Multi-element removal from road-deposited sediments using weak hydrochloric acid

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Abstract Dilute HCl is a widely applied partial digestion procedure in geo-environmental investigations. Concentrations of HCl from 0.07 to 12 M have been applied to digest solid media, either shaken at room temperature ("cold") or heated to temperatures of 80–95 °C ("hot"). From an exhaustive literature survey, 0.5 M HCl was selected as the most appropriate single leach partial digestion procedure for examining potentially contaminated road-deposited sediments (RDSs). Both cold and hot HCl treatments were examined using objective criteria to identify the leach that produced the most environmentally meaningful data. The treatments were judged according to minimal breakdown of the aluminosilicate lattice and/or liberation of refractory-associated fractions of Al, Cr, and Fe; high recoveries for suspected contaminants (Cu, Pb, and Zn); and contaminant concentrations independent of clay content, organic C content, carbonate content, and cation exchange capacity. Based on all criteria, the cold 0.5 M HCl procedure was identified as superior to the hot HCl treatment. The cold treatment showed limited removal of Al, Cr, and Fe from the residual fraction, with the hot treatment having three- to sevenfold greater removal efficiencies. This suggests significant breakdown of structural lattice and liberation of elements from fractions not generally considered environmentally important. Hot HCl removed quantitative amounts of Cu and Zn from the road sediments, indicating structural release. The greater structural breakdown associated with hot HCl is a major disadvantage for studies examining bioavailability. All data point to the superiority of a cold HCl leach for geo-environmental contaminant studies. Electronic supplementary material to this paper can be obtained by using the Springer LINK server located at http://dx.doi.org/10.1007/s00254-002-0598-8.

Keywords Dilute HCl · Multi-element · Partial digestion · Road-deposited sediments · Total digestion

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Introduction

Partial dissolution procedures have a long history of use in the geological, environmental, and geochemical exploration fields. The investigator's choice of dissolution procedure is a function of the goals of the study and availability of a method that has undergone suitable replication. In general, partial dissolution techniques can be classified as single dissolution and sequential multiple dissolution techniques. They can also be classified, on a relative basis, as "nonselective" or "selective" extractions depending on the degree of specificity of the extraction (Chao 1984). Nonselective single extraction procedures are the most popular, with significantly less expertise or effort required compared with sequential extraction procedures. Various inorganic acids have been used to partially digest solid media (Sulcek and Povondra 1989; Bajo 1992; Chao and Sanzolone 1992), with one criterion for selection being limited digestion of the silicate lattice. Dilute acids remove elements from several fractions, such as soluble, exchangeable, specifically adsorbed, held on, or occluded in, oxides of Fe, Al, or Mn, and new precipitates (Beckett 1989).

The one inorganic acid that has been most commonly applied in dilute form in geo-environmental investigations is HCl. Hydrochloric acid, in dilute form, from 0.1 to 1.0 M, is now adopted by at least three countries as a standard procedure, including Canada (Stone and Marsalek 1996), Korea (Yun and others 2000), and in the US (ASTM 1990). Though "standards" exist there are no certified reference materials (CRMs) with certified, or informational values for element concentrations for dilute HCl extractions. This, coupled with the variety of HCl concentrations used (Fig. 1), and

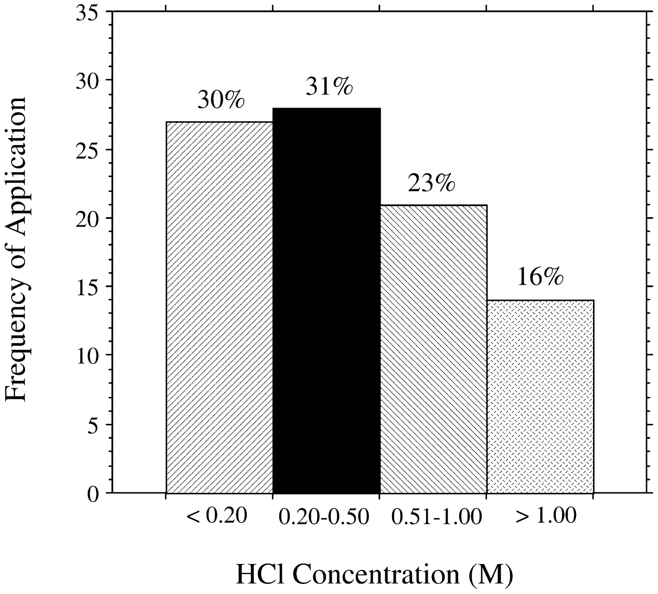


Fig. 1
Literature summary of geo-environmental investigations that have applied HCl in different concentrations to solid media. *Numeric values* above *bars* represent the percentage of applications for a specific range of HCl concentration relative to a total of 90 applications. Note that some reference sources may apply HCl at levels that fall into several different categories in this figure. A complete list of the sources used to develop this figure can be found in the electronic supplementary material of this journal (http://www.link.springer.de/link/service/journals/00254/tocs.htm)

differences in experimental conditions, make meaningful comparison between similar solid media problematic.

