctic National Parklands
National Park Service

by
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Executive Summary

The Red Dog Mine Haul Road traverses 24 miles of National Park Service (NPS) lands in Cape Krusenstern National Monument (CAKR), Alaska. Ore trucks use the road to transport 1.1 million dry tons of lead-zinc concentrate annually from the mine to a port site on the Chukchi Sea. In the summer of 2000, moss and soil samples were collected from six transects perpendicular to the haul road in CAKR. Laboratory analyses were performed on the moss *Hylocomium splendens*, soil parent material, road dust, and substrate from materials sites. Analysis revealed a strong road-related gradient in heavy metal deposition. *H. splendens* was highly enriched in lead (Pb > 400 mg/kg), zinc (Zn > 1800 mg/kg), and cadmium (Cd > 12 mg/kg) near the haul road. Concentrations decreased rapidly with distance from the road, but remained elevated at transect endpoints 1000 m – 1600 m from the road (Pb >30 mg/kg, Zn >165 mg/kg, Cd >0.6 mg/kg). Samples collected on the downwind (north) side of the road had generally higher concentrations of heavy metals than those collected on the upwind (south) side.

Enrichment factor (EF) analysis of moss versus soil parent material demonstrates that remobilized soil (e.g., dust composed of roadbed material) account for only a fraction of the elevated heavy metal concentrations on the road corridor. Enrichment in Pb, Zn, and Cd from airborne sources other than remobilized soil (e.g., ore concentrate) is readily apparent. Analysis of dust shaken from vegetation adjacent to the haul road shows low to average levels of crustal elements (aluminum and iron) and extremely high levels of heavy metals. This is especially striking in comparison to materials site samples that differ in being very low in heavy metals. Considered together, these results suggest that ore concentrate escapement is occurring along the haul road corridor. The fact that EF levels remain elevated even at transect endpoints suggests the additional possibility of contributions of airborne heavy metals from mining activities to the Omikviorok River drainage as a whole. The source of these larger scale contributions is unknown and may include the haul road, port site, mine site, and/or a currently unidentified source.

Results from this study showed Pb levels in excess of 60 mg/kg dw in all transect points ≤ 100 m, with a longer shadow on the downwind (north) side of the road. In the Nordic moss monitoring program, *H. splendens* samples in excess of 60-80 mg/kg dw Pb are considered characteristic of highly polluted areas. Lowest heavy metal concentrations were seen in moss samples 1000 m – 1600 m from the road on the upwind (south) side. However, even these samples greatly exceeded maxima seen in previous *H. splendens* from arctic Alaska and contained 4-7 times as much Pb, Zn, and Cd as heavily dust-laden samples taken adjacent to the Dalton Highway (Prudhoe Bay Haul Road) in north-central Alaska. Highest levels near the Red Dog Haul Road equal or exceed (1.5 – 2.5 times) maxima reported for samples from severely polluted regions in Central European countries.

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Glossary

<	less than
>	greater than
ACRP	Arctic Contaminants Research Program
Ag	silver
Al	aluminum
B	boron
BMSL	Battelle Marine Sciences Laboratory
Ca	calcium
Cd	cadmium
Cr	chromium
Cu	copper
dw	dry weight
Fe	iron
GFAA	graphite furnace atomic absorption
ICP-AES	inductively coupled plasma (atomic emission) spectroscopy
ICP-MS	inductively coupled plasma (mass) spectroscopy
K	potassium
MDL	method detection limit
Mg	magnesium
mg/kg dw	milligram per kilogram (ppm) dry weight
Mn	manganese
MS	materials site
Na	sodium
Ni	nickel
P	phosphorus
Pb	lead
ppm	parts per million (µg/g or mg/kg)
QA	quality assurance
QC	quality control
RPD	relative percent difference
SRM	standard reference material
µg/g dw	microgram per gram (ppm) dry weight
UMN	University of Minnesota
USEPA	United States Environmental Protection Agency
WEAR	Western Arctic National Parks
Zn	zinc

1. Introduction

The Red Dog lead-zinc mine is operated by Cominco Alaska, Inc. in a remote region of northwestern arctic Alaska (Fig.1). It is the largest lead-zinc mine in the world, and the company has worked hard to make the project appealing to local Iñupiat communities by providing jobs and minimizing the environmental footprint of the operation. Along the way, Cominco has met many considerable challenges posed by carrying a mountain of ore 52 miles overland to the port site at the Chuckchi Sea. Ore trucks weighing 100 tons (net 72-ton payload) are dispatched approximately every 15 minutes around the clock; aside from brief hiatuses in the early evening and late at night, this schedule is maintained throughout the year (Warren Hood, Cominco, pers. comm., June 2000).

Much dust is entrained by the continuous heavy traffic, both from the roadbed and perhaps also from the ore truck surfaces. In 1999, staff of Western Arctic National Parks (WEAR) initiated preliminary studies in Cape Krusenstern National Monument (CAKR) along the 24-mile section of the haul road that crosses land administered by WEAR. The purpose was to determine whether lead (Pb) and zinc (Zn) were elevated in near-road grab samples of *Aulocomnium* moss. The approach took advantage of the fact that mosses generally lack vascular systems, so tissue concentrations are minimally confounded by uptake of mineral elements. For this reason, mosses (as well as lichens) have been widely used in studies of atmospheric deposition, particularly for heavy metals, trace elements, and radionuclides. Laboratory treatment may or may not include washing the field samples, depending on whether the project objective is to study tissue (foliar) concentrations, per se, or to study environmental levels that include particulates from atmospheric deposition. In the case of the WEAR study, the objective was the latter, and therefore samples were not washed. Likewise, all data presented here, from our own studies or from studies cited for comparative purposes, used unwashed samples.

Results from the preliminary studies revealed Pb concentrations three orders of magnitude higher than median levels found in regional arctic Alaska studies of a different moss, *Hylocomium splendens* (Ford et al. 1995). However, the comparison was not straightforward due to potential species differences. Further, different laboratories and analytical methods were used, and there was no direct methods overlap or intercalibration with previous arctic Alaska studies. To more carefully evaluate these initial findings, the decision was made to implement a pilot study that would define the area apparently affected by road dust and/or other mining related activities. Results from that pilot study are the subject of this report.

The target species selected for the current study was the monitoring moss *Hylocomium splendens*, largely because of the wealth of pre-existing information on this species. For example, *H. splendens* has been well characterized with

respect to element uptake (e.g., Rühling and Tyler 1970; Berg and Steinnes 1997), field variability (Ford et al.1995), and the relationship between tissue concentration and atmospheric deposition (Ross 1990). Further, an unusual and relevant characteristic of *H. splendens* is that annual increments can be easily distinguished, permitting the analysis of tissue from precisely defined exposure periods.

For all of these reasons, *H. splendens* is one of the mainstays of the long-term Nordic monitoring program to assess regional atmospheric deposition of trace elements and heavy metals in northern Europe and Fennoscandia (e.g., Rühling and Steinnes 1998). Finally, other data on heavy metal concentrations in *H. splendens* are available for arctic Alaska (Ford et al.1995; Ford et al.1997; Wiersma et al.1986), as well as for other parts of Alaska (e.g., Denali [Crock et al. 1992a], Wrangell-St. Elias [Crock et al. 1993], the Kenai Peninsula [Crock et al. 1992b]).

The current study also included the analysis of soils at depth at several positions along each transect. These are presumed to represent local soil parent material and allow the calculation of **enrichment factors** (Nash and Gries 1995; Puckett and Finegan 1980). Enrichment factors relate ratios of contaminant elements to Al, or other conservative soil element, in moss tissue to the same ratios in soils. Ratios <10 generally are taken to represent local lithology (deriving from local soils via road dust or other similar sources), whereas ratios >10 reflect additional atmospheric deposition factors related to long range transport. In this case, such additional factors might include, for example, deposition of lead (Pb)- and zinc (Zn)-enriched ore concentrate from the port (or mine) itself or from concentrate adherence to outer surfaces of ore trucks from unloading/loading operations and subsequent wind dispersal during road travel. In principle, additional factors could also include long-range atmospheric transport from regional or hemispheric sources, but these contributions appear to be small or negligible for arctic Alaska, especially for Pb (Ford et al. 1995).

Other target elements for this study included silver (Ag) and cadmium (Cd). Ag is often associated with Pb-Zn deposits, but is rarely analyzed in air pollution studies. Cd was included because it co-occurs with Zn, to which it is geochemically related; and it is of potential toxicological concern. It is also one of the few contaminant elements found to be elevated relative to local parent material over other parts of arctic Alaska (J. Ford, unpublished data). Mercury (Hg) is also elevated relative to local parent material in arctic Alaska (J. Ford, unpublished data), but was not included in this study due to financial constraints. Ancillary elements included for interpretive purposes included aluminum (Al), iron (Fe), magnesium (Mg), and calcium (Ca).

2. Purpose of this report

This report summarizes results for heavy metals and trace elements found in mosses and soils collected near the Red Dog Haul Road 24 June – 3 July 2000. The primary objective was to determine whether there were significant gradients of Pb, Zn, and Cd deposition with respect to the road. Additional objectives included analyzing the relationship of the data from this study to other data for arctic Alaska, the circumpolar Arctic, and industrialized Europe, and evaluating the quality of data provided from each of two independent analytical laboratories contracted by NPS for this work.

3. Study design

3.1 Fieldwork

Six transects were placed perpendicular to straight stretches of the Red Dog Haul Road where it crosses CAKR land (Fig. 2). Three transects were in upwind positions (south) and three in downwind positions (north) relative to the road.

A single sample of *H. splendens* was collected from a two-meter radius at transect points 3 m, 50 m, 100 m, 250 m, 1000 m, and (at two of the six transects) 1600 m from the road. If sufficient moss was not found within a two-meter radius, then collections were continued along the line perpendicular to the transect (and parallel to the road) within a 4m (± 2 m) sampling strip. Moss collection methods followed those outlined in Ford et al. (1995). All moss samples were cropped to include only the most recent (ca. 3 yrs) growth and air-dried on site inside a closed drying tent at Materials Site 6.

Soil samples of presumptive parent material were collected along each transect at 3 m, 50 m, 100 m, 1000 m, and (at two of the six transects) 1600m. Due to financial constraints, only the 3-m and 1000-m samples were sent for analysis; remaining samples have been archived at WEAR by L. Hasselbach. Soil samples were taken in the same locations as moss samples. A soil plug extending down to permafrost was removed. This newly exposed frozen soil surface was covered by a Ziploc® bag with a small hole in the center, penetrated by a coarse-bit hand-operated drill. The drill was then used to bring up material from depth, while the plastic barrier excluded ambient particulates from above the permafrost floor. Drilling continued until only inorganic (gray) material appeared to be brought to the surface (~35 – 45 cm). The material was collected and transferred to new I-Chem series 200 jars underneath the plastic barrier. Soil plugs were replaced after sampling had taken place.

Dust samples were collected from vegetation at each of the three downwind (north) transects at the 3 m plot by shaking dust off woody vegetation (primarily *Salix* and *Betula* spp.) into a Ziploc® bag. Twigs, leaves, and other visually obvious organic debris were removed from these collections using forceps. Samples of road surfacing materials were also collected from three of the haul road materials sites (MS3, MS5, and MS6). For these collections, a berm of presumptive road surfacing material was located and fine material was sifted from the gravel into an I-Chem jar.

3.2 Laboratory work

Two laboratories with different per-sample price structures were selected for this project. One laboratory (U. Minnesota Soils Laboratory) previously produced high quality results on several studies of elemental concentrations in lichens (Geiser et al. 1994). The second laboratory (Battelle Marine Science Laboratory) had previously produced high quality results on a large study of elemental concentrations in lichens, mosses, soils, sediments, and animal tissues from arctic Alaska (Ford et al. 1995). Battelle's data on samples of *H. splendens* and soils from arctic Alaska was considered the single most useful data set for comparison with the current study.

To ensure comparability with previous results for arctic Alaska, the full suite of samples was directed to Battelle (BMSL). At the same time, splits of selected moss and soil samples were also sent to U. Minnesota Soils Laboratory (UMN) to assess that laboratory's potential to produce data of comparable quality at a lower per-sample cost.

BMSL analyzed the complete range of moss and soil samples submitted and produced acceptable data for all elements in both matrices (appendix I). Their estimates of concentrations of Ag, Al, Ca, Cd, Fe, Mg, Pb, and Zn in moss and soils are used in the following discussions.

UMN provided unique data on dust shaken from roadside plants as well as berm materials from three materials sites. Their estimates of elemental concentrations on these substrates are used in the following discussions, although Al, Fe, and Mg are likely to be underestimates and Cd may be overestimates in these materials, for reasons given in appendix I.

BMSL methods included complete digestions using a mixture of nitric and hydrofluoric acid, ± boric and hydrochloric acids, with samples analyzed by ICP-AES, ICP-MS, or GFAA, depending on the analyte and matrix. UMN methods included dry ashing with 10% HCl (moss) or a nitric acid/microwave digestion that is essentially a leaching technique (soils), with all solutions analyzed by ICP-AES.

Detailed analysis of results from each laboratory, comparisons of results between laboratories, and recommendations for future laboratory work are in appendix I. Appendix II presents criteria for censoring and flagging laboratory data, and appendix III shows quality assurance screening tables. Raw laboratory data is in appendix IV.

4. Influence of the haul road on heavy metal concentrations *in H. splendens* moss

Analysis of changes in moss chemistry with distance from the haul road clearly demonstrates a strong road-related gradient of heavy metal deposition (Fig. 3). Heavy metal elements (Pb, Zn, Cd, Ag) are highly elevated near the road, leveling off between 1000 m and 1600 m. Crustal elements (Al, Fe, Mg) show a generally similar pattern. Calcium (Ca) shows the same pattern as crustal elements; the source of this element is likely to be the Ca compounds that are applied to the road surface for dust control (John Martinisko, Cominco, pers. comm. 3/21/01).

Samples collected on the downwind (north) side of the road have generally higher concentrations of crustal elements that fall off more slowly away from the road than those on the upwind (south) side of the road. This is likely due to prevailing seasonal winds; however, the pattern is more weakly expressed for heavy metal elements, suggesting that road related factors are not the only sources of these elements.

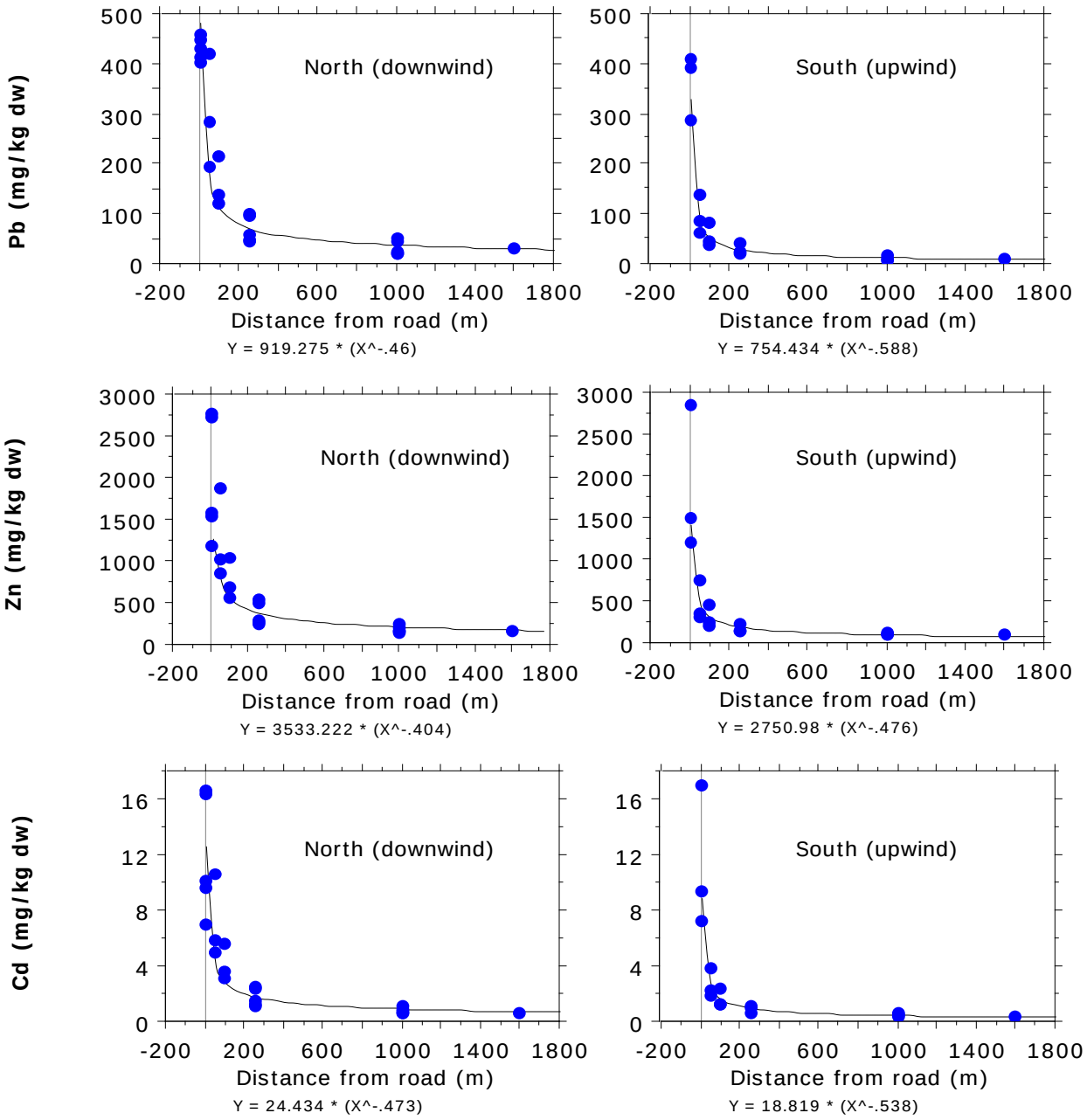

Figure 3. Changes in concentrations of eight elements with distance from the Red Dog Haul Road. For each element, the left panel gives data for downwind (north) transects and the right panel gives data for upwind (south) transects. All units are mg/kg dry weight.

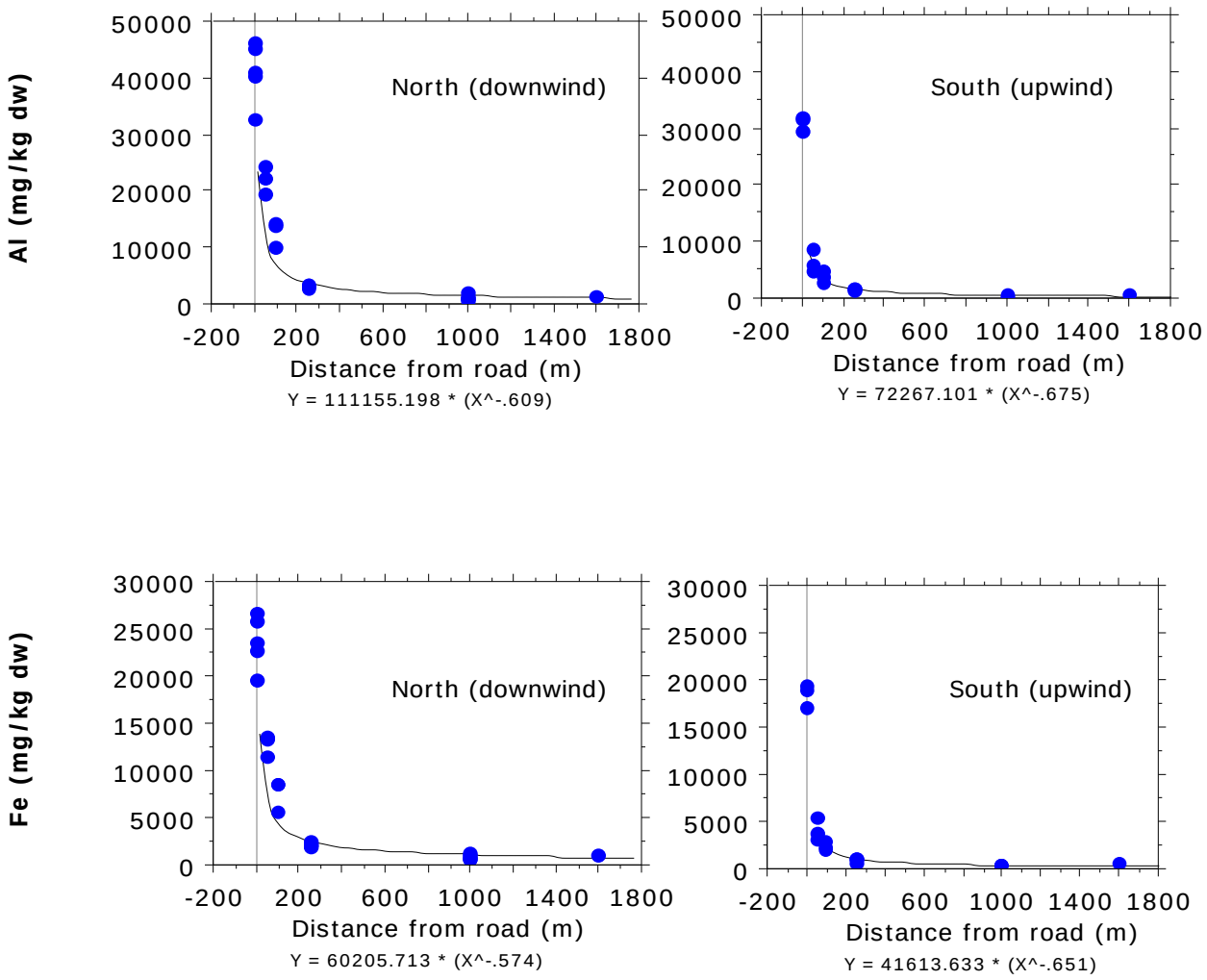

Figure 3 (continued). Changes in concentrations of eight elements with distance from the Red Dog Haul Road. For each element, the left panel gives data for downwind (north) transects and the right panel gives data for upwind (south) transects. All units are mg/kg dry weight.

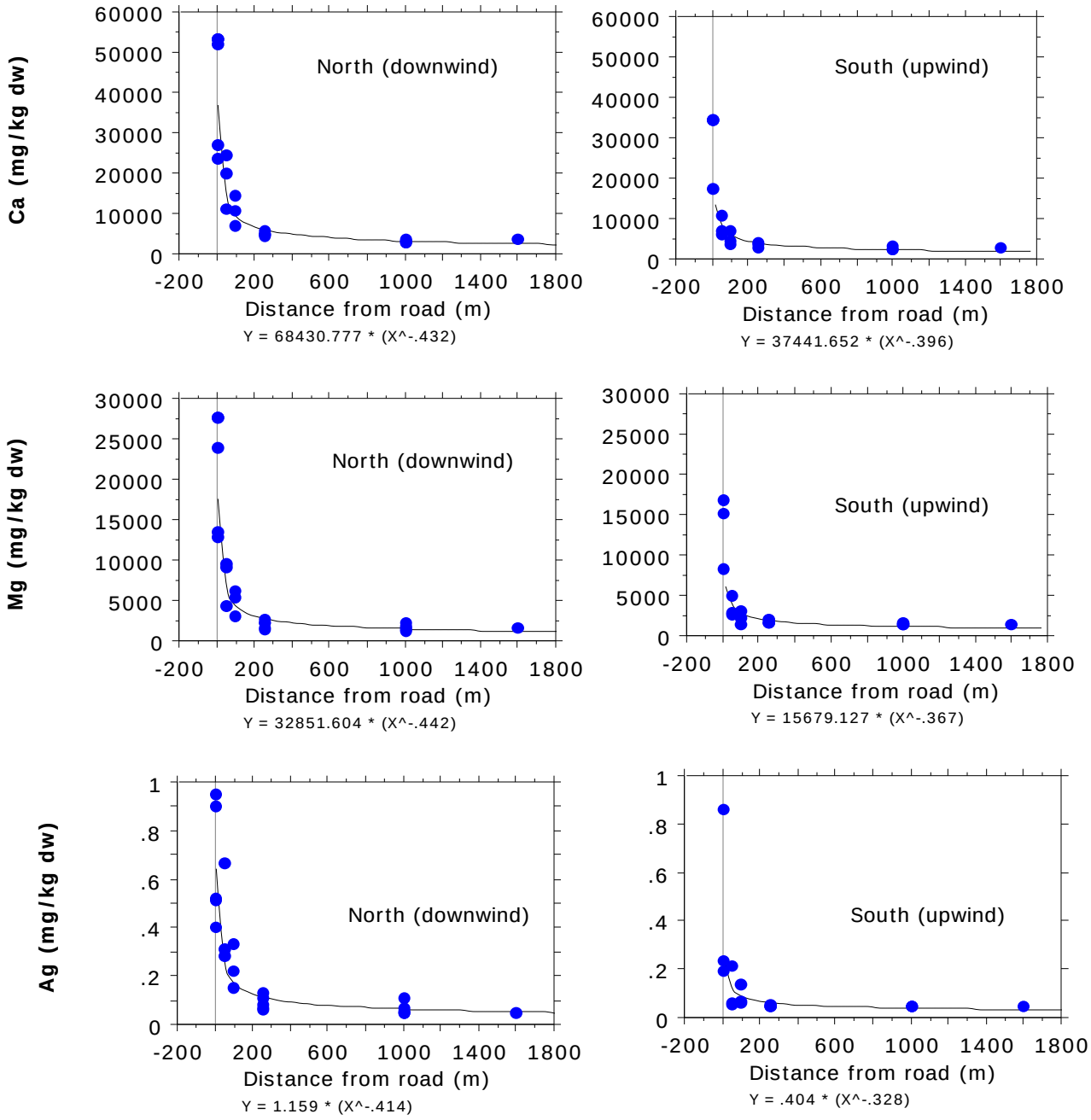

Figure 3 (continued). Changes in concentrations of eight elements with distance from the Red Dog Haul Road. For each element, the left panel gives data for downwind (north) transects and the right panel gives data for upwind (south) transects. All units are mg/kg dry weight.

Heavy metal concentrations in *H. splendens* moss along transects on each side of the road are summarized in Table 1.

Table 1. Means and standard errors for heavy metal concentrations in *Hylocomium splendens* moss at transect points on north and south sides of Red Dog Haul Road. *n* = number of samples. All units are mg/kg dw.

North Side of Haul Road

Transect Point (m)	*n*	Pb	Zn	Cd
3	5	430 (± 38)	1962 (± 328)	12.0 (± 1.9)
50	3	299 (± 66)	1252 (± 318)	7.2 (± 1.7)
100	3	159 (± 29)	763 (± 145)	4.1 (± 0.8)
250	5	71 (±12)	370 (± 60)	1.8 (± 0.3)
1000	6	33 (± 7)	187 (± 22)	0.8 (± 0.1)
1600*	1	30	169	0.6

South Side of Haul Road

Transect Point (m)	*n*	Pb	Zn	Cd
3	3	363 (± 38)	1853 (± 511)	11.2 (± 3.0)
50	3	97 (± 22)	475 (± 139)	2.6 (± 0.6)
100	3	55 (± 14)	305 (± 76)	1.6 (± 0.4)
250	3	29 (± 6)	169 (± 27)	0.9 (± 0.1)
1000	3	12 (± 3)	114 (± 2)	0.5 (± 0.1)
1600*	1	12	96	0.4

* 1600 m samples were collected on transects 1S and 1N only

Enrichment factor analysis was employed to determine the extent to which the elevated concentrations were due simply to remobilized parent material (road dust, cryogenically exposed parent material, and so on) as opposed to airborne deposition (e.g., ore concentrate escapement).

Enrichment factor analysis is a standard technique that was developed to disentangle the role of remobilized parent material from other potential sources of atmospheric inputs (e.g., Puckett and Finegan 1980; Nash and Gries 1995). This technique is typically applied in studies in which mosses and/or lichens are being used as passive air quality monitors, and there is concern that elevated elemental concentrations may in fact simply represent remobilized parent material that is naturally enriched in the contaminants under analysis. The enrichment factor (EF) compares the concentration of individual elements of interest to the concentration of a conservative soil element, usually Al, in vegetation versus local parent material (Puckett and Finegan 1980; Nash and Gries 1995). For example, an EF for Pb would be calculated as:

$$\frac{\frac{[Pb]\text{ in lichen or moss}}{[Al]\text{ in lichen or moss}}}{\frac{[Pb]\text{ in soil parent material}}{[Al]\text{ in soil parent material}}}$$

If the ratio of elements is the same in both vegetation and parent material, the overall ratio will be equal to 1.0. In practice, a more conservative ratio of 10.0 is generally used to reflect plant contaminant concentrations in excess of what would normally be supplied by the local geological substrate.

The present study analyzed deep soils at two locations (3 m and 1000 m) in each of the six transects. To confirm that deep soils reflected parent material, soils were also analyzed for total organic carbon (TOC). Eight of the 12 samples were highly inorganic, with TOCs less than 5%. However, four samples from two transects (2N and 3N) had TOC ranging from 10% to 29%, a clear indication of organic soils. Whether this is due to insufficient sample depth or failure of the plastic barrier to shield samples from ambient particulates from above the permafrost floor is unknown. Regardless, these four samples were inappropriate for use as denominators in the EF calculations because they do not represent parent material. The validity of using *mean* (high TOC samples excluded) rather than *transect-specific* parent material composition in the denominator was assessed for the remaining four transects by comparing EFs calculated using (1) site-specific parent material, (2) average transect parent material, and (3) overall average parent material. In these analyses, R^2 ranged from .31 to .99, with R^2 > 0.89 for Cd, Fe, and Pb. Examination of actual regressions indicated that in no case did overall interpretation change when this substitution was made, even when R^2 was low. Consequently, mean parent material ratios were used as the denominator for all plots in the analyses reported here.

Figure 4 corrects the raw elemental concentrations in *H. splendens* moss, reported in Figure 3, for the influence of local geological substrate, using the enrichment factor formula. The resulting graphs are slightly counterintuitive; Figure 3 clearly indicates high levels of heavy metals decreasing with distance from the road, while Figure 4 seems to indicate the opposite pattern, with low EFs near the road. This is because dust from remobilized parent material is settling out on the moss, thereby swamping inputs of other materials (e.g., ore concentrate if present) near the haul road and resulting in a cluster of low EFs. The signal becomes clearer with increasing distance from the road, even though raw metal concentrations (Fig. 3) are lower.

Using the general guideline of EF ≥ 10 as the cutoff for presence of non-parent material enrichment, several points immediately become clear:

1. Neither iron (Fe) nor silver (Ag) demonstrates clear enrichment from substances other than parent material (EFs < 10).

2. Magnesium (Mg) and to some extent calcium (Ca) concentrations close to the road are dominated by the signal from parent material (EFs close to 10).

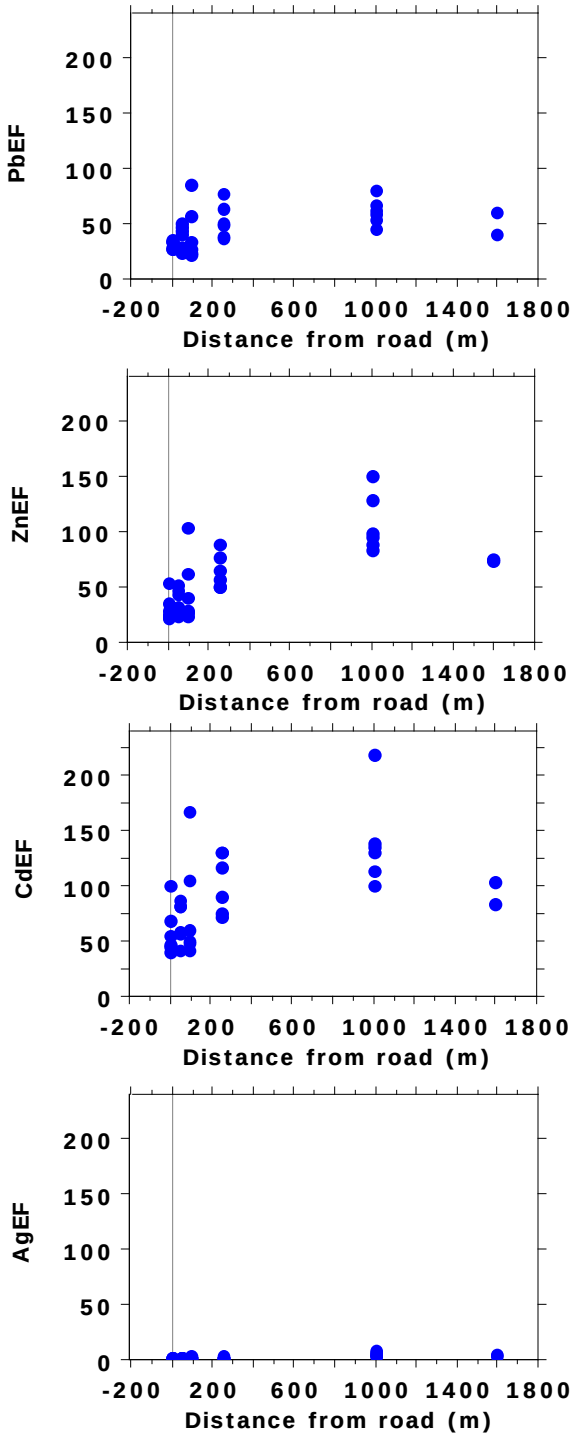

Figure 4. Changes in moss enrichment factors (EFs) plotted with distance from the Red Dog Haul Road (upwind and downwind transects combined). EFs are calculated against aluminum (Al) (see text for equation). Values close to unity (EF = 1) represent concentrations derived from soils; values > 1 represent concentrations in excess of soil parent material (in this case road dust). EFs are dimensionless because units in the numerator and denominator cancel each other out.

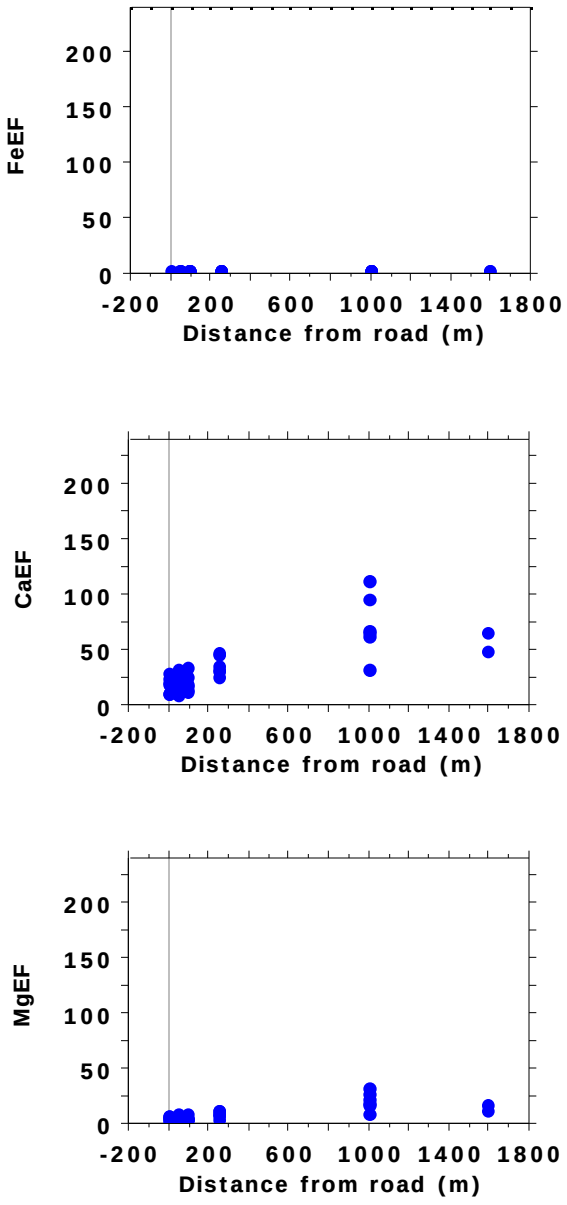

Figure 4 (continued). Changes in moss enrichment factors (EFs) plotted with distance from the Red Dog Haul Road (upwind and downwind transects combined). EFs are calculated against aluminum (Al) (see text for equation). Values close to unity (EF = 1) represent concentrations derived from soils; values > 1 represent concentrations in excess of soil parent material (in this case road dust). EFs are dimensionless because units in the numerator and denominator cancel each other out.

3. Pb, Zn, and Cd concentrations show enrichment by substances other than local parent material even close to the haul road (EFs > 10).

In fact, all Pb, Zn, and Cd EFs greatly exceed 10, ranging from 20 – 220 (Fig. 4), suggesting that the road corridor is being affected by both remobilized parent material (e.g., road dust) and airborne heavy metal deposition from other sources (e.g., windblown concentrate from truck surfaces). High EF levels combined with elevated heavy metal levels at transect endpoints (Table 1) suggest that the affected area extends beyond our study area and may include broader portions of the Omikviorok River drainage.

5. Relationship of Red Dog data to other data for arctic Alaska

5.1 Soils at depth

Data for Red Dog soils at depth are compared to data previously produced (Ford et al. 1997) on arctic Alaska soils at depth by BMSL for the USEPA Arctic Contaminants Research Program (ACRP). The ACRP soils data are drawn from soil cores at three sites: the Barrow Environmental Observatory (BEO), the calcareous Elusive Lake watershed (north of Toolik Lake about 15 km east of the Dalton Highway), and the Feniak Lake watershed (Noatak National Park and Preserve), which is known to contain pockets of serpentine soils.

Within the ACRP data set, the highest inorganic soil concentrations of Ca, Cd, Pb, and Zn are found in the two Elusive Lake cores. The comparison of Red Dog subsoils data to arctic Alaska ACRP subsoils data (Fig. 5) indicates that the Red Dog area is not highly calcareous. All in all, however, aside from being less calcareous (and correspondingly richer in Al), subsoils in this part of Cape Krusenstern National Monument are not strikingly different from those found in other areas of arctic Alaska. The 2-5 times enrichment in Pb in these samples is probably the tail end of the (presumably much richer) deposit being mined farther up the valley.

5.2 Road dust, materials sites samples, and ore concentrate

Despite the unremarkable chemical profile of soils at depth, samples of dust shaken from birch and willows adjacent to the Red Dog Haul Road are conspicuously elevated in Cd, Pb, Zn, and Mg (Fig. 6). Possible sources include road materials from Red Dog Materials Sites, and ore concentrate. The order of magnitude elevation in Pb and Zn in the road dust relative to other materials (e.g., soils at depth, and so on) is consistent with the results from enrichment factor analysis (section 4) in suggesting that much of the source material for dust probably derives from ore concentrate.