Of the 90 applications examined to develop Fig. 1, the HCl concentrations applied varied from 0.07 M "cold" (room temperature) to 12 M "hot" (95 °C), with 10% of the studies using heat. The most frequent concentration applied was 0.5 M (26 of 90). The use of cold 0.5 M HCl (pH ~0.3) was recommended by Agemian and Chau (1976, 1977) because it met several criteria: it satisfied the essential requirement of minimal attack on the silicate detrital lattice; liberated metals from organic matter; had satisfactory simultaneous ability to extract selected metals from the non-detrital fractions in sediment; and produced high contrasts between anomalous and background samples. Duinker and others (1974) examined several dilute concentrations of HCl (0.01, 0.1, 0.5, 1, and 2 M) and concluded that considering effectiveness and selectivity in terms of clay mineral lattice breakdown, treatment with 0.1 M HCl (pH ~1.2) for 18 h appeared to be the most preferable leaching agent. Malo (1977) recommended hot 0.3 M HCl (pH ~0.7) for routine use in monitoring the readily acid-soluble minor elements in aquatic sediments. Few detailed geo-environmental studies are available in the literature documenting the multi-element recovery of partial digestions relative to total contents. Therefore, this study quantifies the extraction efficiencies of 17 elements from road-deposited sediment using a cold and hot dilute HCl protocol. Additionally, criteria are established to judge the geo-environmental applicability of hot and cold extractions for selected elements.

Materials and methods

Study site

Palolo Valley watershed (11.4 km^2) is located in SE Oahu. Land use consists of 55% undeveloped conservation land, 41% urbanized, and 4% agricultural (Ikeno 1996). In 1990, Palolo had 4,097 households and a resident population of 13,034 (State of Hawaii 1998). Of 2,640 housing units in Palolo in 1980, 80.1% were single family, 10.6% apartment buildings, 8.6% duplexes, and 0.7% townhouses (City and County of Honolulu 1980). Streets in Palolo Valley are scheduled for street sweeping on a weekly basis, subject to availability of equipment and personnel (G. Takayesu, City and County of Honolulu, personal communication, Oct. 1999). However, there are a number of narrow streets within the valley with numerous parked and abandoned vehicles, preventing implementation of a comprehensive street-sweeping program. Storm event runoff from roads is directly input into stream channels via a storm sewer network. A storm runoff study has shown that streams in this valley are more adversely affected by urban runoff than neighboring systems (Ikeno 1996).

Sample collection, processing, and analyses

Road sediments from curbside sites were collected using a Nalgene scoop and placed in plastic bags for transport to the laboratory. Sites were selected to provide representative coverage of all traffic levels anticipated in the basin. Typical sample mass collected was between 300 and 600 g, over a curb length of about 1 m. Subsamples of RDS were oven-dried at 105 °C to a constant mass (~24 h) and passed through an acid-washed 2,000-μm nylon sieve. All bulk (<2 mm) RDS samples were analyzed for texture, organic carbon, pH, calcium carbonate, and cation exchange capacity following the methods described in Sutherland and others (2000).

Road sediments for elemental analysis and grain size analysis were oven-dried at ≤ 40 °C for 7 days. Samples were passed through a 2,000-μm nylon sieve to isolate a bulk sample. All material was then ground in a Pica Blender Mill (Model 2601, Cianflone Scientific Instruments Corp., Pittsburgh, PA) for 5 min in 30-ml tungsten carbide vials with tungsten carbide balls to pass a 63-μm nylon mesh sieve.

One 0.5 g aliquot was totally digested with HNO_3, $HClO_4$, and HF acids to dryness overnight on a hot plate. The residue was solubilized with HCl and diluted to volume. The first partial extraction involved shaking 0.5 g samples with 10 ml of room temperature 0.5 M HCl for 1 h (Sutherland 2002). Though cold ≤ 0.1 M HCl exhibits less breakdown of the silicate lattice compared with 0.5 M HCl, there are problems with the quantity of metal liberated being a function of $CaCO_3$ content (McNeal and others 1985), and re-adsorption produces low element recoveries (Sholkovitz 1989). The second procedure involved the same sample mass:volume ratio of 0.5 M HCl, but samples were heated for 1 h at 90–95 °C in a water bath. Seventeen elements (Al, Ba, Ca, Co, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, Sr, V, and Zn) were analyzed with ICP-AES for all digestions.