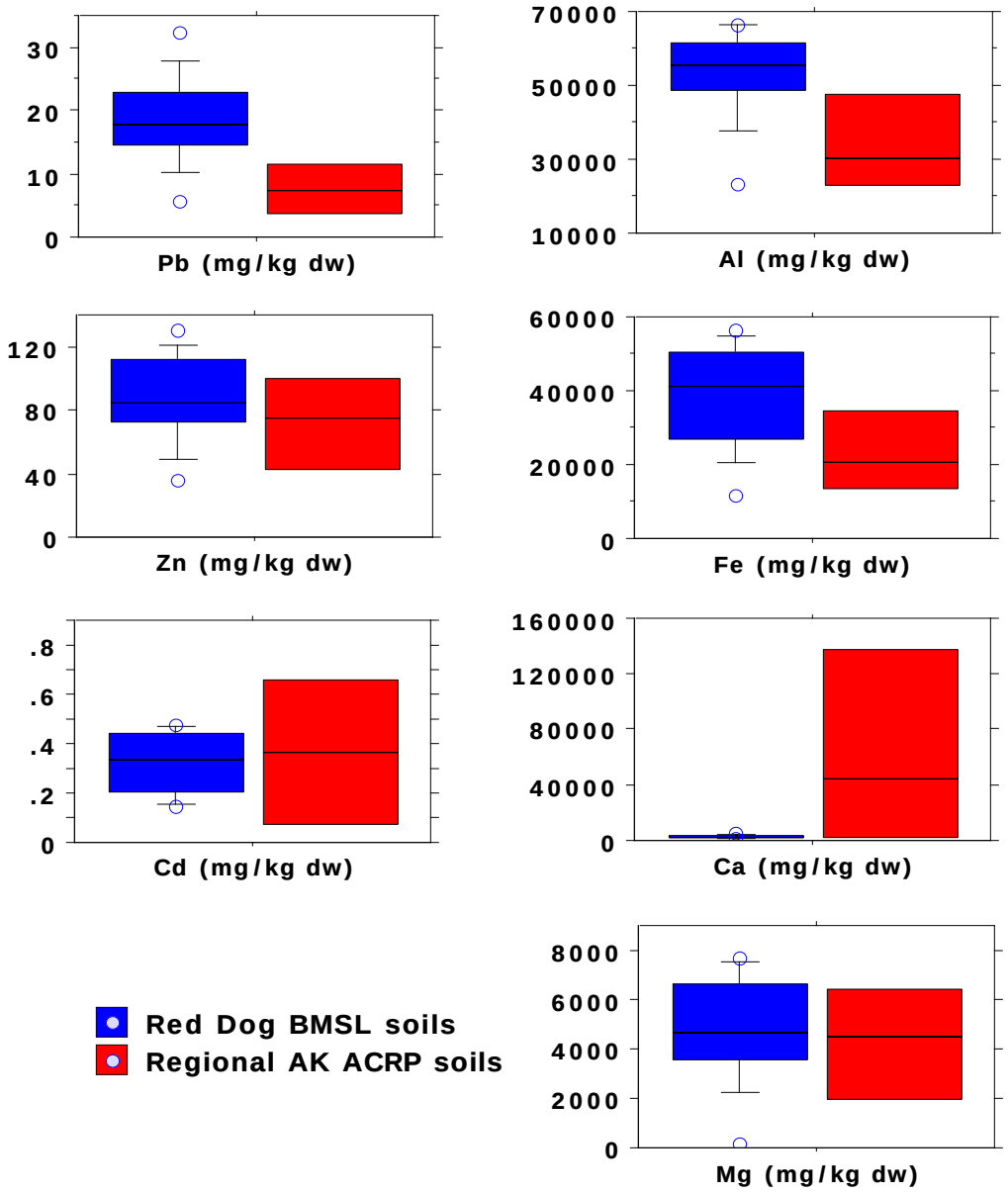

Figure 5. Element concentrations in Red Dog soils at depth compared to those for three sites in arctic Alaska (Schrader/Peters Lake, Elusive Lake (n=2), and Barrow). Boxplots give the median as the line within the box; 75th and 25th percentiles are the upper and lower bounds of the box. Vertical lines extend to the 10th and the 90th percentiles. Open circles represent all remaining (outlier) values. For regional AK, there are data for only four samples; the distribution is fully defined by the median and the box boundaries, and does not have outliers.

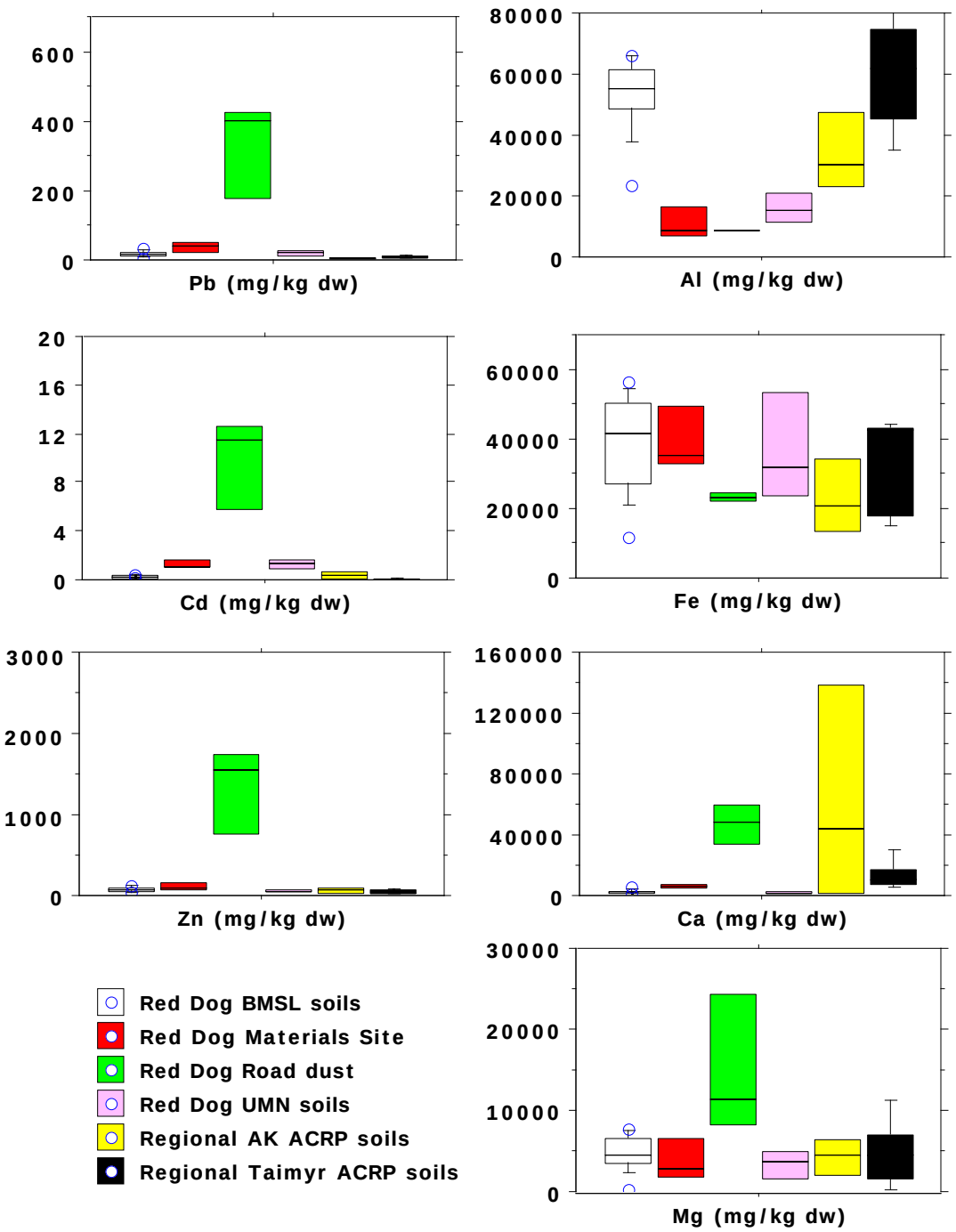

Figure 6. Element concentrations in Red Dog soils at depth, Red Dog road dust, and Red Dog Material Site samples compared to soils at depth from three sites in arctic Alaska Alaska (Schrader/Peters Lake, Elusive Lake (n=2), and Barrow) and five sites on the Taimyr Peninsula, Russia. Boxplots represent data as described in Figure 5.

Elevated concentrations of calcareous materials also appear to be associated with the road. Ca in roadside dust is extremely elevated relative to soils at depth, approaching levels seen at the calcareous Elusive Lake watershed (Fig. 6). The likeliest candidates are the Ca compounds (calcium chloride and/or, in 2000, calcium lignosulfate) used on the road for dust control (John Martinisko, Cominco, pers. comm. March 21, 2000).

5.3 *Hylocomium splendens* moss

Comparison of elemental concentrations in the monitoring moss *H. splendens* to USEPA ACRP regional data for arctic Alaska demonstrates that the Red Dog mosses have extremely high concentrations of all analytes (Fig. 7). Elevated Al, Ca, Fe, and Mg concentrations in the Red Dog mosses suggest contributions of aluminosilicate matrix (i.e., road dust), which is consistent with the depressed enrichment factors found close to the road (section 4). Heavy metals (Pb, Zn, Cd) are also highly elevated relative to the ACRP samples.

Pb is typically low in arctic Alaska *H. splendens* (Ford et al. 1995). The highest concentration previously encountered in the USEPA ACRP regional data set for arctic Alaska was 2.78 mg/kg dw, reported from a heavily dust-contaminated sample collected within 10 m of the Dalton Highway (Ford et al. 1995). Only four other samples in the ACRP data set had values > 1.0 mg/kg dw. By contrast, *H. splendens* Pb values in the current study range from 8.6 to 458 mg/kg dw. A similar although more attenuated pattern exists for Zn and Cd. Interestingly, mosses at 1000 m and 1600 m from the Red Dog Haul Road are rich in Pb, Cd, and Zn, even relative to the samples adjacent to the Dalton Highway.

By contrast to other arctic Alaska sites, moss samples farthest from the haul road on the upwind side contain 4-7 times as much Pb as dust-laden samples taken immediately adjacent to the Dalton Highway. For downwind transects, the comparable figure is 7-19 times the levels adjacent to the Dalton Highway (Ford et al. 1995).

Concentrations of heavy metal elements in *H. splendens* moss from this study are compared to concentrations reported from other Alaska studies in Table 2. The minimum values from the Red Dog moss samples are generally higher than the maximum values from other studies, and the maximum values exceed other reported maxima by one to two orders of magnitude.

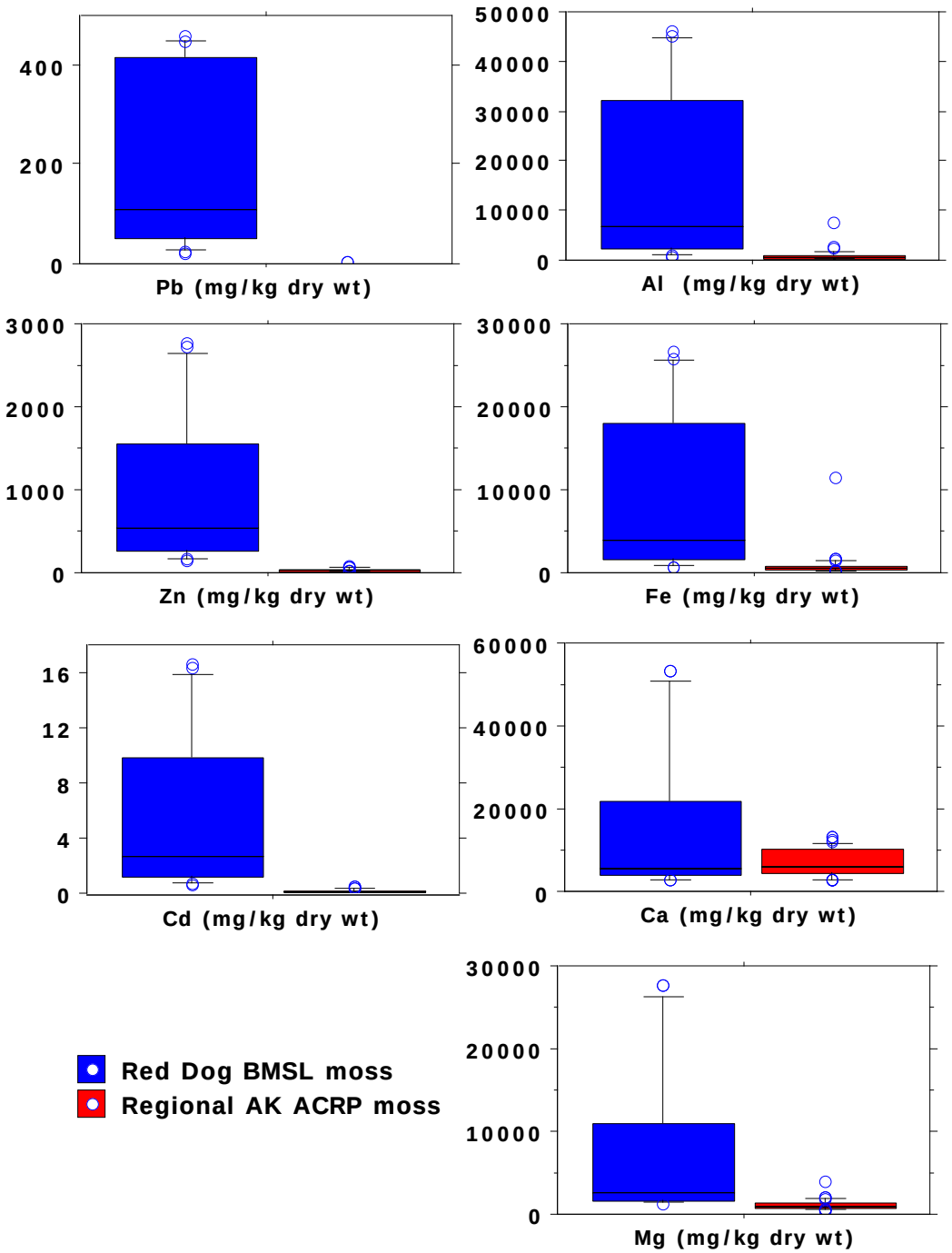

Figure 7. Element concentrations in the monitoring moss ***Hylocomium splendens*** at Red Dog compared to ***H. splendens*** from 18 sites in arctic Alaska. Boxplots represent data as described in Figure 5.

Table 2. Comparison of tissue concentration ranges in *H. splendens* moss in this study to concentrations from other Alaska studies. All units are in mg/kg dw.

Study	Location	Cd	Pb	Zn
This study	Red Dog	0.34 – 17.00	8.6 – 458.0	96 – 2860
Ford et al. 1995	Regional Arctic Alaska	0.03 – 0.42	0.3 – 2.8	14 – 86
Wiersma 1986	Noatak	ND	1.9 – 6.8	58 – 65
Crock et al. 1992a, 1993	Denali	<0.20 – 1.00	0.8 – 10.0	22 – 81
Crock et al. 1993	Kenai	ND	0.6 – 7.0	16 – 77
Crock et al. 1993	Wrangell-St. Elias	ND	<0.6 – 3.2	24 – 60

ND = No data reported

Results from this study show Pb levels exceeding 60 mg/kg dw in all transect points ≤ 100 m. In the Nordic moss monitoring program, *H. splendens* samples exceeding 60-80 mg/kg dw Pb are considered characteristic of highly polluted areas (Rühling and Steinnes 1998). In the 1995 round of the Nordic moss monitoring program, levels exceeding 60 mg/g dw were only attained in Romania, Bulgaria, and hot spots in the Czech Republic, Italy, and Spain (Rühling and Steinnes 1998). Those lead pollution centers were all related to Pb-Zn mining (and sometimes smelting) operations.

The environmental levels of Cd in the Red Dog data set far exceed the maxima reported for severely polluted locations in Central European countries such as the Czech Republic, Poland, Romania, and Bulgaria (Rühling and Steinnes 1998). Almost all moss concentrations from this Red Dog study are greater than the Cd endpoint considered highly polluted in the Nordic moss monitoring program (0.8 mg/kg dw) (Rühling and Steinnes 1998). In fact, moss concentrations of Cd only fall below 1 mg/kg dw at distances of 1 km from the road on the downwind (north) side, and samples with more than 5 mg/kg dw occur even at 50 m on the north side of the road. Whether concentrations fall off further at greater distances from the road is unknown.

Few previous *H. splendens* studies have used the enrichment factor approach to tease out the signal from parent material. Such calculations have, however, been made for *H. splendens* in arctic Alaska, including the western Arctic Coastal Plain and the Noatak Valley (i.e., regional Arctic Alaska) and the Taimyr Peninsula, Russia. Table 3 summarizes the Red Dog data relative to these data. For Pb and Zn, regional arctic Alaska enrichment is considerably less than Red Dog enrichment, strongly supporting the perspective that atmospheric enrichment is qualitatively different in the vicinity of Red Dog than it is in other parts of arctic Alaska. For the Taimyr, the highest enrichment of Pb is seen at the sampling site closest to (ca. 80 km north of) the industrial city of Noril'sk. Cd appears to have a

clear atmospheric component in samples from all areas (i.e., EF>10), but enrichment is substantially higher in the Red Dog samples; and maximum Red Dog raw concentrations (Table 2) exceed regional maxima by two orders of magnitude.

Table 3. Comparison of enrichment factors for *H. splendens* moss in this study to enrichment factors from other arctic Alaska and Siberian studies

	Location	Cd EFs	Pb EFs	Zn EFs
This study	Red Dog	40 - 219	21 - 86	21 - 150
Ford (unpublished)	Regional Arctic Alaska	6 - 132	1 - 12	NC
Allen-Gil et al. (submitted)	Taimyr Peninsula, Russia	3 - 53	2 – 19	2 - 17

NC = Not calculated

6. Summary

A strong road-related depositional gradient was found for all analytes (Ag, Al, Ca, Cd, Fe, Mg, Pb, and Zn) in *Hylocomium splendens* moss, with highest concentrations adjacent to the haul road. Concentrations of Cd in these near-road samples exceed concentrations in regional samples from arctic Alaska, Europe, and Fennoscandia, as well as concentrations in samples from heavily polluted regions in Eastern Europe. Concentrations of all elements decrease rapidly with distance from the road, although heavy metal levels remain elevated 1000 m – 1600 m from the road at transect endpoints. Concentrations of Cd and Pb even at 1000 m and 1600 m from the road exceed medians (and in most cases maxima) from all 28 countries in the Nordic moss monitoring program, including many of the most polluted countries in Central and Eastern Europe and all areas of western Russia.

Enrichment factor analysis of moss versus local soil parent material demonstrates that remobilized soils (e.g., dust composed of roadbed material) account for only a fraction of the elevated heavy metal concentrations in the road corridor. Enrichment in Pb, Zn, and Cd from airborne sources other than remobilized soils (e.g., ore concentrate) is readily apparent. Dust on roadside willow and birch contains very high levels of heavy metals, relative to metal levels in soils at depth and materials sites samples. These findings raise the possibility of airborne heavy metal contributions from mining activities not only to the haul road corridor (via ore concentrate escapement) but also to the Omikviorok River drainage as a whole.

7. Acknowledgments

Shanti Berryman and Todd Bohle provided high quality collection and handling of field samples and thorough sample documentation. We thank Cominco for its cooperation and logistic support to pursue these studies and the NANA-Lynden drivers for their friendliness and courtesy when the field crew was collecting samples close to the road.

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Appendix I

Analysis of data quality from Battelle Marine Sciences Laboratory and U. Minnesota Soils Laboratory

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This appendix summarizes data quality for analyses from both Battelle Marine Sciences Laboratory (BMSL)(12 soil samples, 38 moss samples) and the U. Minnesota Soils Lab (UMN)(4 soil samples, 3 dust samples, 3 materials site samples, and 16 moss samples). Performance on substrate materials is discussed in Section 1.1, and performance on *Hylocomium splendens* moss is discussed in Section 1.2. A summary of performance of both laboratories on all elements appears in a table at the end of each section.

Data were screened for completeness, accuracy, precision, recovery, and contamination. Method detection limits (MDLs) were compared to the actual range of field values to determine whether these potentially affected data use or quality (values above but close to detection limits, while they may pass quality control (QC) screens, are not considered robust). Standard screening criteria were ± 20% of target QC values. An additional 10% "grace" envelope was allowed, which allows flagging rather than failure if targets are narrowly missed. Most data, however, were within ± 10% of target QC values. Data flags were assigned to document where these grace envelopes were applied and to call attention to cases in which QC targets were near or below the MDL. Details of flagging and censoring are described in Appendix II and used in the QA screening tables in Appendix III.

1 Soils.

1.1 Battelle Marine Sciences Laboratory

The 12 soil samples sent to BMSL included samples at depth from all six transects at 3m and 1000m from the road. Samples were analyzed for eight elements (Ag, Al, Ca, Cd, Fe, Mg, Pb, and Zn). One analysis digested samples using a mixture of nitric and hydrofluoric acids with digestates analyzed by ICP-AES for Ag, Pb, and Zn and by GFAA for Cd. A second analysis digested samples using a mixture of nitric, hydrochloric, hydrofluoric, and boric acids with digestates analyzed by ICP-AES for Al, Ca, Fe, and Mg.

Soil loss-on-ignition (= total volatile solids) was determined by ashing for 30 minutes at 550° C. The standard conversion factor of 0.4 was applied to estimate percent total organic carbon (%TOC).

Quality control samples included one reagent blank to assess contamination, two Standard Reference Materials (SRMs) to assess accuracy (used singly or in combination), one duplicate to assess precision, and (for Ag, Cd, Pb, and Zn) two replicate matrix spikes and one blank spike to assess recovery.

1.1.1 BMSL soils detection limits. Method detection limits (MDLs) were suggested for three of the elements analyzed in soils) based on BMSL data produced for other basal soils from arctic Alaska (Ford et al., 1997). MDLs actually achieved by BMSL were 500X and 24X higher than requested for Mg and Pb, respectively, and an order of magnitude lower than requested for Cd. For Mg, the relatively high MDL is much less than 10% of the concentration of the

field samples and so does not pose a problem. The only analyte for which field samples were routinely < 10*MDL was Ag.

1.1.2 BMSL soils contamination. Element concentrations in reagent blanks were above the MDL only for Zn. Blank-correction might be considered for this element, as the blank value was 3.8% of the mean value for field samples. However, the impact of such a correction would be minor. For the purpose of this project, no BMSL soil elements have been blank-corrected.

1.1.3 BMSL soils accuracy. Accuracy SRMs were similar to the range in field samples for Ag, Al, and Fe. Performance was acceptable for all three elements. Accuracy SRMs were 1-2 orders of magnitude higher than field samples for Ca, Cd, Mg, Pb, and Zn; and performance was acceptable for all five elements.

1.1.4 BMSL soils precision. Field samples used for precision estimates came from transects 1S (100m) and 3N (1000m). Precision was acceptable for all elements.

1.1.5 BMSL soils blank spike recoveries. Blank spike recoveries were acceptable when performed. Blank spikes were not performed for Al, Ca, Fe, and Mg.

1.1.6 BMSL soils matrix spike recoveries. Matrix spike recoveries were acceptable when performed. Matrix spikes were not performed for Al, Ca, Fe, and Mg.

1.1.7 BMSL Total Organic Carbon. The 12 soil samples analyzed by BMSL varied from 1.9 to 29.3% TOC. Eight samples from four transects were ≤ 4.1%, and four samples from two transects were >10%, with one sample 29.3% (i.e., a peat soil) (Table 1). Elevated %TOC was seen in both samples from Transects 2N and 3S.

1.1.8 BMSL soil summary. Performance on all elements was acceptable.

1.2 U. Minnesota Soils Laboratory

The 10 substrate samples sent to UMN included

- four soils (samples from transects 1N and 2N at 3m and 1000m from the road)
- three samples of dust shaken from vegetation at plots nearest to the road (3m) at all three downwind transects (1N, 2N, and 3N); and
- samples of fines from berms at three of the materials sites.

All 10 samples were analyzed in a single batch for 15 elements (Al, B, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, and Zn). A 0.5 g sample was digested using EPA method 3051. Soil was leached with 10 ml of trace metal grade HNO_3 for 10 minutes in a sealed Teflon^{TM} vessel. After cooling, the sample was diluted to a final volume of 40 ml with deionized water. All elements were determined by ICP-AES. Soil carbon was determined by ashing a 0.5 g sample in a 1400° C furnace in an oxygen rich atmosphere. Under these conditions, carbon compounds are converted to CO_2, which is measured by an infrared detector.

Quality control samples included one reagent blank to assess contamination, one Standard Reference Material (SRM) to assess accuracy,

three duplicates to assess precision, and (except for Al, B, K, Na, and P) one matrix spike to assess recovery.

1.2.1 UMN soils detection limits. Method detection limits (MDLs) were suggested for three elements analyzed in soil. In a 6 June 2000 memo to L. Hasselbach, UMN responded with actual achievable detection levels on these elements using their standard techniques. Performance was slightly better than predicted. MDLs actually achieved were 9.6X, 1520X, and 134.4X of requested, respectively. For both Pb and Cd, the higher actual MDL resulted in values for most field samples near the detection limit (<10*MDL). For Mg, the actual MDL was still much less than 10% of the concentration of field samples and so does not pose a problem. Other analytes for which routine field values were close to detection limits were B (all field samples <MDL) and Na.

1.2.2 UMN soils contamination. Element concentrations in reagent blanks were > MDL only for Mn and Na. If Na were a desirable target variable, blank correction would be suggested because blank concentrations of Na were 20.6% of the mean value for field samples.

1.2.3 UMN soils accuracy. Unlike BMSL, the UMN laboratory did not do complete digests of substrate materials, but rather only leached the samples. However, the endpoint of interest for the purpose of enrichment factor calculations is totals (not leachables). Therefore, for the purpose of this report, performance on accuracy targets is screened against certified values for total element concentrations in the SRM. It is understood that reanalysis by UMN would likely not improve performance.

Accuracy targets were similar to field samples for Ca, Fe, Mg, Mn, Ni, and Zn. Of these, performance was acceptable for Ca, Mn, Ni, and Zn. Accuracy targets were high relative to field samples for Al, Cd, Cu, K, Na, P, and Pb. Of these, performance was acceptable for all but Al (leaching does not completely decompose aluminosilicates), K and Na (which may also be part of the local aluminosilicate matrix if feldspars are present). As with BMSL, accuracy targets were 1-2 orders of magnitude higher than field samples for Cd and Pb.

Although performance on the SRM was surprisingly good for several target analytes, it was unacceptable for seven elements, including the key element Al that is required to calculate enrichment factors.

1.2.4 UMN soils precision. Three field samples were used for precision estimates (1N3, 1N1000, and MS6). All three were run once with the suite of field samples and then twice as duplicates. For four elements (Al, K, Mg, and Na), concentrations were noticeably higher in the field run than in the duplicate runs. The laboratory explained that the poor match between the field sample and the original duplicate was noticed and generated an additional extraction and analysis (R. Eliason, pers. comm.). In these cases, laboratory policy requires reporting of all data.

Precision was acceptable for all elements except Al, Cr, and K and was within the grace envelope for Cd and Na.

1.2.5 UMN soils blank spike recoveries. Blank spikes were not run with these samples. This is the least useful of the QC targets, and dropping this endpoint is acceptable.

1.2.6 UMN soils matrix spike recoveries. Five elements (Al, B, K, Na, and P) did not include recovery studies using a matrix spike. This was because these elements are not included in the multielement standard routinely used by the laboratory for this purpose. Of the elements spiked, performance was acceptable for Cd, Cr, Cu, Mn, Ni, Pb, and Zn and within the grace envelope for Mg. Recoveries were unacceptable for Ca and Fe.

1.2.7 UMN Total Organic Carbon. The four soil samples analyzed by UMN varied from 1.7% to 30.4 % TOC. As with the analyses by BMSL, samples were either less than 5% or greater than 10% TOC, with very high %TOC for one sample (Table I-1). As with BMSL, elevated %TOC was seen in both samples from Transect 2N.

Table I-1
Comparative laboratory performance on soils at depth

Element	BMSL	UMN
Ag	**A**	na
Al	**F9**	R
Ca	**F9**	**F8**
Cd	**A**	**F5**
Fe	**F9**	R
Mg	**F9**	R
Pb	**A**	**A**
Zn	**F7**	**A**
B	na	R
Cr	na	R
Cu	na	**A**
K	na	R
Mn	na	**F1**
Na	na	R
Ni	na	**F1**
P	na	**F9**
%TOC 1N3	4.8	4.1
%TOC 1N1000	1.7	2.5
%TOC 2N3	11.4	10.6
%TOC 2N1000	30.4	29.3

na = Not analyzed
A = Accept
R = Reject
Fx = Accepted with caution (see Appendix II for key to flags)

1.2.8 UMN soil summary. Performance on Cu, P, Pb, and Zn is acceptable; and performance on Ca, Cd, Ni, and Mn is within grace envelopes. However, high MDLs for Cd and Pb may affect data interpretation. Performance on Al, B, Cr, Fe, K, Mg, and Na is not acceptable.

1.3 Interlaboratory comparisons

Table I-1 compares overall laboratory performance on each element for soils. Of the seven elements analyzed in common, both labs produced acceptable results for Ca, Cd, Pb, and Zn. BMSL, but not UMN, produced acceptable results for soil matrix elements Al, Fe, and Mg, and also for Ag (not analyzed by UMN). UMN also produced acceptable results for four elements not analyzed by BMSL (Cu, Mn, Ni, and P).

On samples from four locations analyzed in common, comparison of the raw data from the two labs yields interesting insights. Data are displayed as bar graphs in Figure I-1. Regressions [UMN on BMSL] are given in Figure I-2. Results are discussed by element.

Aluminum: Unacceptable results by UMN on Al are related to the use of leaching rather than complete digestion techniques. UMN systematically found less than one-quarter the concentration of Al found by BMSL (UMN_{Al} = 3747 + 0.228*$BMSL_{Al}$; R^2 = .817). This is not surprising given the differences in extraction methodologies.

Calcium: Although both laboratories produced acceptable results on Ca, UMN systematically reported lower concentrations on sample splits relative to BMSL (UMN_{Ca} = 1,100 + 0.386*$BMSL_{Ca}$; R^2 = 0.479). These results and the low R^2 are likely due to variable extraction on Red Dog subsoil by the UMN lab.

Cadmium: Both laboratories produced acceptable results, yet BMSL estimates are considerably lower than UMN estimates (UMN_{Cd} = -0.81 + 5.072*$BMSL_{Cd}$; R^2 = 0.673). Neither laboratory used an accuracy target for this element in the same low range of concentrations as actually found in field samples, so neither laboratory has particularly strong grounds on which to accept their results rather than those of the other laboratory. However, BMSL's SRM was an order of magnitude lower than the one employed by UMN. Further, BMSL results are consistent with their previous results for the USEPA ACRP arctic Alaska subsoils (see section 5.3 and Fig. 6). The analyses for that program in turn included round-robin samples from other programs on which BMSL performed comparably to participating laboratories. Finally, BMSL employed a more sensitive analytical technique for this element (GFAA vs ICP-AES). All in all, the available evidence suggests that the BMSL results are likely to be better estimates of actual Cd concentration than the UMN results.

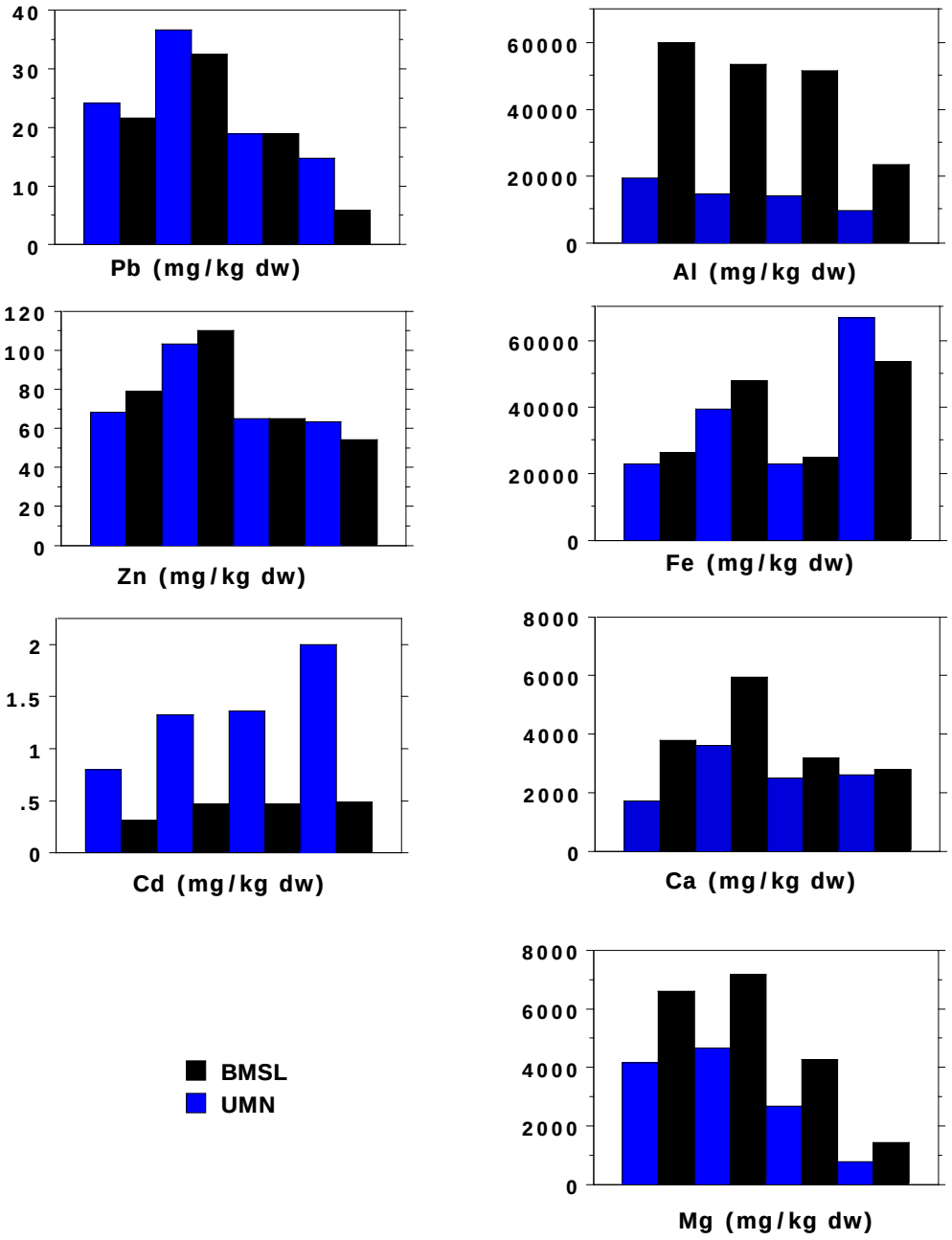

Figure I-1. Bar charts comparing BMSL and UMN laboratory performance on seven heavy metals and trace elements in Red Dog soils at depth.

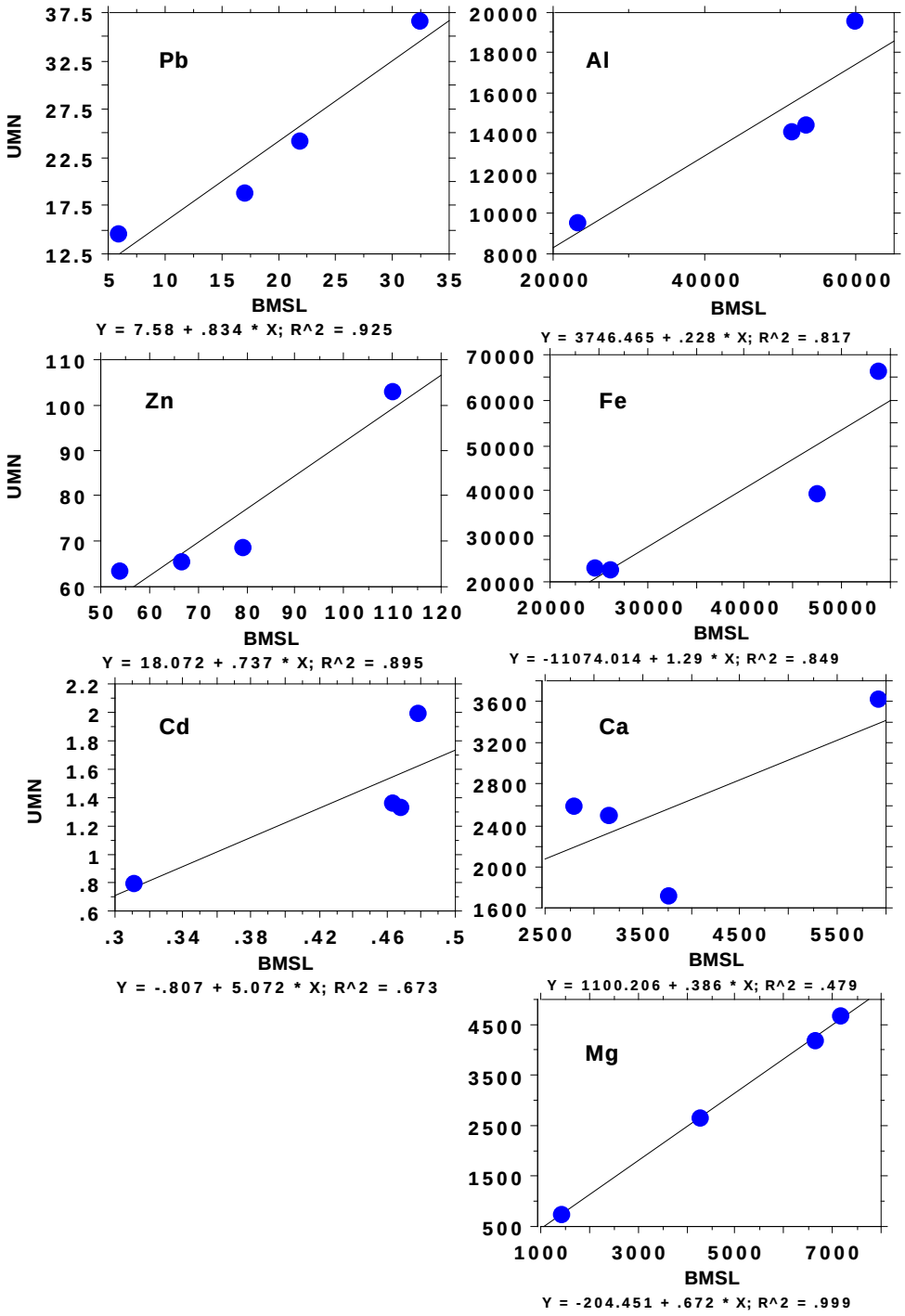

Figure I-2. Regressions of UMN on BMSL results for seven heavy metals and trace elements in Red Dog soils at depth.

Iron: As with Al, UMN produced unacceptable results on iron, probably due to digestion/extraction methodology. Three of the four samples analyzed in common had lower estimated concentrations in the UMN analyses, but the highest concentration sample had a much higher estimated concentration. The predictive equation (UMN_{Fe} = -11074 + 1.29*$BMSL_{Fe}$; R^2 = 0.849) reflects this erratic relationship. The regression is anchored by the two lowest concentration samples.

Magnesium: As with Al and Fe, BMSL but not UMN produced acceptable results on this element. Despite UMN's low estimates of concentration (UMN_{Mg} = -204 + 0.672*$BMSL_{Mg}$), there is an extremely tight relationship between the two data sets (R^2 = 0.999). This suggests that some fraction of the Mg is bound in a labile, easily recoverable fraction, and another fraction is entirely resistant to UMN extraction methodologies.

Lead: Both laboratories produced acceptable results on Pb, although as with Cd UMN produced higher estimates of concentration than did BMSL, especially at the lower end of the range. It is important to note that (1) UMN was working closer to its detection limit than BMSL, (2) UMN results for accuracy on the SRM were biased high, and (3) the UMN SRM provided only an extremely high target value. For these reasons, BMSL results are most likely a more accurate reflection of actual values. The predictive equation (UMN_{Pb} = 7.6 + 0.834*$BMSL_{Pb}$) has R^2 = 0.93.

Zinc: Both laboratories produced acceptable results on this element. The predictive equation (UMN_{Zn} = 18 + 0.737*$BMSL_{Zn}$) has R^2 = 0.895.