Quality control

Precision and accuracy for total digestion element concentrations were determined using five replicates of the certified reference materials (CRMs) GSD-7 (fluvial sediment) certified by the China National Analysis Center for Iron and Steel, and SRM 2711 (Montana soil) from the US National Institute of Standards and Technology (NIST). Precision was very satisfactory for both CRMs (Table 1) with a median of 1.6% for GSD-7 and a range from 1.1 (Ba) to 4.4% (Cu); and for SRM 2711 the median was 1.7%, with a range from 0.8 (Sr) to 5.7% (Co). Accuracy was also satisfactory, with a median for GSD-7 of 2.2% (–6.1% for Cr to +11.2% for Pb), and for SRM 2711 –0.5% (–10.2% for Cr to +9.7% for Fe).

Only measurement precision could be evaluated for the dilute HCl digests because there are no CRMs for this leach. Five replicates of GSD-7 were used to assess precision for both partial digestions, and the results were satisfactory (Table 2), but those from the hot HCl procedure were generally more precise. The median for the hot HCl procedure was 1.1% (range from 0.8% for Mg to 3.2% for Co), and for the cold HCl the median was 5.6% (range from 0% for Co to 9.3% for V).

Table 1
Precision and accuracy of the total digestion procedure applied to two certified reference materials (five replicates)

Element	GSD-7 Precision (%)[a]	GSD-7 Accuracy (%)[b]	SRM 2711 Precision (%)	SRM 2711 Accuracy (%)
Al	1.35	–2.27	1.14	–1.62
Ba	1.10	0.75	1.70	0.14
Ca	1.60	2.50	1.47	0.76
Co	2.56	2.88	5.71	–4.00
Cr	1.32	–6.07	2.60	–10.2
Cu	4.42	–2.11	4.91	–1.05
Fe	3.27	3.76	1.96	9.69
K	1.25	0.17	1.01	0.33
Mg	1.95	–0.72	1.47	–1.52
Mn	1.36	5.74	0.99	3.51
Na	1.69	–1.48	0.82	5.35
Ni	1.46	7.92	2.79	–4.85
P	1.79	3.17	2.35	–1.16
Pb	1.96	11.2	1.66	6.99
Sr	1.15	2.18	0.77	–0.53
V	1.61	6.04	3.12	6.13
Zn	2.64	–4.29	2.57	–7.65

[a] Precision is estimated by the coefficient of variation $CV=[SD/\bar{X}]\times 100$

[b] Accuracy (bias) is estimated using $\left[\frac{\bar{X}_{\text{ThisStudy}}-\bar{X}_{\text{Certified}}}{\bar{X}_{\text{Certified}}}\right]\times 100$

Digestion assessment criteria

Criteria need to be established to assess the superiority of a given partial digestion procedure. Given the geo-environmental focus of this study, the following will be used to guide the final decision, with the most appropriate single digestion procedure producing (1) limited breakdown of the aluminosilicate lattice and release of refractory-associated elements, quantification will be assessed by Al, Cr, and Fe recoveries [recovery = (partial digestion/total digestion) ×100]; (2) high recoveries for suspected anthropogenic contaminants (e.g., Cu, Pb, and Zn), but not quantitative; and (3) recovery of contaminants should be independent of physical and chemical controlling factors such as clay content, organic C content, carbonate content, and CEC.

Results

The texture of the road sediments was dominated by sand (64%), with lesser amounts of silt (30%) and clay (6%). The median pH was 7.6, with a range from 6.8 to 9.5. Organic C and $CaCO_3$ contents were high with median values of 10 and 9% respectively. Cation exchange capacities were generally high with a median of 31 $\text{cmol}_c\ \text{kg}^{-1}$, with a wide range from 7 to 77 $\text{cmol}_c\ \text{kg}^{-1}$.

Element concentrations

A ranked logarithmic dot plot (Cleveland 1985) was used to display the median road sediment concentrations for 17 elements for a total digestion and two partial digestions (Fig. 2). Concentrations ranged over four orders of

Table 2
Precision of hot and cold 0.5-M HCl treatments of certified reference material GSD-7 (five replicates) and median concentrations liberated by each treatment

Element	Concentration liberated hot HCl (mg/kg)	Hot HCl precision (%)	Concentration liberated cold HCl (mg/kg)	Cold HCl precision (%)
Al	14,940	0.76	5,440	4.43
Ba	189	1.83	102	3.40
Ca	4,120	1.09	2,820	5.83
Co	14	3.24	6	0.00
Cr	44	1.89	8	5.45
Cu	24	2.24	6	8.56
Fe	22,100	0.96	5,140	5.77
K	4,480	1.00	1,800	5.56
Mg	9,400	0.75	2,400	5.89
Mn	569	0.77	329	4.98
Na	442	1.01	256	5.24
Ni	30	1.85	8	5.73
P	534	1.67	440	2.78
Pb	336	1.06	225	5.46
Sr	48	0.94	34	6.52
V	39	2.16	10	9.32
Zn	218	1.23	72	6.36

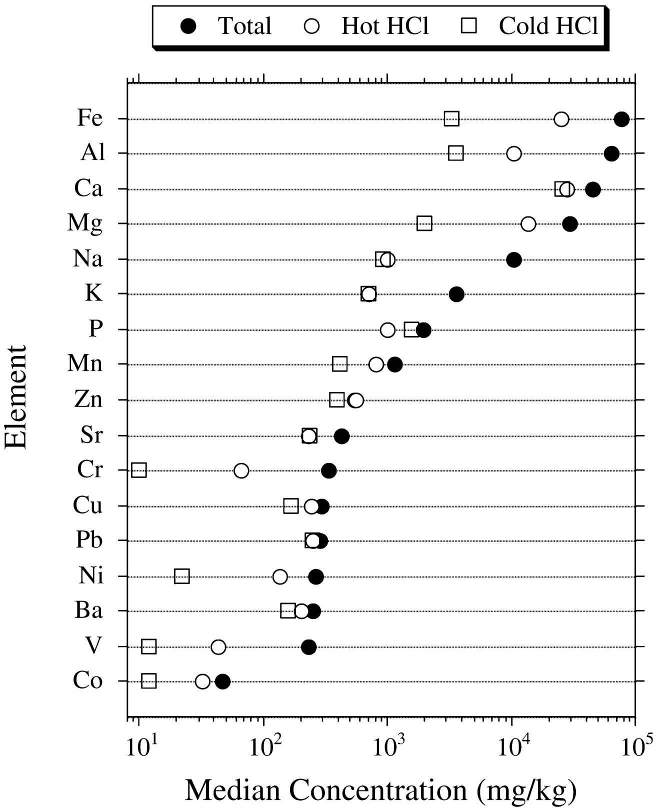


Fig. 2
Dot plot of the median element concentrations of 19 road sediments liberated by a total digestion, a 0.5-M hot HCl treatment, and a 0.5-M cold HCl treatment

magnitude, from 10±6 mg/kg (median ±95% confidence interval) for Cr from a cold HCl digestion ($\mathrm{Cr_{Cold}}$) to 77±0.5 g/kg for Fe from a total digestion ($\mathrm{Fe_{Tot}}$). Note the ordering of the elements on the *y*-axis in Fig. 2 is based on median total digestion data, and the same sequence is not followed by either the hot or cold HCl leaches.

Both partial HCl digestions removed significantly lower concentrations of Al from the RDS than a total digestion (Fig. 3), with the $\mathrm{Al_{Hot}}$ significantly greater than $\mathrm{Al_{Cold}}$ (Fig. 4). On average, the hot HCl procedure liberated 6.3±1.2 g/kg more Al than the cold HCl procedure. Phosphorus and Sr were the only elements liberated in greater amounts by the cold HCl procedure (Fig. 2).

The close association of Pb released by the partial digestions and the total digestion procedure is shown in Fig. 5. The non-parametric Spearman correlation coefficient (r_s) for Pb and other elements are shown in Table 3. The r_s value for $\mathrm{Pb_{Tot}}$ vs. $\mathrm{Pb_{Hot}}$=0.98, between $\mathrm{Pb_{Tot}}$ and $\mathrm{Pb_{Cold}}$=0.99, and between $\mathrm{Pb_{Hot}}$ and $\mathrm{Pb_{Cold}}$=0.99 (see Fig. 6a). The median difference between the 19 RDS samples for Pb extracted by hot and cold HCl was 14±18 mg/kg (Fig. 6b), with a range from –26 mg/kg (cold >hot) to +344 mg/kg (hot >cold).

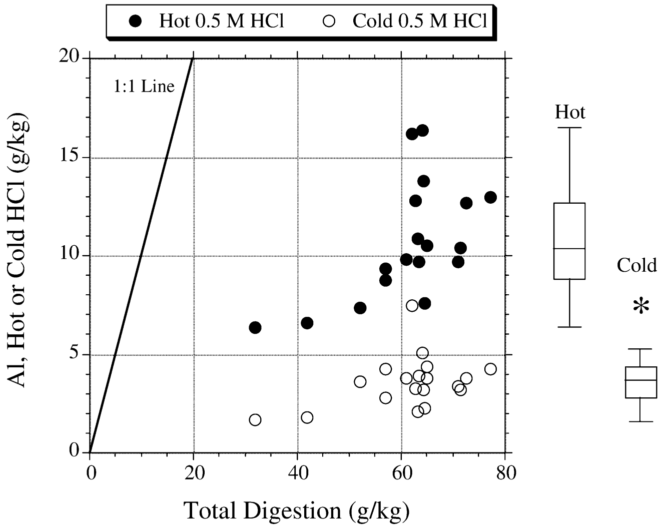


Fig. 3
Scatter plot of Al liberation from 19 road sediments with hot and cold HCl leaches relative to a total digestion. Box plots are appended to allow visual comparison between overall hot and cold HCl concentrations. *Central boxes* represent 50% of the data distribution, the *central line* in the box is the median. An *asterisk* is shown when a sample is considered an "extreme" outlier by an empirical rule