Total organic carbon: Field replicates indicate good agreement between laboratories despite methodological differences (Table I-1).

In summary, BMSL provided acceptable results for all target elements. The leaching method currently employed by UMN is unsuitable for complete (total) soil analysis. Extraction techniques are operationally defined procedures, and the fraction of resistant minerals removed depends entirely on the nature of the substrate. UMN would need to develop methods and demonstrate satisfactory performance using appropriate soil SRMs before it should be contracted for analyses of inorganic substrates.

2 Vegetation

2.1 Battelle Marine Sciences Laboratory

The 38 moss samples sent to BMSL represented five stations (3m, 50m, 100m, 250m, and 1000m) at each of six transects, plus an additional station at 1600 m for two of the transects (one upwind and one downwind of the road). In addition,

field replicates were collected at six of the stations. All samples were analyzed for eight elements (Ag, Al, Ca, Cd, Fe, Mg, Pb, and Zn).

Two aliquots of moss were digested. One digestion used nitric and hydrofluoric acids with digestates analyzed by ICP-MS for Ag, Cd, and Pb, and by ICP-AES for Zn. A second digestion using boric and hydrochloric acids in addition to nitric and hydrofluoric acids was used to analyze Al, Ca, Fe, and Mg by ICP-AES.

Two reagent blanks were used to assess contamination. One certified Standard Reference Material (SRM) was used to assess accuracy (NIST 1571). One USGS *H. splendens* intercalibration sample that probably contains dust as well as moss was also used as an uncertified accuracy target. Two duplicates drawn from four samples were used in various combinations to assess precision. Up to four replicate matrix spikes and two blank spikes were used to assess recovery (Ag, Cd, Pb, and Zn only).

2.1.1 BMSL moss detection limits. Method detection limits (MDLs) were suggested for seven of the elements based on previous BMSL analyses of *H. splendens* samples from arctic Alaska (Ford et al., 1997). MDLs actually achieved by BMSL were significantly lower than requested (0.1x to 0.5x) for all elements. The only analyte for which field samples were routinely < 10* MDL was Ag.

2.1.2 BMSL moss contamination. Element concentrations in reagent blanks were > MDL for Al, Ca., Fe, Mg, and Zn, but in no case were they significant relative to the concentrations encountered in field samples. Thus, no blank correction was performed.

2.1.3 BMSL moss accuracy. The certified SRM and the USGS intercalibration sample bracketed the range of concentrations adequately for Ca, Fe, and Zn, and were 1-2 orders of magnitude lower than field samples for Cd, Mg, Pb, and Zn. According to BMSL no accuracy standard exists for Ag in vegetation. Results on two certified accuracy standards were reported for Al in a separate set of analyses after the batch was run; this batch included two field samples for which recoveries were 110% and 92% of the those reported in the original batch (E. Crecelius, pers. comm. 2/9/01). Performance was acceptable for all elements having a certified accuracy target.

2.1.4 BMSL moss precision. Field samples used for precision estimates came from transects 1N (100m), 1S (3 m and 100m), and 2S (1000m). Precision was acceptable for all elements.

2.1.5 BMSL moss blank spike recoveries. Blank spike recoveries, when performed, were acceptable for all elements except Ag, which was underspiked. Blank spikes were not performed for Al, Ca, Fe, and Mg.

2.1.6 BMSL moss matrix spike recoveries. Matrix spike recoveries were acceptable for Ag and Cd and within the grace envelope for Pb. Zn was underspiked. Matrix spikes were not performed for Al, Ca, Fe, and Mg.

2.1.7 BMSL moss summary. Performance on Al, Ca, Cd, Fe, Mg, Pb, and Zn is acceptable. The lack of an accuracy target for Ag causes an automatic failure for this element. However, for the purpose of this report, Ag has been

tentatively accepted, with the caveat that future work must identify an accuracy target.

2.2 U. Minnesota Soils Laboratory

The 16 moss samples sent to UMN included samples from five stations from each of two transects, plus six field replicates. Samples were analyzed in a single batch by ICP-AES for 15 elements (Al, B, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, and Zn).

Quality control samples included reagent blanks at two dilution levels to assess contamination, two Standard Reference materials (SRMs) to assess accuracy, and three duplicates to assess precision. There were no samples independent of the SRM with which to assess recovery.

2.2.1 UMN moss detection limits. Method detection limits (MDLs) were suggested for 10 of the elements analyzed in soils (Al, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, and Zn). In a 6 June 2000 memo to L. Hasselbach, UMN indicated achievable detection levels using two of their standard techniques. MDLs actually achieved were generally between those two targets and were quite close to those requested for all elements except for Pb (requested: 0.5; achieved: 1.68). For the moss data set as a whole, only 8 results from the matrix of 16 samples x 15 analytes (i.e., 3.3%) were <10*MDL. This included two results on Cd, two on Ni, and four on Cr. None of these was <3*MDL.

2.2.2 UMN moss contamination. Element concentrations in reagent blanks were above the method detection for one or both blanks for Ca, Fe, and Zn. However, none of the elements required blank-correction, as field values are consistently >> 100*MDL.

2.2.3 UMN moss accuracy. Accuracy targets were similar to concentrations in field samples for B, Cr, Mg, and P and were high for Ca and K. For all other elements, accuracy targets were low relative to concentrations in field samples, generally by 1-2 orders of magnitude. Performance was acceptable for all elements.

2.2.4 UMN moss precision. Three field samples were used for precision estimates (1N3, 2N3, and 1N1000). Precision was acceptable for all elements.

2.2.5 UMN moss blank spike recoveries. Blank spikes were not run with these samples. This is the least useful of the QC targets, and dropping this endpoint is acceptable.

2.2.6 UMN moss matrix spike recoveries. Matrix spikes were not run with these samples, and they are flagged accordingly (see list of flags in Appendix II).

2.2.7 UMN moss summary. Performance on all elements is acceptable, although additional information on recovery would be helpful, particularly given the high concentrations of most elements in field samples relative to concentrations in the SRM accuracy target.

2.3 Interlaboratory comparisons

Table I-2 compares overall laboratory performance on each element for mosses. Of the seven elements analyzed in common, both labs produced acceptable results for all elements.

Comparison of raw data from the two labs on the 16 samples analyzed in common yields interesting insights. Data are displayed as bar graphs in Figure I-3; with regressions [UMN on BMSL] in Figure I-4. Results are discussed by element.

Aluminum: As with the soil samples, UMN estimates of Al concentration are only about a quarter those of BMSL (Fig. 3). The regression equation reflects this situation ($UMN_{Al} = 920 + 0.289*BMSL_{Al}$). The high R^2 (0.979) suggests that UMN is systematically failing to extract a particular component of dust on the plant surfaces. Taken in combination, these findings suggest that a significant fraction of the moss Al is aluminosilicate matrix (soil or ore dust), because QA indicates that UMN is adequately recovering plant tissue. BMSL's rigorous soil digestion method was also applied to the vegetation samples and so would have dealt successfully with any aluminosilicate matrix present.

Table I-2
Comparative laboratory performance on *H. splendens* moss

Element	BMSL	UMN
Ag	R*	na
Al	**F9**	**F9**
Ca	**F9**	**F9**
Cd	**A**	**F9**
Fe	**F9**	**F9**
Mg	**F9**	**F9**
Pb	**F4, F7**	**F9**
Zn	**F9**	**F9**
B	na	**F9**
Cr	na	**F9**
Cu	na	**F9**
K	na	**F9**
Mn	na	**F9**
Na	na	**F9**
Ni	na	**F9**
P	na	**F9**

na = Not analyzed
A = Accepted
R* = Technically fails, for lack of a certified SRM. Accepted for the purposes of this report only
Fx = Accepted with caution (see Appendix II for key to flags)

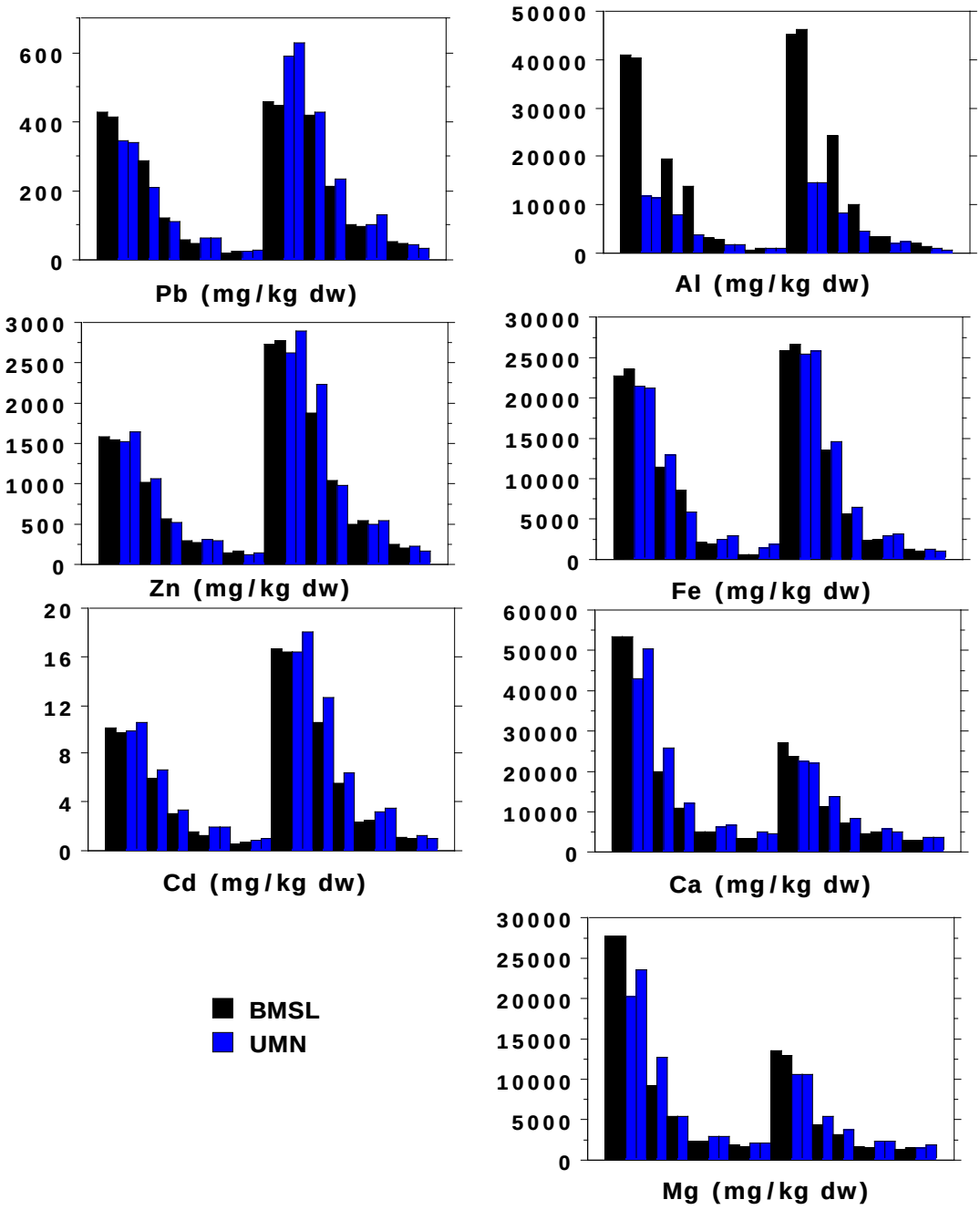

Figure I-3. Bar charts comparing BMSL and UMN laboratory performance on seven trace elements and heavy metals in Red Dog ***Hylocomium splendens*** moss. Laboratory replicates are averaged; values for pairs of field duplicates analyzed by each laboratory are displayed. Samples are sequentially arrayed from plots closest to the road to those farthest from the road in each of two transects (1N and 2N).

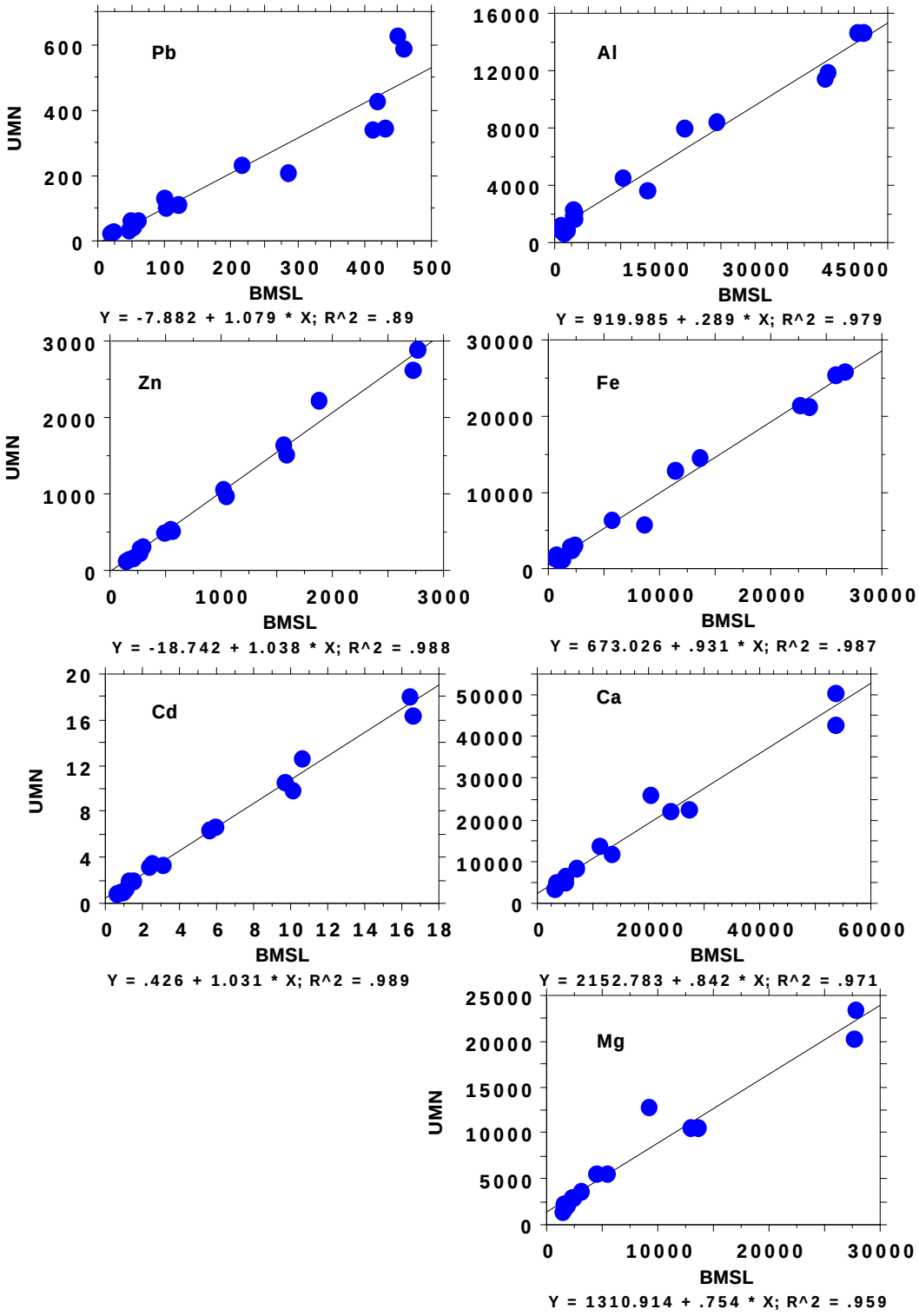

Figure I-4. Regressions of UMN on BMSL results for seven trace elements and heavy metals in Red Dog ***Hylocomium splendens*** moss. All laboratory replicates averaged, but values for pairs of field duplicates analyzed by each laboratory are retained. Units are mg/kg dw.

Calcium: Performance of both laboratories was acceptable on this element, and the predictive equation ($UMN_{Ca} = 2153 + 0.842*BMSL_{Ca}$) has a high R^2 (0.971).

Cadmium: Performance of both laboratories was acceptable on this element. Results from the two laboratories were more similar than they were for soils, although UMN results were still slightly higher than those from BMSL (compare Fig. I-3 to Fig. I-1). The predictive equation ($UMN_{Cd} = -0.426 + 1.031*BMSL_{Cd}$) has a very high R^2 (0.989). Taken together, these results suggest that the bulk of the Cd analyzed may not come from inorganic substrates, which UMN appears to overestimate, but rather from the more analytically tractable moss tissue itself, perhaps as a result of passive or active foliar uptake of Cd from solution.

Iron: Performance of both laboratories was acceptable on this element, and the predictive equation ($UMN_{Fe} = -673 + 0.931*BMSL_{Fe}$) had a high R^2 (0.987).

Magnesium: Performance of both laboratories was acceptable on this element. The predictive equation ($UMN_{Mg} = 1311+ 0.754*BMSL_{Mg}$) had a relatively high R^2 (0.959). Unlike the situation with soil, UMN does not systematically underestimate Mg relative to BMSL (compare Fig. I-3 and Fig. I-1).

Lead: Both laboratories produced acceptable results on this element, although the predictive equation ($UMN_{Pb} = -7.9 + 1.079*BMSL_{Pb}$) has a lower R^2 (0.89) than the other elements.

Zinc: Both laboratories produced acceptable results on this element. The predictive equation ($UMN_{Zn} = -18.7 + 1.038*BMSL_{Zn}$) has a high R^2 (0.988).

In summary, both laboratories produced acceptable results on all seven elements in *H. splendens* moss tissue. Future work on Red Dog moss samples should keep in mind the large range of anticipated concentrations and use the data from this report to plan a multiple SRM strategy accordingly. The lack of a certified SRM for Ag in vegetation will continue to hamper efforts to provide data of documented quality for this analyte on vegetation samples until and unless this deficiency is remedied.