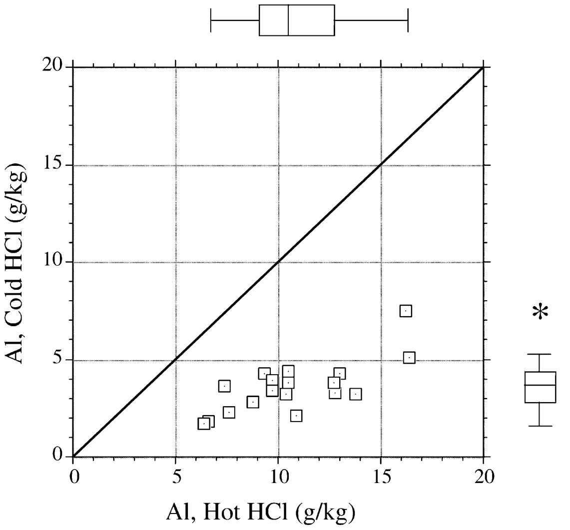


Fig. 4
Scatter plot of Al concentrations for 19 road sediments for cold and hot 0.5 M HCl treatments. Equivalence is depicted by the *solid 1:1 line*, with *values below the line* indicating hot >cold

Recoveries

Median recoveries and 95% confidence intervals, ranked from low to high based on a hot HCl digestion, are shown in a dot plot (Fig. 7a). The recoveries of elements with a cold HCl leach were significantly different for most elements. The five highest recoveries for the hot HCl procedure were Zn >Cu >Pb >Mn >Ba, whereas for the cold HCl procedure Pb >P >Zn >Ba >Ca. The absolute difference in recoveries (hot–cold) are shown in Fig. 7b. Three elements, Co, Mg, and Ni, had absolute hot HCl recoveries ≥40% higher than from a cold procedure. As noted for concentrations, only Sr and P recoveries were statistically (α=0.01) greater for a cold procedure, with median differences of 4±2 and 28±5%, respectively. The elements with the least difference in recoveries were Na (1±0.4%) and K (3±2%).

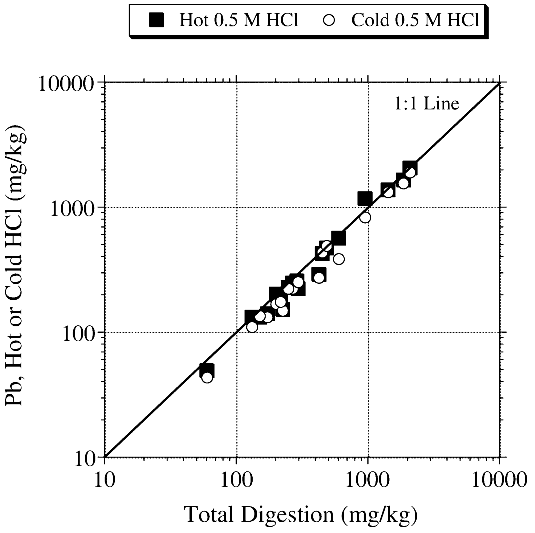


Fig. 5
Scatter plot of Pb liberation from 19 road sediments with hot and cold HCl leaches relative to a total digestion. Box plots are appended to allow visual comparison between overall hot and cold HCl concentrations. *Open circles* associated with box plots reflect "outliers" flagged by an empirical rule, whereas *asterisks* reflect "extreme" outliers flagged by an empirical rule

Table 3
Summary of Spearman correlation coefficients (r_s) and associated *P*-values for concentration comparisons between different digestion procedures for 17 elements from 19 road-deposited sediments