Appendix II

Criteria for censoring and flagging data reported from the analytical laboratories

FLAGS

FLAG	QC TYPE	MEANING
F1	Accuracy	Target is >3*MDL; performance is > (target ±20%) but < (target ±30%)
F2	Accuracy	Target is < 3*MDL; performance is > (target ±20%) but < (target ± 50%) of target, or performance < 3*MDL if target is non-detect (<MDL)
F3	Accuracy	Both target and performance are < 3*MDL
F4	Precision	Both replicates > 3*MDL; RPD > 20% but < 30%
F5	Precision	Both replicates > MDL but < 3*MDL; RPD > 20% but < 50%
F6	Precision	One replicate < MDL; one > MDL but < 3*MDL
F7	Recovery	Performance on one or both matrix spikes is > (100 ± 20%) but < (100 ± 50%) of target; neither > (100 + 50%)
F8	Recovery	Performance on one or both matrix spikes is > (100 + 50%)
F9	Recovery	No matrix spike

DATA CENSORING

Data for a given batch x analyte will be rejected and removed (censored) from the final qualified database if for a given batch x analyte combination:

1. Accuracy QC is missing, or performance is beyond F1 or F2
2. Precision QC is missing, or performance is beyond F4 or F5
3. (F1 or F2) and (F4 or F5)
4. (Matrix spike is missing or F8) and {(F1 or F2) or (F4 or F5)}

Appendix III

Quality assurance screening tables

Analyte **Lab** **Matrix**	**Ag** **BMSL soils**	**Al** **BMSL soils**	**Ca** **BMSL soils**	**Cd** **BMSL soils**	**Fe** **BMSL soils**
Method/Date results rec'd by JF	ICP-AES 1/22/01**	ICP-MS 1/16/01	ICP-MS 1/16/01	GFAA 1/22/01**	ICP-MS 8/10/00
Method Detection Limit (MDL)(µg/g dw)	0.11	5.0	5	0.008	0.5
BLANKS (#)	1	1	1	1	1
#>Method Detection Limit (Value)	0	0	0	0	1 (3.1)
%>Method Detection Limit	0	0	0	0	100
Accept/Reject/Flag	*A*	*A*	*A*	*A*	*R*
BLANK SPIKES (#)	1	NS	NS	1	1
# > 100 ± 20%	0			0	0
% > ± 20%	0			0	0
Accept/Reject/Flag	*A*	*R*	*R*	*A*	*A*
MATRIX SPIKES (#)	2	NS	NS	2	NS
# > 100 ± 20% (recovery)	0			0	
% > ± 20%	0			0	
Accept/Reject/Flag	*A*	*R*	*R*	*A*	*R*
DUPLICATES (#)	1	1	1	1	1
Field sample used for duplicate analyses	1S-100	3N-1000	3N-1000	1S-100	1S-100
# > ± 20%	0	0	0	0	0
% > ± 20%	0	0	0	0	0
Accept/Reject/Flag	*A*	*A*	*A*	*A*	*A*
SRM (# used)	1	1	1	2	2
Standard(s) used	PACS-2*	2704	2704	2704/PACS-2	2704/PACS-2
# > 100 ± 20%	0	0	0	0	0
% > ± 20%	0	0	0	0	0
Accept/Reject/Flag	*A*	*A*	*A*	*A*	*A*
Accept/Reject/ Flag analyte	**A**	**F9**	**F9**	**A**	**F9**

A = Accept
R = Reject
Fx = Flag (see Appendix II for key to flags)
NS = Not spiked
*** = reported 1/12/01**
**** = final BMSL REPORT indicates probably incorrect analysis date of 8/10/00**

Analyte	**Mg**	**Pb**	**Zn**
Lab/Matrix	**BMSL soils**	**BMSL soils**	**BMSL soils**
Method/Date results rec'd by JF	ICP-MS 1/16/01	ICP-AES 1/22/01**	ICP-AES 1/22/01**
Method Detection Limit (MDL) (µg/g dw)	5	1.2	0.2
BLANKS (#)	1	1	1
#>Method Detection Limit (Value)	0	0	1 (3.4)
%>Method Detection Limit	0	0	0
Accept/Reject/Flag	*A*	*A*	*R*
BLANK SPIKES (#)	NS	1	1
# > 100 ± 20%		0	0
% > ± 20%		0	0
Accept/Reject/Flag	*R*	*A*	*A*
MATRIX SPIKES (#)	NS	2	2
# > 100 ± 20% (recovery)		0	1 (121%)
% > ± 20%		0%	50%
Accept/Reject/Flag	*R*	*A*	*R*
DUPLICATES (#)	1	1	1
Field sample used for duplicate analyses	3N-1000	1S-100	1S-100
# > ± 20%	0	0	0
% > ± 20%	0	0	0
Accept/Reject/Flag	*A*	*A*	*A*
SRM (# used)	1	2	2
Standard(s) used	2704	2704/PACS-2	2704/PACS-2
# > 100 ± 20%	0	0	0
% > ± 20%	0	0	0
Accept/Reject/Flag	*A*	*A*	*A*
Accept/Reject/Flag analyte	**F9**	**A**	**F7**

A = Accept
R = Reject
Fx = Flag (see Appendix II for key to flags)
NS = Not spiked
*** = reported 1/12/01**
**** = final BMSL REPORT shows probably incorrect analysis date of 8/10/00**

Analyte	**P**	**K**	**Ca**	**Mg**	**Mn**	**Al**	**Fe**
Lab	**UMN**	**UMN**	**UMN**	**UMN**	**UMN**	**UMN**	**UMN**
Matrix	**soils**	**soils**	**soils**	**soils**	**soils**	**soils**	**soils**
Method/Date reported	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00
Method Detection Limit (MDL) (µg/g dw)	2.8	56.56	3.28	15.2		14.32	1.36
BLANKS (#)	1	1	1	1	1	1	1
#>MDL	0	0	0	0	1	0	0
%>MDL	0	0	0	0	100	0	0
Accept/Reject	*A*	*A*	*A*	*A*	*R*	*A*	*A*
BLANK SPIKES (#)	NS	NS	NS	NS	NS	NS	NS
# > 100 ± 20%							
% > ± 20%							
Accept/Reject	*R*	*R*	*R*	*R*	*R*	*R*	*R*
MATRIX SPIKES (#)	NS	NS	1	1	1	NS	1
# > 100 ± 20%			1	1	0		1
(% Recovery)			(153%)	(125%)			(206%)
% > ± 20%			100	100	0		100
Accept/Reject	*R*	*R*	*R*	*F7*	*A*	*R*	*R*
DUPLICATES (#)	3	3	3	3	3	3	3
Field samples used for duplicates	1N3;1N1000;MS	1N3;1N1000;MS	1N3;1N1000;MS	1N3;1N1000;MS	1N3;1N1000;MS	1N3;1N1000;MS	1N3;1N1000;MS
# > ± 20% (RDP)	0	3	1	0	0	3	0
% > ± 20%	0	100	33	0	0	100	0
Accept/Reject	*A*	*R*	*A*	*A*	*A*	*R*	*A*
SRM (#)	1 (2 runs)	1 (2 runs)	1 (2 runs)	1 (2 runs)	1 (2 runs)	1 (2 runs)	1 (2 runs)
Standard(s) used	NIST2711	NIST2711	NIST2711	NIST2711	NIST2711	NIST2711	NIST2711
# > 100 ± 20%	0	2	0	2	2	2	2
(% Recovery)		(14,14)		(65, 65)	(77,77)	(17, 17)	(52,52)
% > ± 20%	0	100	0	100	100	100	100
Accept/Reject/comments	*A*	*R*	*A*	*R*	*F1*	*R*	*R*
Accept/Reject/ Flag analyte	**F9**	**R**	**F8**	**R**	**F1**	**R**	**R**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)
NC = Not certified

Analyte	**Na**	**Zn**	**Cu**	**B**	**Pb**
Lab	**UMN**	**UMN**	**UMN**	**UMN**	**UMN**
Matrix	**soils**	**soils**	**soils**	**soils**	**soils**
Method/Date reported	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00
Method Detection Limit (µg/g dw)	27.8	0.56	2.08	1.84	6.72
BLANKS (#)	1	1	1	1	1
#>MDL	1	0	0	0	0
%>MDL	100	0	0	0	0
Accept/Reject	*R*	*A*	*A*	*A*	*A*
BLANK SPIKES (#)	NS	NS	NS	NS	NS
# > 100 ± 20%					
% > ± 20%					
Accept/Reject	*R*	*R*	*R*	*R*	*R*
MATRIX SPIKES (#)	NS	1	1	NS	1
# > 100 ± 20% (Recovery)		0	0		0
% > ± 20%		0	0		0
Accept/Reject	*R*	*A*	*A*	*R*	*A*
DUPLICATES (#)	3	3	3	3	3
Field samples used for duplicates	1N3;1N10 00;MS	1N3;1N10 00;MS	1N3;1N10 00;MS	1N3;1N10 00;MS	1N3;1N10 00;MS
# > ± 20% (RPD)	3	0	0	0	1 (34%)
% > ± 20%	100	0	0	0	33
Accept/Reject	*F5*	*A*	*A*	*A*	*A*
SRM (#)	1 (2 runs)	1 (2 runs)	1 (2 runs)	not certified	1 (2 runs)
Standard(s) used	NIST2711	NIST2711	NIST2711	NIST2711	NIST2711
# > 100 ± 20% (Recovery)	2 (2%,2%)	0	0		0
% > ± 20%	100	0	0		0
Accept/Reject/comments	*R*	*A*	*A*	*R*	*A*
Accept/Reject/ Flag analyte	**R**	**A**	**A**	**R**	**A**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)
NC = Not certified

Analyte	**Ni**	**Cr**	**Cd**
Lab	**UMN**	**UMN**	**UMN**
Matrix	**soils**	**soils**	**soils**
Method/Date reported	ICP-AES 10/25/00	ICP-AES 10/25/00	ICP-AES 10/25/00
Method Detection Limit (MDL) (µg/g dw)	1.76	1.12	0.48
BLANKS (#)	1	1	1
#>MDL	0	0	0
%>MDL	0	0	0
Accept/Reject	*A*	*A*	*A*
BLANK SPIKES (#)	NS	NS	NS
# > 100 ± 20%			
% > ± 20%			
Accept/Reject	*R*	*R*	*R*
MATRIX SPIKES (#)	1	1	1
# > 100 ± 20% (Recovery)	0	0	0
% > ± 20%	0	0	0
Accept/Reject	*A*	*A*	*A*
DUPLICATES (#)	3	3	3
Field samples used for duplicates	1N3;1N10 00;MS	1N3;1N10 00;MS	1N3;1N10 00;MS
# > ± 20% (RPD)	0	3 (23%,30%, 40%)	2 (23,44%)
% > ± 20%	0	100	67
Accept/Reject	*A*	*R*	*F5*
SRM (#)	1 (2 runs)	NC	1 (2 runs)
Standard(s) used	NIST2711	NIST2711	NIST2711
# > 100 ± 20% (Recovery)	1 (79%)		0
% > ± 20%	50		0
Accept/Reject	*F1*	*R*	*A*
Accept/Reject/ Flag analyte	**F1**	**R**	**F5**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)
NC = Not certified

Analyte	**Ag**	**Al**	**Ca**	**Cd**	**Fe**
Lab/Matrix	**BMSL moss**	**BMSL moss**	**BMSL moss**	**BMSL moss**	**BMSL moss**
Method/Date reported	ICP-MS (11/29/00)	ICP-AES 12/7/00	ICP-AES 12/7/00	ICP-MS 11/29/00	ICP-AES 12/7/00
Method Detection Limits (MDL) (µg/g dw)	0.05	1.0	1.0	0.05	1.0
BLANKS (#)	2	2	2	2	2
#>MDL (Value)	0	2 (5.68, 7.12)	2 (9.47, 26.4)	0	2 (2.41, 2.99)
%>MDL	0	100	100	0	100
Accept/Reject	*A*	*R*	*R*	*A*	*R*
BLANK SPIKES (#)	SL	NS	NS	2	NS
# > 100 ± 20%				0	
% > ± 20%				0	
Accept/Reject	*R*	*R*	*R*	*A*	*R*
MATRIX SPIKES (#)	2	NS	NS	4	NS
# > 100 ± 20% (Recovery)	0			0	
% > ± 20%	0			0	
Accept/Reject	*A*	*R*	*R*	*A*	*R*
DUPLICATES (#)	2	2	2	2	2
Field samples used for duplicates	1N-100;1S-100	1S-003;2S-100	1S-3;2S-100	1N-100;1S-100	1S-3;2S-100
# > ± 20%	0	0	0	0	0
% > ± 20%	0	0	0	0	0
Accept/Reject	*A*	*A*	*A*	*A*	*A*
SRM:#/ reps each	0	2	3	1(4x)	3
Standard(s) used	None used	1547**, IAEA-336**	1571 (+1547 + IAEA336**)	1571	1571 (+ 1547 + IAEA336**)
# > 100 ± 20%	(% Recovery)	0	0	0	1 (76)
% > ± 20%		0	0	0	25
Accept/Reject	*R*	*A*	*A*	*A*	*A*
Accept/Reject/ Flag analyte	**R**	**F9**	**F9**	**A**	**F9**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)
SL = Inappropriate spike level
****SRM results reported 2/9/01**

Analyte	**Mg**	**Pb**	**Zn**
Lab/Matrix	**BMSL moss**	**BMSL moss**	**BMSL moss**
Method/Date reported	ICP-AES 12/7/00	ICP-MS 11/29/00	ICP-AES 8/14/00
Method Detection Limits (MDL) (µg/g dw)	1.0	0.05	0.16
BLANKS (#)	2	2	2
#>MDL (Value)	2(2.86, 3.35)	0	2 (.654,.619)
%>MDL	2	0	100
Accept/Reject	*R*	*A*	*R*
BLANK SPIKES (#)	NS	2	2
# > 100 ± 20%		0	0
% > ± 20%		0	0
Accept/Reject	*R*	*A*	*A*
MATRIX SPIKES (#)	NS	2	SL
# > 100 ± 20% (Recovery)		1(122%)	
% > ± 20%		50	
Accept/Reject	*R*	*R*	*R*
DUPLICATES (#)	2	2	2
Field samples used for duplicates	1S-3;2S-100	1N-100;1S-100	1N-100;1S-100
# > ± 20%	0	0	0
% > ± 20%	0	0	0
Accept/Reject	*A*	*A*	*A*
SRM:#/ reps each	3	2 (4x)	2 (4x)
Standard(s) used	1571 (+ 1547 + IAEA336**)	366/1571	366/1571
# > 100 ± 20%	0	3	1
(% Recovery)		(121,127,136)	(125)
% > ± 20%	0	38	13
Accept/Reject	*A*	*R*	*A*
Accept/Reject/Flag analyte	**F9**	**F4, F7**	**F9**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)
SL = Inappropriate spike level
****SRM results reported 2/9/01**

Analyte	**P**	**K**	**Ca**	**Mg**	**Mn**	**Al**	**Fe**
Lab/Matrix	**UMN moss**	**UMN moss**	**UMN moss**	**UMN moss**	**UMN moss**	**UMN moss**	**UMN moss**
Method	ICP-AES	ICP-AES	ICP-AES	ICP-AES	ICP-AES	ICP-AES	ICP-AES
Date reported	10/26/00	10/26/00	10/26/00	10/26/00	10/26/00	10/26/00	10/26/00
Method Detection Limit (MDL) (µg/g dw)	0.7	14.14	4.36	3.8	0.06	3.58	0.96
BLANKS (#)	1	1	1	1	1	1	1
#>MDL	0	0	1	0	0	0	1
%>MDL	0	0	100	0	0	0	100
Accept/Reject	*A*	*A*	*R*	*A*	*A*	*A*	*R*
BLANK SPIKES (#)	NS	NS	NS	NS	NS	NS	NS
# > 100 ± 20%							
% > ± 20%							
Accept/Reject	*R*	*R*	*R*	*R*	*R*	*R*	*R*
MATRIX SPIKES (#)	NS	NS	NS	NS	NS	NS	NS
# > 100 ± 20%							
% > ± 20%							
Accept/Reject	*R*	*R*	*R*	*R*	*R*	*R*	*R*
DUPLICATES (#)	3	3	3	3	3	3	3
Field samples used for duplicate analyses	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000
# > ± 20%	0	0	0	0	0	0	0
% > ± 20%	0	0	0	0	0	0	0
Accept/Reject	*A*	*A*	*A*	*A*	*A*	*A*	*A*
SRM (#)	2	2	2	2	2	2	2
Standard(s) used	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547
# > 100 ± 20%	0	0	0	0	0	0	0
% > ± 20%	0	0	0	0	0	0	0
Accept/Reject	*A*	*A*	*A*	*A*	*A*	*A*	*A*
Accept/Reject/ Flag analyte	**F9**	**F9**	**F9**	**F9**	**F9**	**F9**	**F9**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)

Analyte	**Na**	**Zn**	**Cu**	**B**	**Pb**
Lab/Matrix	**UMN moss**	**UMN moss**	**UMN moss**	**UMN moss**	**UMN moss**
Method	ICP-AES	ICP-AES	ICP-AES	ICP-AES	ICP-AES
	10/26/00	10/26/00	10/26/00	10/26/00	10/26/00
Method Detection Limit (MDL) (µg/g dw)	3.6	**0.4**	0.52	0.46	1.68
BLANKS (#)					
#>MDL	0	1	0	0	0
%>MDL	0	100	0	0	0
Accept/Reject	*A*	*R*	*A*	*A*	*A*
BLANK SPIKES (#)	NS	NS	NS	NS	NS
# > 100 ± 20%					
% > ± 20%					
Accept/Reject	*R*	*R*	*R*	*R*	*R*
MATRIX SPIKES (#)	NS	NS	NS	NS	NS
# > 100 ± 20%					
% > ± 20%					
Accept/Reject	*R*	*R*	*R*	*R*	*R*
DUPLICATES (#)	3	3	3	3	3
Field samples used for duplicate analyses	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000	1N3,2N3, 2N1000
# > ± 20%	0	0	0	0	0
% > ± 20%	0	0	0	0	0
Accept/Reject	*A*	*A*	*A*	*A*	*A*
SRM (#)	2	2	2	2	2
Standard(s) used	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547
# > 100 ± 20%	0	0	0	0	0
% > ± 20%	0	0	0	0	0
Accept/Reject	*A*	*A*	*A*	*A*	*A*
Accept/Reject/Flag analyte	**F9**	**F9**	**F9**	**F9**	**F9**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)

Analyte	**Ni**	**Cr**	**Cd**
Lab/Matrix	**UMN moss**	**UMN moss**	**UMN moss**
Method	ICP-AES	ICP-AES	ICP-AES
	10/26/00	10/26/00	10/26/00
Method Detection Limit (MDL) (µg/g dw)	0.44	0.28	0.12
BLANKS (#)			
#>MDL	0	0	0
%>MDL	0	0	0
Accept/Reject	*A*	*A*	*A*
BLANK SPIKES (#)	NS	NS	NS
# > 100 ± 20%			
% > ± 20%			
Accept/Reject	*R*	*R*	*R*
MATRIX SPIKES (#)	NS	NS	NS
# > 100 ± 20%			
% > ± 20%			
Accept/Reject	*R*	*R*	*R*
DUPLICATES (#)	3	3	3
Field samples used for duplicate analyses	1N3,2N3,2 N1000	1N3,2N3,2 N1000	1N3,2N3,2 N1000
# > ± 20%	0	0	0
% > ± 20%	0	0	0
Accept/Reject	*A*	*A*	*A*
SRM (#)	2	2	2
Standard(s) used	NIST 1515, NIST 1547	NIST 1515, NIST 1547	NIST 1515, NIST 1547
# > 100 ± 20%	0	0	0
% > ± 20%	0	0	0
Accept/Reject	*A*	*A*	*A*
Accept/Reject/Flag analyte	**F9**	**F9**	**F9**

A= Accept
Fx = Flag (see Appendix II for key to flags)
MDL = Method detection limit
NS = Not spiked
R = Reject (see Appendix II for rejection/censoring criteria)

Appendix IV

Raw data provided by the analytical laboratories

BATTELLE MARINE SCIENCES LABORATORY
1529 West Sequim Bay Road
Sequim, Washington 98382-9099

360/681-3604

RED DOG HAUL ROAD
METALS IN SOIL

(concentrations in µg/g dry wt - not blank corrected)

Sponsor Code	Lab Code	Ag	Al	Ca	Cd	Fe
	Instrument	*ICP-AES*	*ICP-MS*	*ICP-MS*	*GFAA*	*ICP-MS*
	Date reported to Jford	*22 Jan 2001*	*16 Jan 2001*	*16 Jan 2001*	*22 Jan 2001*	*14 Aug 2000*
00-100-SO-1S-0003-M-99-37	1542-1	0.864	66089	2761	0.366	53961
00-107-SO-1S-100-M-99-26	1542-2	0.692	54375	2364	0.436	27443
00-171-SO-2S-0003-M-99-42	1542-3	0.876	62663	3283	0.252	56368
00-173-SO-2S-1000-M-99-27	1542-4	0.979	66307	2679	0.150	46229
00-181-SO-3S-0003-M-99-43	1542-5	1.256	43683	1473	0.206	35397
00-187-SO-3S-1000-M-99-36	1542-6	0.983	46776	2049	0.157	11614
00-109-SO-1N-0003-M-99-33	1542-7	0.888	59701	3760	0.311	26163
00-151-SO-1N-1000-M-99-48	1542-8	0.747	53390	5927	0.468	47405
00-129-SO-2N-0003-M-99-43	1542-9	2.682	51466	3154	0.463	24611
00-136-SO-2N-1000-M-99-43	1542-10	0.400	23153	2785	0.478	53685
00-160-SO-3N-0003-M-99-54	1542-11	0.831	60913	3690	0.371	46538
00-165-SO-3N-1000-M-99-53	1542-12	0.798	56603	2743	0.205	36902

RED = mean value of laboratory replicates

BATTELLE MARINE SCIENCES LABORATORY
1529 West Sequim Bay Road
Sequim, Washington 98382-9099
360/681-3604

RED DOG HAUL ROAD

METALS IN SOIL

Sponsor Code	**Mg**	**Pb**	**Zn**	%Total Volatile Solids	% Total organic carbon
	ICP-MS	*ICP-AES*	*ICP-AES*	*% Loss-on -ignitiion @ 550C*	*calculated by J. Ford as 0.4 * LOI*
	16 Jan 2001	*22 Jan 2001*	*22 Jan 2001*	*17 Nov 2000*	*N/A*
00-100-SO-1S-0003-M-99-37	6501	17.7	115.0	9.39	3.8
00-107-SO-1S-100-M-99-26	4807	24.4	98.0	5.83	2.3
00-171-SO-2S-0003-M-99-42	7719	18.1	117.0	4.78	1.9
00-173-SO-2S-1000-M-99-27	7480	17.1	80.3	9.27	3.7
00-181-SO-3S-0003-M-99-43	3357	12.4	89.2	36.2	14.5
00-187-SO-3S-1000-M-99-36	4420	11.9	35.6	25.8	10.3
00-109-SO-1N-0003-M-99-33	6636	21.8	79.1	10.3	4.1
00-151-SO-1N-1000-M-99-48	7158	32.4	110.0	6.19	2.5
00-129-SO-2N-0003-M-99-43	4271	17.0	66.6	26.6	10.6
00-136-SO-2N-1000-M-99-43	1421	5.8	54.0	73.2	29.3
00-160-SO-3N-0003-M-99-54	6843	21.8	130.0	6.93	2.8
00-165-SO-3N-1000-M-99-53	5828	25.8	78.6	6.31	2.5

RED = mean value of laboratory replicates

BATTELLE MARINE SCIENCE CENTER: RED DOG METALS IN SOILS QC DATA

	Ag	Al	Ca	Cd	Fe
Detection limits	0.11	5.0	5.0	0.008	0.5
METHOD BLANKS					
Blank	0.11 U	5.0 U	5.0 U	0.008 U	3.1
DUPLICATE PRECISION					
1542-2 R1	0.654			0.447	28251
1542-2 R2	0.729	NA	NA	0.425	26634
RPD	**11%**	**ND**	**ND**	**5%**	**6%**
1542*12 R1		56551	2790		
1542*12 R2	NA	56654	2696	NA	NA
RPD	**ND**	**0%**	**3%**	**ND**	**ND**
STANDARD REFERENCE MATERIAL					
2704	1.07	59622	26081	3.38	36871
Certified value		61100	26000	3.45	41100
range	NC				
Percent difference	**ND**	**2%**	**0%**	**2%**	**10%**
PACS-2	1.41	NA	NA	2.24	42517
Certified value	1.22			2.1	40891
range					
Percent difference	**16%**	**ND**	**ND**	**7%**	**4%**
BLANK SPIKES					
Concentration spiked	25.0	NS	NS	25.0	NS
BLANK	0.11 U			0.008 U	
BS1	24.7			26.4	
Concentration Recovered	24.7			26.4	
% REC	**98%**	**ND**	**ND**	**105%**	**ND**
MATRIX SPIKE S					
Concentration spiked	25.0	NS	NS	25.0	NS
1542 (MEAN)	0.692			0.436	
1542-2 MSD	24.8			25.527	
Concentration Recovered	24.1			25.5	
% REC	**97%**	**ND**	**ND**	**102%**	**ND**
Concentration spiked	25.0	NS	NS	25.0	NS
1542 (MEAN)	0.692			0.436	
1542-2 MSD	24.5			25.7	
Concentration Recovered	23.8			25.3	
% REC	**95%**	**ND**	**ND**	**101%**	**ND**

NA = NOT ANALYZED; ND= NOT DETERMINED; NS= NOT SPIKED

BATTELLE MARINE SCIENCE CENTER: RED DOG METALS IN SOILS QC DATA (cont'd)

		Mg	Pb	Zn
Detection limits		5.0	1.2	0.2
METHOD BLANKS				
Blank		5.0 U	1.2 U	3.36
Duplicate Precision				
1542-2 R1			22.9	99.7
1542-2 R2		NA	26.0	96.4
RPD		**ND**	**13%**	**3%**
1542*12 R1		5834		
1542*12 R2		5823	NA	NA
RPD		**0%**	**ND**	**ND**
STANDARD REFERENCE MATERIAL				
2704		11472	150	430
	Certified value range	12000	161	438
	Percent difference	**4%**	**7%**	**2%**
PACS-2		NA	168	344
	Certified value range		183	364
	Percent difference	**ND**	**8%**	**5%**
BLANK SPIKES				
Concentration spiked	NS		25.0	25.0
BLANK			0.958	3.36
BS1			26.9	27.3
Concentration Recovered			26.9	23.9
% REC	**ND**		**104%**	**96%**
MATRIX SPIKE RESULTS				
Concentration spiked	NS		25.0	25.0
1542 (MEAN)			24.4	98.0
1542-2 MSD			47.5	124.0
Concentration Recovered			23.1	25.7
% REC	**ND**		**92%**	**103%**
Concentration spiked	NS		25.0	25.0
1542 (MEAN)			24.4	98.0
1542-2 MSD			46.1	128.0
Concentration Recovered			21.7	30.3
% REC	**ND**		**87%**	**121%**

NA = NOT ANALYZED; ND= NOT DETERMINED; NS= NOT SPIKED

U. Minnesota Soils Laboratory RED DOG SOILS

all data in µg/g dry wt

Lab ID	Transect	Distance	Matrix x	P (reported 8/18/00)	K (reported 8/18/00)	CA (reported 8/18/00)	MG (reported 8/18/00)	MN (reported 8/18/00)	AL (reported 8/18/00)
117	1N	3	SOIL	157.8	1637	2046	4655	177.2	24637
152	1N	1000	SOIL	420.8	1367	3946	5137	871.0	17524
146	2N	3	SOIL	658.0	983	2502	2652	78.5	14141
135	2N	1000	SOIL	1502.6	647	2591	763	613.9	9574
150	1N	3	DUST	517.3	1340	63055	28539	440.7	8608
127	2N	3	DUST	482.9	1677	30438	11517	424.5	9043
191	3N	3	DUST	680.5	1253	48652	7246	371.6	8707
159			Mat. Site 3	584.6	1302	4613	1672	992.2	6749
189			Mat. Site 5	900.4	2780	7655	7933	2147.2	18954
190			Mat. Site 6	361.0	1186	6992	2872	2601.6	9078
				RED = mean value of laboratory replicates					
Blank				<2.8	56.56	<3.28	<15.2	0.72	<14.32
Duplicates									
117 Dup				145.025	633.273	1575.095	3961.082	152.473	17601.606
117 Dup2				149.700	552.022	1531.197	3901.401	148.819	16521.230
152 Dup				423.127	725.462	3511.408	4477.919	898.377	13147.433
152 Dup2				432.112	715.436	3425.426	4429.150	890.571	12701.505
190 Dup				374.054	767.968	6879.281	2607.648	2691.432	7007.489
190 Dup2				325.997	689.116	6281.500	2405.212	2547.204	6770.817
Standard Reference Material									
NIST 2711 Ck2				789.8	3432	23456	6813	491.3	11003
NIST 2711 Ck				780.5	3393	23424	6799	490.5	10923
NIST 2711 certified value				860	24,500	28,800	10,500	638	65,300

U. Minnesota Soils Laboratory **RED DOG SOILS**

all data in µg/g dry wt

Lab ID	Transect	Distance	Matrix	FE (reported 8/18/00)	NA (reported 8/18/00)	ZN (reported 8/18/00)	CU (reported 8/18/00)	B (reported 8/18/00)	PB (reported 8/18/00)
117	1N	3	SOIL	24160	321.1	72.6	20.3	< 1.841	25.6
152	1N	1000	SOIL	40306	214.6	107.8	24.7	< 1.839	35.3
146	2N	3	SOIL	23006	110.2	65.4	48.5	< 1.843	18.8
135	2N	1000	SOIL	66606	148.5	63.5	30.6	< 2.303	14.7
150	1N	3	DUST	21848	132.9	1568.0	26.2	< 1.841	405.4
127	2N	3	DUST	23490	102.4	1808.7	26.4	< 1.842	432.8
191	3N	3	DUST	25318	64.2	497.3	19.2	< 1.841	104.9
159			Mat. Site 3	35568	117.9	110.4	28.1	< 1.842	45.1
189			Mat. Site 5	54121	278.5	184.6	23.7	< 1.844	53.8
190			Mat. Site 6	32298	164.4	65.4	36.7	< 1.844	17.3
				RED = mean value of laboratory replicates					
Blank				<1.36	27.76	<.56	<2.08	<1.840	<6.720
Duplicates									
117 Dup				22004.410	75.916	65.986	19.459	<1.842	25.305
117 Dup2				21798.955	119.984	67.121	19.624	<1.842	21.946
152 Dup				38994.001	66.978	100.956	24.705	<2.046	37.803
152 Dup2				38588.999	97.538	100.340	24.817	<1.842	36.917
190 Dup				27252.860	43.564	65.265	35.475	<1.842	17.858
190 Dup2				26573.666	100.301	59.571	30.467	<1.844	12.668
Standard Reference Material									
NIST 2711 Ck2				15063	184.4	304.7	110		1280.1
NIST 2711 Ck				15005	176.9	303.5	108		1283.9
NIST 2711 certified value				28,900	11,400	350.4	114		1162

U. Minnesota Soils Laboratory **RED DOG SOILS**

all data in µg/g dry wt

Lab ID	Transect	Distance	Matrix	NI (reported 8/18/00)	CR (reported 8/18/00)	CD (reported 8/18/00)	% Total Carbon (7/24/00 report)
117	1N	3	SOIL	41.393	47.88	0.801	4.79
152	1N	1000	SOIL	62.443	40.70	1.279	1.73
146	2N	3	SOIL	29.327	26.44	1.362	11.37
135	2N	1000	SOIL	28.142	18.83	2.003	30.37
150	1N	3	DUST	32.74	19.05	11.527	3.54
127	2N	3	DUST	33.24	21.63	13.056	2.66
191	3N	3	DUST	33.38	17.61	3.922	2.82
159			Mat. Site 3	34.755	12.41	1.121	1.02
189			Mat. Site 5	49.389	38.40	1.844	0.96
190			Mat. Site 6	46.021	23.33	1.122	0.49

RED = mean value of laboratory replicates

Blank	<1.760	<1.120	<0.480
Duplicates			
117 Dup	36.597	36.196	0.881
117 Dup2	36.203	34.762	0.721
152 Dup	59.328	32.555	1.512
152 Dup2	56.857	32.112	1.201
190 Dup	40.120	15.616	0.721
190 Dup2	36.560	15.474	1.122
Standard Reference Material			
NIST 2711 Ck2	16.3		47.1
NIST 2711 Ck	16.6		46.8
NIST 2711 certified value	20.6		41.7

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

Sponsor Code	Lab Code	Al	Ca	Fe	Mg
	Instrument	***ICP-AES***	***ICP-AES***	***ICP-AES***	***ICP-AES***
	Analysis Date	***12/7/00***	***12/7/00***	***12/7/00***	***12/7/00***
00-101-HS-1S-0003-M	1542-13 R1	31800	34500	19300	16600
00-101-HS-1S-0003-M	1542-13 R2	32400	35000	19600	17000
00-102-HS-1S-0050-M	1542-14	4750	7000	3060	2640
00-104-HS-1S-0100-M	1542-15 R1	4710	4660	3020	2310
00-104-HS-1S-0100-M	1542-15 R2	NA	NA	NA	NA
00-106-HS-1S-0250-M	1542-16	1370	3580	685	1760
00-108-HS-1S-1000-M	1542-17	511	3260	402	1460
00-116-HS-1S-1600-M	1542-18	762	2840	532	1400
00-172-HS-2S-0003-M	1542-19	29400	34700	17100	15300
00-179-HS-2S-0050-M	1542-20	5930	10900	3650	4960
00-177-HS-2S-0100-M	1542-21 R1	3570	7020	2260	3020
00-177-HS-2S-0100-M	1542-21 R2	3860	7250	2410	3180
00-175-HS-2S-0250-M	1542-22	1620	4150	1030	2050
00-174-HS-2S-1000-M	1542-23	465	2520	325	1630
00-180-HS-3S-0003-M	1542-24	31700	17500	18900	8260
00-183-HS-3S-0050-M	1542-25	8750	6450	5410	2920
00-185-HS-3S-0100-M	1542-26	2610	3720	2140	1470
00-186-HS-3S-0250-M	1542-27	1730	3070	1070	1590
00-188-HS-3S-1000-M	1542-28	678	2570	445	1560
00-118-HS-1N-0003-M2	1542-29	40900	53500	22700	27700
00-110-HS-1N-0003-M	1542-30	40400	53500	23500	27800
00-112-HS-1N-0050-M	1542-31	19400	20206	11400	9230
00-114-HS-1N-0100-M	1542-32 R1	13800	10900	8600	5470
00-114-HS-1N-0100-M	1542-32 R2	NA	NA	NA	NA
00-114-HS-1N-0100-M	1542-32	NA	NA	NA	NA
00-122-HS-1N-0250-M2	1542-33	3050	5080	2100	2300
00-120-HS-1N-0250-M	1542-34	2790	4990	1970	2250
00-154-HS-1N-1000-M	1542-35	861	3440	571	1870
00-153-HS-1N-1000-M2	1542-36	1040	3310	702	1630
00-128-HS-2N-0003-M	1542-37	45300	27100	25900	13600
00-147-HS-2N-0003-M2	1542-38	46300	23800	26600	12900
00-131-HS-2N-0050-M	1542-39	24300	11100	13600	4470
00-133-HS-2N-0100-M	1542-40	10100	6940	5690	3090
00-134-HS-2N-0250-M	1542-41	3500	4620	2290	1580
00-141-HS-2N-0250-M2	1542-42	3470	4980	2420	1540
00-138-HS-2N-1000-M2	1542-43	1920	2950	1250	1330
00-137-HS-2N-1000-M	1542-44	1430	3040	1020	1540
00-161-HS-3N-0003-M	1542-45	32800	51900	19500	24000
00-169-HS-3N-0050-M	1542-46	22200	24600	13400	9600
00-168-HS-3N-0100-M	1542-47	14200	14700	8600	6350
00-166-HS-3N-0250-M	1542-48	3060	5960	2120	2780

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

Sponsor Code	Lab Code	Al	Ca	Fe	Mg
00-164-HS-3N-1000-M	1542-49	1070	3950	802	2230
00-163-HS-3N-1600-M	1542-50	1370	3730	964	1750
Detection Limits		1.0	1.0	1.0	1.0
METHOD BLANKS					
Blank R1		5.68	9.47	2.41	2.86
Blank R2		7.12	26.4	2.99	3.35
STANDARD REFERENCE MATERIAL					
366 (USGS Moss)		8072	9831	4643	2765
		7281	9438	4181	2683
		NA	NA	NA	NA
		NA	NA	NA	NA
	reference value	11000	10800	5500	3300
	percent difference	**27%** &	**9%**	**16%**	**16%**
		34% &	**13%**	**24%** &	**19%**
		NA	NA	NA	NA
		NA	NA	NA	NA
1571 (Orchard Leaves)		391	20595	309	5920
		378	20378	293	5801
		NA	NA	NA	NA
		NA	NA	NA	NA
	certified value	**NC**	20900	300	6200
	range		±300	±20.0	±200
	percent difference	NA	**1%**	**3%**	**5%**
		NA	**2%**	**2%**	**6%**
		NA	NA	NA	NA
		NA	NA	NA	NA
BLANK SPIKE RESULTS					
Amount Spiked		NS	NS	NS	NS
Blank R1		NS	NS	NS	NS
Blank Spike		NS	NS	NS	NS
Amount Recovered		NS	NS	NS	NS
Percent Recovery		NS	NS	NS	NS
Amount Spiked		NS	NS	NS	NS
Blank R2		NS	NS	NS	NS
Blank Spike		NS	NS	NS	NS
Amount Recovered		NS	NS	NS	NS
Percent Recovery		NS	NS	NS	NS

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

	Al	Ca	Fe	Mg
MATRIX SPIKE RESULTS				
Amount Spiked	NS	NS	NS	NS
00-104-HS-1S-0100-M 1542-15 (mean)	NS	NS	NS	NS
00-104-HS-1S-0100-M 1542-15 MS	NS	NS	NS	NS
Amount Recovered	NS	NS	NS	NS
Percent Recovery	NS	NS	NS	NS
Amount Spiked	NS	NS	NS	NS
00-104-HS-1S-0100-M 1542-15 (mean)	NS	NS	NS	NS
00-104-HS-1S-0100-M 1542-15 MSD	NS	NS	NS	NS
Amount Recovered	NS	NS	NS	NS
Percent Recovery	NS	NS	NS	NS
Amount Spiked	NS	NS	NS	NS
00-114-HS-1N-0100-M 1542-32 (mean)	NS	NS	NS	NS
00-114-HS-1N-0100-M 1542-32 MS	NS	NS	NS	NS
Amount Recovered	NS	NS	NS	NS
Percent Recovery	NS	NS	NS	NS
Amount Spiked	NS	NS	NS	NS
00-114-HS-1N-0100-M 1542-32 (mean)	NS	NS	NS	NS
00-114-HS-1N-0100-M 1542-32 MSD	NS	NS	NS	NS
Amount Recovered	NS	NS	NS	NS
Percent Recovery	NS	NS	NS	NS
POST DIGESTION MATRIX SPIKE RESULTS				
Amount Spiked	NS	NS	NS	NS
00-104-HS-1S-0100-M 1542-15 (mean)	NS	NS	NS	NS
00-104-HS-1S-0100-M 1542-15 MS	NS	NS	NS	NS
Amount Recovered	NS	NS	NS	NS
Percent Recovery	NS	NS	NS	NS
Amount Spiked	NS	NS	NS	NS
00-114-HS-1N-0100-M 1542-32 (mean)	NS	NS	NS	NS
00-114-HS-1N-0100-M 1542-32 MS	NS	NS	NS	NS
Amount Recovered	NS	NS	NS	NS
Percent Recovery	NS	NS	NS	NS

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

		Al	Ca	Fe	Mg
REPLICATE ANALYSIS RESULTS					
00-101-HS-1S-0003-M	1542-13 R1	31800	34500	19300	16600
00-101-HS-1S-0003-M	1542-13 R2	32400	35000	19600	17000
	Relative percent difference	**2%**	**1%**	**2%**	**2%**
00-104-HS-1S-0100-M	1542-15 R1	4710	4660	3020	2310
00-104-HS-1S-0100-M	1542-15 R2	NA	NA	NA	NA
	Relative percent difference	NA	NA	NA	NA
00-177-HS-2S-0100-M	1542-21 R1	3570	7020	2260	3020
00-177-HS-2S-0100-M	1542-21 R2	3860	7250	2410	3180
	Relative percent difference	**8%**	**3%**	**6%**	**5%**
00-114-HS-1N-0100-M	1542-32 R1	13800	10900	8600	5470
00-114-HS-1N-0100-M	1542-32 R2	NA	NA	NA	NA
	Relative percent difference	NA	NA	NA	NA

U Not detected at or above DL shown

& QC value outside the accuracy or precision criteria goal: spike accuracy ± 20% recovery; replicate precision <20% (RPD); SRM accuracy <20% (PD).