Element	Total vs. hot HCl r_s (*P*-value)	Total vs. Cold HCl r_s (*P*-value)	Hot vs. cold HCl r_s (*P*-value)
Al	0.53 (0.025)	0.33 (0.16)	0.57 (0.016)
Ba	0.93 (<0.0001)	0.96 (<0.0001)	0.98 (<0.0001)
Ca	0.79 (0.0008)	0.83 (0.0005)	0.96 (<0.0001)
Co	0.87 (0.0002)	0.81 (0.0006)	0.88 (0.0002)
Cr	0.14 (0.56)	0.33 (0.17)	0.40 (0.095)
Cu	0.95 (<0.0001)	0.96 (<0.0001)	0.96 (<0.0001)
Fe	0.13 (0.59)	0.61 (0.009)	−0.01 (0.95)
K	0.26 (0.27)	0.29 (0.22)	0.94 (<0.0001)
Mg	0.58 (0.013)	−0.04 (0.85)	0.11 (0.65)
Mn	0.87 (0.0002)	0.84 (0.0004)	0.97 (<0.0001)
Na	0.42 (0.077)	0.42 (0.078)	0.95 (<0.0001)
Ni	0.90 (0.0001)	0.67 (0.0046)	0.64 (0.0071)
P	0.73 (0.0020)	0.81 (0.0006)	0.81 (0.0006)
Pb	0.98 (<0.0001)	0.99 (<0.0001)	0.99 (<0.0001)
Sr	0.70 (0.0031)	0.76 (0.0013)	0.95 (<0.0001)
V	0.44 (0.063)	0.38 (0.12)	0.56 (0.018)
Zn	0.97 (<0.0001)	0.97 (<0.0001)	0.98 (<0.0001)

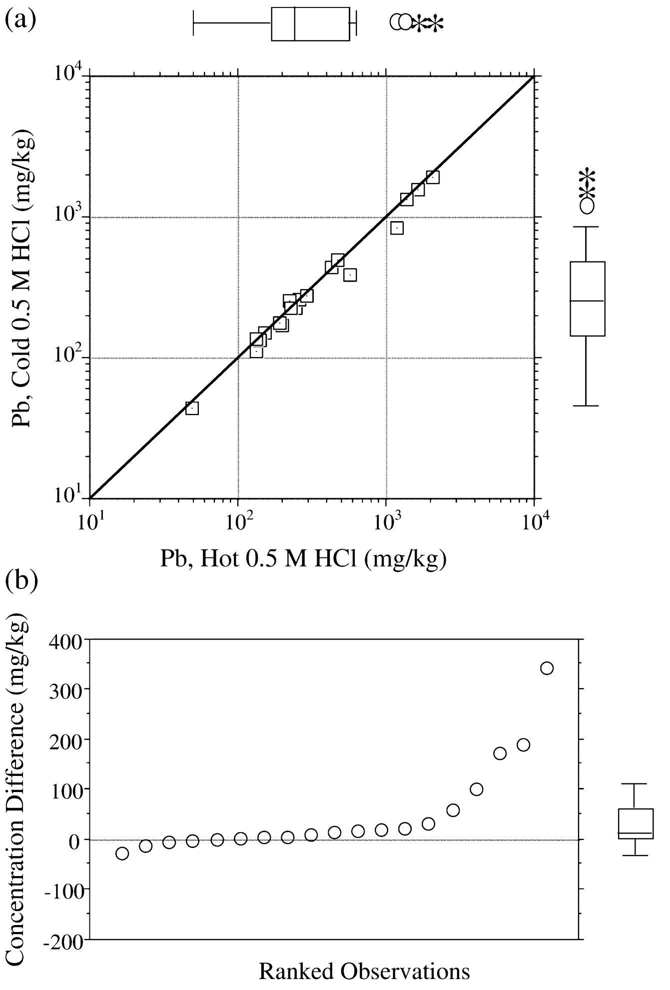


Fig. 6
a Scatter plot of Pb concentrations for 19 road sediments for cold and hot 0.5 M HCl treatments. Equivalence is depicted by the *solid 1:1 line*, with *values below the line* indicating hot >cold. **b** Ranked absolute Pb concentration difference plot for a hot 0.5 M HCl leach minus a cold 0.5 M HCl leach for 19 road-deposited sediments. Positive values reflect greater Pb concentrations liberated by a hot leach

Recoveries of the aluminosilicate and refractory-associated elements Al, Cr, and Fe are plotted for individual RDS samples for both partial digestions in Fig. 8a–c. Median values in the dot plots are shown as vertically dashed lines. Median removals for the cold leach follow the order of Al (5.4±0.6%) >Fe (4.3±0.5%) >Cr (3.2±1.5%). For the hot HCl leach, the recovery order was Fe (29.2±2.9%) >Cr (19.3±3.9) >Al (16.2±1.5%). The median absolute recovery differences (hot–cold) between RDS samples were ~12% for Al, ~16% for Cr, and ~26% for Fe.

In addition to a limited breakdown of the structural lattice, a partial digestion should also exhibit high recoveries of contaminants. In a neighboring watershed, Sutherland and Tolosa (2000) found Pb and Zn in RDS to have significant anthropogenic signals and Cu to have a moderate signal. Concentrations of Cu, Pb, and Zn in the present study exceed those observed by Sutherland and Tolosa (2000). Thus, it is inferred that these elements reflect anthropogenic inputs.

Copper, Pb, and Zn recoveries are shown in Fig. 9a–c ranked from low to high according to the hot HCl leach. Dashed vertical lines are medians, and a solid vertical line is drawn at a recovery of 100% for reference, i.e., partial digestion = total digestion. Median recoveries for a hot HCl leach were highest for Zn (108±9%) >Cu (94±15%) >Pb (93±6%). The ordering for the cold HCl leach was Pb (86±5%) >Zn (70±7%) >Cu (55±16%). The paired Wilcoxon test indicated recoveries for the three contaminants were significantly greater for the hot HCl procedure (α=0.01).

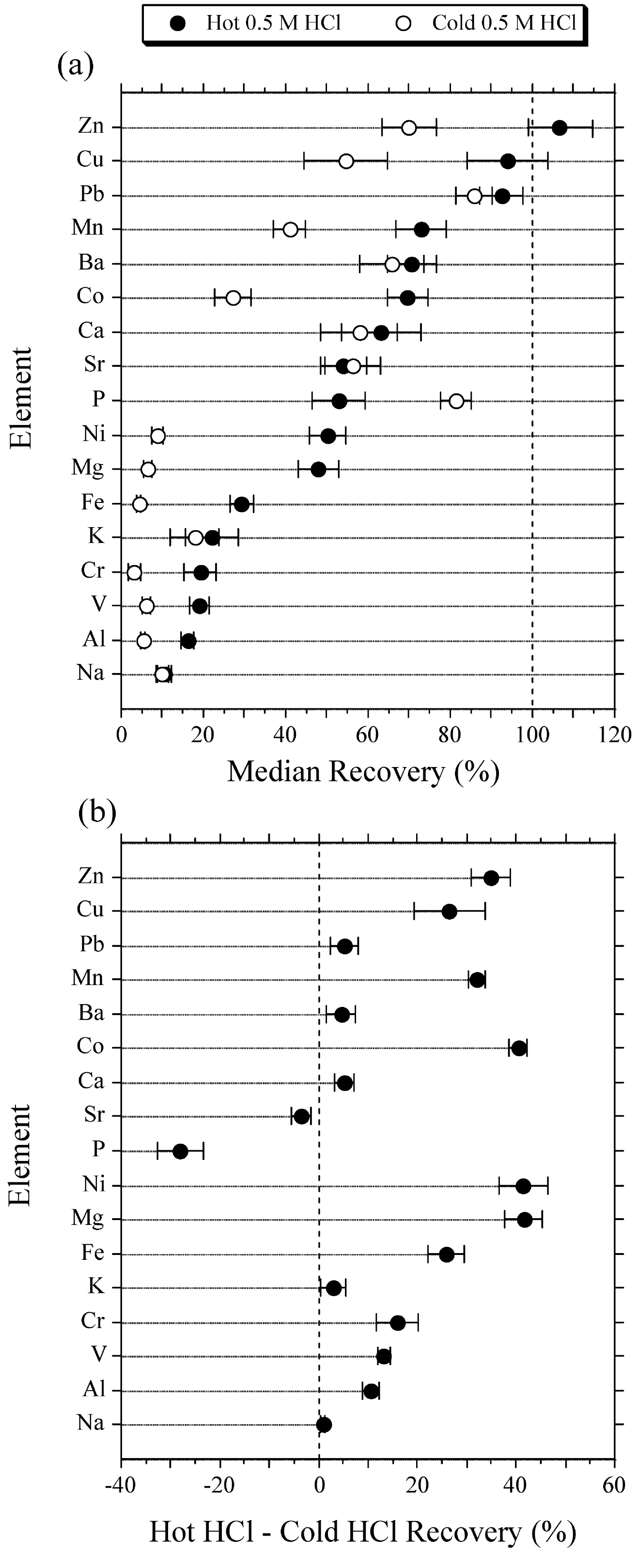


Fig. 7
a Dot plot of median element recoveries for 19-road sediments for 0.5 M HCl hot and cold leaches. The *error bars* associated with each element are 95% confidence bands of the median. **b** Dot plot of absolute median differences (and 95% confidence bands) for hot minus cold 0.5 M HCl recovery

To test whether each of the three elements for a particular digestion differed significantly from 100%, i.e., quantitative recovery, a non-parametric one-sample sign test was used (two-tailed test). The cold HCl procedure was not quantitative for any of the three elements. For the hot HCl leach, Cu and Zn were quantitative. For Cu_{Hot}, 6 of 19 RDS samples had recoveries >100% (Fig. 9a), and for 13 samples, Zn recovery was greater than a total digestion (Fig. 9c). Lead recovery was not quantitative using the hot HCl leach. Quantitative removal of Cu and Zn for a hot HCl leach means that this procedure, on average, digests all residual phases and liberated those that were not environmentally meaningful.