SL Inappropriate spike level

NS Not spiked

NA Not applicable

NC Not certified

(1) Analyzed by GFAA

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

Sponsor Code	Lab Code	Cd	Pb	Ag	Zn
	Instrument	***ICP-MS***	***ICP-MS***	***ICP-MS***	***ICP-AES***
	Analysis Date	***11/30/04***	***11/30/04***	***11/30/04***	***8/15/04***
00-101-HS-1S-0003-M	1542-13 R1	9.42	392	0.235	1500
00-101-HS-1S-0003-M	1542-13 R2	NA	NA	NA	NA
00-102-HS-1S-0050-M	1542-14	2.20	87.2	0.0640	352
00-104-HS-1S-0100-M	1542-15 R1	1.29	38.8	0.0750 (1)	208
00-104-HS-1S-0100-M	1542-15 R2	1.19	35.4	0.0685 (1)	205
00-106-HS-1S-0250-M	1542-16	0.945	25.0	0.05 U	148
00-108-HS-1S-1000-M	1542-17	0.597	8.56	0.05 U	111
00-116-HS-1S-1600-M	1542-18	0.418	11.6	0.05 U	96.1
00-172-HS-2S-0003-M	1542-19	7.22	288	0.191	1200
00-179-HS-2S-0050-M	1542-20	1.82	64.1	0.0563	321
00-177-HS-2S-0100-M	1542-21 R1	1.19	45.5	0.0627	255
00-177-HS-2S-0100-M	1542-21 R2	NA	NA	NA	NA
00-175-HS-2S-0250-M	1542-22	0.655	22.4	0.05 U	138
00-174-HS-2S-1000-M	1542-23	0.343	10.1	0.05 U	118
00-180-HS-3S-0003-M	1542-24	17.0	408	0.864	2860
00-183-HS-3S-0050-M	1542-25	3.84	139	0.215	751
00-185-HS-3S-0100-M	1542-26	2.33	83.5	0.138	453
00-186-HS-3S-0250-M	1542-27	1.07	40.5	0.0582	222
00-188-HS-3S-1000-M	1542-28	0.488	16.8	0.05 U	112
00-118-HS-1N-0003-M2	1542-29	10.1	430	0.518	1590
00-110-HS-1N-0003-M	1542-30	9.66	413	0.516	1550
00-112-HS-1N-0050-M	1542-31	5.92	285	0.311	1020
00-114-HS-1N-0100-M	1542-32 R1	3.13	122	0.152	550
00-114-HS-1N-0100-M	1542-32 R2	3.05	120	0.157	557
00-114-HS-1N-0100-M	1542-32	NA	NA	0.210 (1)	NA
00-122-HS-1N-0250-M2	1542-33	1.53	60.6	0.0844	297
00-120-HS-1N-0250-M	1542-34	1.27	49.6	0.0656	265
00-154-HS-1N-1000-M	1542-35	0.597	19.4	0.05 U	141
00-153-HS-1N-1000-M2	1542-36	0.727	24.1	0.05 U	164
00-128-HS-2N-0003-M	1542-37	16.6	458	0.904	2720
00-147-HS-2N-0003-M2	1542-38	16.4	448	0.954	2770
00-131-HS-2N-0050-M	1542-39	10.6	419	0.668	1880
00-133-HS-2N-0100-M	1542-40	5.61	215	0.335	1040
00-134-HS-2N-0250-M	1542-41	2.37	102	0.114	493
00-141-HS-2N-0250-M2	1542-42	2.48	98.7	0.130	538
00-138-HS-2N-1000-M2	1542-43	1.11	53.0	0.114	258
00-137-HS-2N-1000-M	1542-44	0.915	46.4	0.0578	215
00-161-HS-3N-0003-M	1542-45	7.00	402	0.404	1180
00-169-HS-3N-0050-M	1542-46	4.95	193	0.287	856
00-168-HS-3N-0100-M	1542-47	3.67	140	0.220	695
00-166-HS-3N-0250-M	1542-48	1.17	44.0	0.0711	259

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

Sponsor Code	Lab Code	Cd	Pb	Ag	Zn
	Instrument	***ICP-MS***	***ICP-MS***	***ICP-MS***	***ICP-AES***
	Date	***11/30/04***	***11/30/04***	***11/30/04***	***8/15/04***
00-164-HS-3N-1000-M	1542-49	0.572	21.4	0.0663	158
00-163-HS-3N-1600-M	1542-50	0.614	30.4	0.05 U	169
Detection Limits		0.05	0.05	0.05	0.16
METHOD BLANKS					
Blank R1		0.05 U	0.05 U	0.05 U	0.654
Blank R2		0.05 U	0.05 U	0.05 U	0.619
STANDARD REFERENCE MATERIAL					
366 (USGS Moss)		NR	3.29	0.05 U	39.2
		NR	3.05	0.05 U	40.7
		NR	3.53	0.05 U	40.2
		NR	3.18	0.05 U	41.3
	reference value		2.60		36.0
	PD	NA	**27%** &	NA	**9%**
		NA	**17%**	NA	**13%**
		NA	**36%** &	NA	**12%**
		NA	**22%**	NA	**15%**
1571 (Orchard Leaves)		0.120	43.0	0.05 U	31.2
		0.109	40.5	0.05 U	27.3
		0.108	43.0	0.05 U	27.2
		0.105	40.6	0.05 U	27.6
	certified value	0.11	45	NC	25.0
	range	±0.01	±3.0		±3.0
	PD	**9%**	**4%**	NA	**25%**
		1%	**10%**	NA	**9%**
		2%	**4%**	NA	**9%**
		5%	**10%**	NA	**11%**
BLANK SPIKE RESULTS					
Amount Spiked		12.5	12.5	SL	12.5
Blank R1		0.05 U	0.05 U	SL	0.654
Blank Spike		13.1	14.1	SL	13.0
Amount Recovered		13.1	14.1	SL	12.3
Percent Recovery		105%	113%	SL	99%
Amount Spiked		12.5	12.5	SL	12.5
Blank R2		0.05 U	0.05 U	SL	0.619
Blank Spike		13.4	14.9	SL	14.1
Amount Recovered		13.4	14.9	SL	13.5
Percent Recovery		107%	119%	SL	108%

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

Sponsor Code	Lab Code	Cd	Pb	Ag	Zn
	Instrument	***ICP-MS***	***ICP-MS***	***ICP-MS***	***ICP-AES***
	Date	***11/30/04***	***11/30/04***	***11/30/04***	***8/15/04***
MATRIX SPIKE RESULTS					
Amount Spiked		12.5	12.5	2.00	SL
00-104-HS-1S-0100-M	1542-15 (mean)	1.24	37.1	0.0718 (1)	SL
00-104-HS-1S-0100-M	1542-15 MS	14.6	50.7	1.87 (1)	SL
Amount Recovered		13.4	13.6	1.80	SL
Percent Recovery		107%	109%	90%	SL
Amount Spiked		12.5	12.5	NS	SL
00-104-HS-1S-0100-M	1542-15 (mean)	1.24	37.1	NS	SL
00-104-HS-1S-0100-M	1542-15 MSD	14.5	52.4	NS	SL
Amount Recovered		13.3	15.3	NS	SL
Percent Recovery		106%	122%	NS	SL
MATRIX SPIKE RESULTS					
Amount Spiked		12.5	SL	2.00	SL
00-114-HS-1N-0100-M	1542-32 (mean)	3.09	SL	0.210 (1)	SL
00-114-HS-1N-0100-M	1542-32 MS	16.8	SL	2.10 (1)	SL
Amount Recovered		13.7	SL	1.89	SL
Percent Recovery		110%	SL	95%	SL
Amount Spiked		12.5	SL	NS	SL
00-114-HS-1N-0100-M	1542-32 (mean)	3.09	SL	NS	SL
00-114-HS-1N-0100-M	1542-32 MSD	15.8	SL	NS	SL
Amount Recovered		12.7	SL	NS	SL
Percent Recovery		102%	SL	NS	SL
POST DIGESTION MATRIX SPIKE RESULTS					
Amount Spiked		2.00	SL	SL	NS
00-104-HS-1S-0100-M	1542-15 (mean)	1.24	SL	SL	NS
00-104-HS-1S-0100-M	1542-15 MS	3.20	SL	SL	NS
Amount Recovered		1.96	SL	SL	NS
Percent Recovery		98%	SL	SL	NS
Amount Spiked		2.00	SL	SL	NS
00-114-HS-1N-0100-M	1542-32 (mean)	3.09	SL	SL	NS
00-114-HS-1N-0100-M	1542-32 MS	5.12	SL	SL	NS
Amount Recovered		2.03	SL	SL	NS
Percent Recovery		102%	SL	SL	NS

BATTELLE MARINE SCIENCES LABORATORY

RED DOG: METALS IN MOSS (concentrations in µg/g dry wt - not blank corrected)

Sponsor Code	Lab Code	Cd	Pb	Ag	Zn
	Instrument	***ICP-MS***	***ICP-MS***	***ICP-MS***	***ICP-AES***
	Analysis Date	***11/30/04***	***11/30/04***	***11/30/04***	***8/15/04***
REPLICATE ANALYSIS RESULTS					
00-101-HS-1S-0003-M	1542-13 R1	9.42	392	0.235	1500
00-101-HS-1S-0003-M	1542-13 R2	NA	NA	NA	NA
	Relative percent difference	NA	NA	NA	NA
00-104-HS-1S-0100-M	1542-15 R1	1.29	38.8	0.0232 (1)	208
00-104-HS-1S-0100-M	1542-15 R2	1.19	35.4	0.0231 (1)	205
	Relative percent difference	**8%**	**9%**	**0%**	**1%**
00-177-HS-2S-0100-M	1542-21 R1	1.19	45.5	0.0627	255
00-177-HS-2S-0100-M	1542-21 R2	NA	NA	NA	NA
	Relative percent difference	NA	NA	NA	NA
00-114-HS-1N-0100-M	1542-32 R1	3.13	122	0.152	550
00-114-HS-1N-0100-M	1542-32 R2	3.05	120	0.157	557
	Relative percent difference	**3%**	**2%**	**3%**	**1%**

U Not detected at or above DL shown

& QC value outside the accuracy or precision criteria goal: spike accuracy ± 25% recovery; replicate precision <25% (RPD); SRM accuracy <25% (PD).

SL Inappropriate spike level

NS Not spiked

NA Not applicable

NC Not certified

(1) Analyzed by GFAA

U. MINNESOTA SOILS LABORATORY: RED DOG MOSS
all data in µg/g dry wt

Sample ID	Field ID	P	K	CA	MG	MN
DilBlk		<7.0	<141.4	<8.2	<38.0	<0.6
hc1s01 Ck		263.17	1001.1	1108.6	263.2	11.09
hc1s01 Ck%		%105.3	%100.1	%110.9	%105.3	%110.9
BL		<0.700	< 14.140	4.36	< 3.800	< 0.060
CRM482 Ck		678.52	3538.6	2381.6	524.4	30.6
CRM482 Ck%						
SRM1515 Ck3		1596.84	16041.2	15623.2	2685.6	52.72
SRM1547 Ck2		1417.58	24184	15896.6	4306.8	96.04
119	00-119-HS-1N-0003-M-M2	505	2505.2	40558	19197.4	470.64
119 Dup	00-119-HS-1N-0003-M-M2	561.4	2474.2	44952	21282	470.9
119 Avg	00-119-HS-1N-0003-M-M2	533.2	2489.7	42755	20239.7	470.77
119 rd%	00-119-HS-1N-0003-M-M2	%10.578	%1.245	%10.277	%10.300	%0.055
124	00-124-HS-1N-0003-M-M1	579.6	2280.4	50372	23444	389.9
158	00-158-HS-1N-0050-M-M1	712.32	2343.6	25918	12776.2	441.72
157	00-157-HS-1N-0100-M-M1	937.62	3179.4	12021.8	5481.8	478.54
123	00-123-HS-1N-0250-M-M2	1261.42	3544.4	6095.4	2943.2	417.3
121	00-121-HS-1N-0250-M-M1	1221.1	3438.8	6491.8	2940.6	480.8
155	00-155-HS-1N-1000-M-M1	1173.38	3525.8	4873.2	2048.4	589.48
156	00-156-HS-2N-1000-M-M2	1125.68	3446.8	4766.2	2163.8	512.6
148	00-148-HS-2N-0003-M-M1	685.2	2665.8	22654	10672.6	626.2
149	00-149-HS-2N-0003-M-M2	642.6	2431.4	22334	10598.4	650.52
149 Dup	00-149-HS-2N-0003-M-M2	648	2495	22234	10487.2	653.14
149 Avg	00-149-HS-2N-0003-M-M2	645.3	2463.2	22284	10542.8	651.83
149 rd%	00-149-HS-2N-0003-M-M2	%0.837	%2.582	%0.449	%1.055	%0.402
145	00-149-HS-2N-0050-M-M1	1044.86	2752.2	13571.6	5509.4	722.3
144	00-144-HS-2N-0100-M-M1	1204.04	2921.8	8531	3668.6	891.98
142	00-142-HS-2N-0250-M-M1	1397.16	3101.6	5653.4	2239.4	277.94
143	00-143-HS-2N-0250-MP-M2	1559.48	3274	4896.8	2299	448.04
139	00-139-HS-2N-1000-MP-M1	813.62	2393.4	3586.6	1420.14	920.16
140	00-140-HS-2N-1000-M-M2	1223.76	2955.6	3549.2	1800.9	958.06
140 Dup	00-140-HS-2N-1000-M-M2	1248.52	2986.8	3556.6	1803.94	964.88
140 Avg	00-140-HS-2N-1000-M-M2	1236.14	2971.2	3552.9	1802.42	961.47
140 rd%	00-140-HS-2N-1000-M-M2	%2.003	%1.050	%0.208	%0.169	%0.709

U. MINNESOTA SOILS LABORATORY: RED DOG MOSS

all data in µg/g dry wt

Sample ID	Field ID	AL	FE	NA	ZN	CU
DilBlk		< 35.800	< 3.400	< 36.000	< 1.400	< 5.200
hc1s01 Ck		10.72	11.19	99.83	10.93	10.6
hc1s01 Ck%		%107.2	%111.9	%99.8	%109.3	%106.0
BL		< 3.580	0.96	< 3.600	0.4	< 0.520
CRM482 Ck		733.86	788.38	51.34	102.82	7
CRM482 Ck%		* 66.533			* 102.207	% 99.573
SRM1515 Ck3		270.24	74.66	25.18	12.82	6.08
SRM1547 Ck2		225.34	204.94	26.64	18.66	4.26
119	00-119-HS-1N-0003-M-M2	11344.8	20396	260.22	1428	25.96
119 Dup	00-119-HS-1N-0003-M-M2	12498.6	22640	262.68	1596.2	26.2
119 Avg	00-119-HS-1N-0003-M-M2	11921.7	21518	261.45	1512.1	26.08
119 rd%	00-119-HS-1N-0003-M-M2	%9.678	%10.428	%0.941	%11.124	%0.920
124	00-124-HS-1N-0003-M-M1	11442	21170	286.04	1655.8	30.3
158	00-158-HS-1N-0050-M-M1	7966.4	13019.2	262.88	1066.3	18.34
157	00-157-HS-1N-0100-M-M1	3690	5847.4	177.74	515.92	11.56
123	00-123-HS-1N-0250-M-M2	1701.64	2526.6	140.58	303.28	11.66
121	00-121-HS-1N-0250-M-M1	1907.62	2840	162.38	296.08	10.92
155	00-155-HS-1N-1000-M-M1	905.96	1417.32	207.82	132.48	9.32
156	00-156-HS-2N-1000-M-M2	1199.28	1916.24	193.66	144	6.86
148	00-148-HS-2N-0003-M-M1	14701.2	25422	255.56	2618.6	32.2
149	00-149-HS-2N-0003-M-M2	14679.2	25758	236.4	2890	33.48
149 Dup	00-149-HS-2N-0003-M-M2	14725.4	25702	240	2900	33.2
149 Avg	00-149-HS-2N-0003-M-M2	14702.3	25730	238.2	2895	33.34
149 rd%	00-149-HS-2N-0003-M-M2	%0.314	%0.218	%1.511	%0.345	%0.840
145	00-149-HS-2N-0050-M-M1	8446	14503.6	224.8	2232.8	22.1
144	00-144-HS-2N-0100-M-M1	4527.4	6560.8	164.1	970.44	14.22
142	00-142-HS-2N-0250-M-M1	2099.2	2861.4	163.98	507.66	7.8
143	00-143-HS-2N-0250-MP-M2	2339.2	3150.8	107.68	534.62	10.56
139	00-139-HS-2N-1000-MP-M1	885.18	1267.26	98.64	224.38	6.54
140	00-140-HS-2N-1000-M-M2	697.8	971.12	119.02	169.72	10.46
140 Dup	00-140-HS-2N-1000-M-M2	701.64	965.76	117.88	167.92	8.74
140 Avg	00-140-HS-2N-1000-M-M2	699.72	968.44	118.45	168.82	9.6
140 rd%	00-140-HS-2N-1000-M-M2	%0.549	%0.553	%0.962	%1.066	%17.917

U. MINNESOTA SOILS LABORATORY: RED DOG MOSS
all data in µg/g dry wt

Sample ID	Field ID	B	PB	NI	CR	CD
DilBlk		< 4.600	<16.800	< 4.400	< 2.800	< 1.200
hc1s01 Ck		2.11	5.64	2.25	2.19	2.29
hc1s01 Ck%		%105.5	%112.8	%112.5	%109.5	%114.5
BL		< 0.460	< 1.680	< 0.440	< 0.280	< 0.120
CRM482 Ck		2.68	38.74	2.56	2.66	0.56
CRM482 Ck%			*94.7	*103.644	*64.63	%100.0
SRM1515 Ck3		28.28	< 1.680	0.98	0.5	< 0.120
SRM1547 Ck2		27.84	< 1.680	0.86	1.04	< 0.120
119	00-119-HS-1N-0003-M-M2	14.24	324.8	30.4	22.4	9.4
119 Dup	00-119-HS-1N-0003-M-M2	14.16	361.8	34.2	24.4	10.2
119 Avg	00-119-HS-1N-0003-M-M2	14.2	343.3	32.3	23.4	9.8
119 rd%	00-119-HS-1N-0003-M-M2	%0.6	%10.8	%11.8	%8.5	%8.2
124	00-124-HS-1N-0003-M-M1	13	342.4	33.4	23.2	10.6
158	00-158-HS-1N-0050-M-M1	11.8	209.5	21.14	13.24	6.72
157	00-157-HS-1N-0100-M-M1	8.18	110.82	9.64	6.26	3.3
123	00-123-HS-1N-0250-M-M2	6.64	61.66	5.12	2.92	1.9
121	00-121-HS-1N-0250-M-M1	7.74	62.38	5.68	3.32	1.88
155	00-155-HS-1N-1000-M-M1	9.62	24.44	5.06	1.8	0.9
156	00-156-HS-2N-1000-M-M2	8.6	27.84	6	2.46	0.96
148	00-148-HS-2N-0003-M-M1	15.52	588.8	34.6	25.2	16.4
149	00-149-HS-2N-0003-M-M2	14.78	624.4	34.6	25	17.6
149 Dup	00-149-HS-2N-0003-M-M2	14.76	633.6	34.6	25.6	18.4
149 Avg	00-149-HS-2N-0003-M-M2	14.77	629	34.6	25.3	18
149 rd%	00-149-HS-2N-0003-M-M2	%0.1	%1.5	%0.0	%2.4	%4.4
145	00-149-HS-2N-0050-M-M1	12.94	427.6	18.24	12.24	12.64
144	00-144-HS-2N-0100-M-M1	15.16	235.38	9.76	6.26	6.44
142	00-142-HS-2N-0250-M-M1	9.46	102.52	5	3.24	3.26
143	00-143-HS-2N-0250-MP-M2	8.84	129.54	5.16	3.38	3.52
139	00-139-HS-2N-1000-MP-M1	6.62	44.98	3.68	1.34	1.2
140	00-140-HS-2N-1000-M-M2	8.1	33.12	2.74	1.02	1.06
140 Dup	00-140-HS-2N-1000-M-M2	8.32	33.18	2.7	1.04	1
140 Avg	00-140-HS-2N-1000-M-M2	8.21	33.15	2.72	1.03	1.03
140 rd%	00-140-HS-2N-1000-M-M2	%2.7	%0.2	%1.5	%1.9	%5.8