Physical and chemical factor controls

For an extraction protocol to have wide environmental applicability the concentrations of known or suspected contaminants should be independent of variations in physical and chemical factors of the media sampled. For Cu, Pb, and Zn recoveries there were no statistically significant Spearman correlations (α=0.01) with clay content, carbonates, organic C, or CEC for either of the partial extractions. Both dilute HCl extractions are judged equivalent for this criterion.

Discussion

Based on the criteria established for this study, the cold 0.5 M HCl partial digestion is considered to be superior to hot 0.5 M HCl. The cold HCl treatment had statistically lower recoveries for Al, Cr, and Fe, with median values of between 3 and 5% compared with 16 and 29% for the hot HCl treatment. If an arbitrary median threshold can be established for future work a value of about 15% may be given consideration, above which a partial digestion procedure can be considered to significantly degrade the silicate lattice and liberate refractory elements. Careful consideration must be given to this threshold as it would not be applicable to those elements that are associated with industrial contamination sources, particularly Cr and Fe. Thus, if one element is to be examined, preference should be given to Al.

Literature data for Al recoveries with a 0.5 M HCl cold leach were less than the proposed threshold. Duinker and others (1974) used stoichiometric formulae of clay minerals in suspended sediment and Al data and computed clay mineral lattice breakdown with 0.5 M HCl after 18 h of leaching was 9%. Agemian and Chau (1976, 1977) found Al recoveries of 8.5 and 9.3% after 16 h of shaking with cold 0.5 M HCl. Van Valin and Morse (1982) found a median Al recovery of 4.5% (range 2–5%) after 16 h shaking with 0.5 M HCl. These authors also found Al recoveries with cold 12 M HCl for the same sediment ranged from 17 to 32% (median =21%).

The addition of heat causes substantial, but variable, breakdown of the aluminosilicate lattice depending on mineralogy. Ray and others (1957) noted this early on

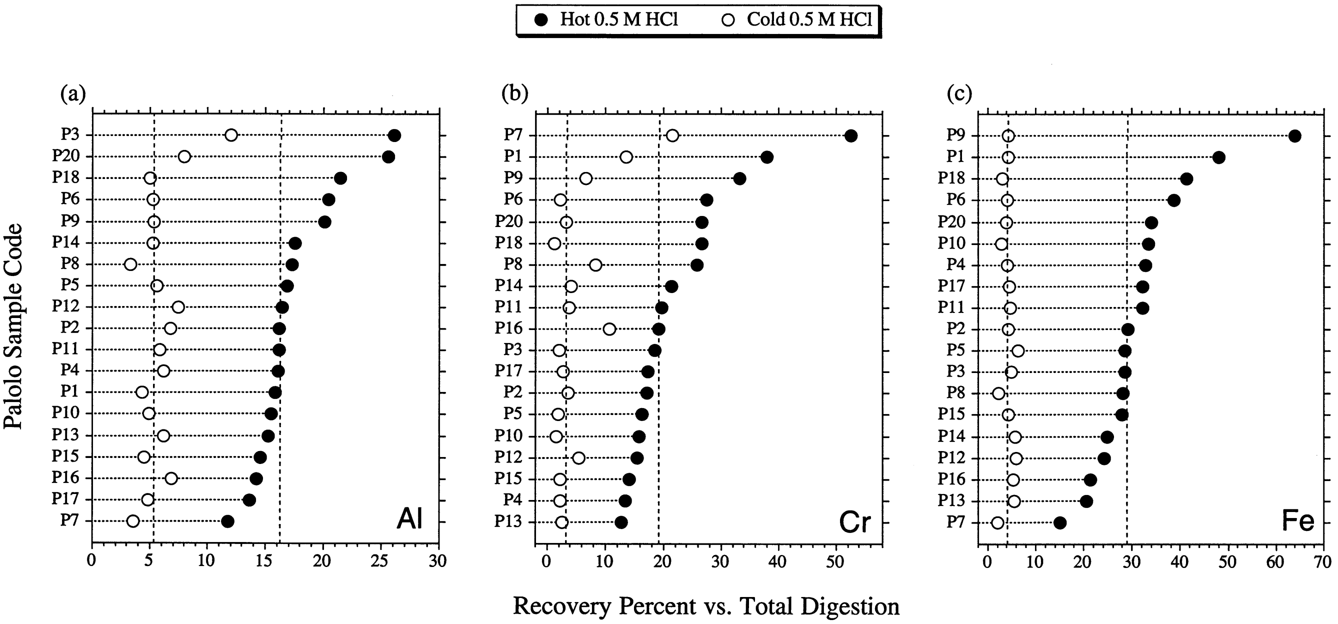
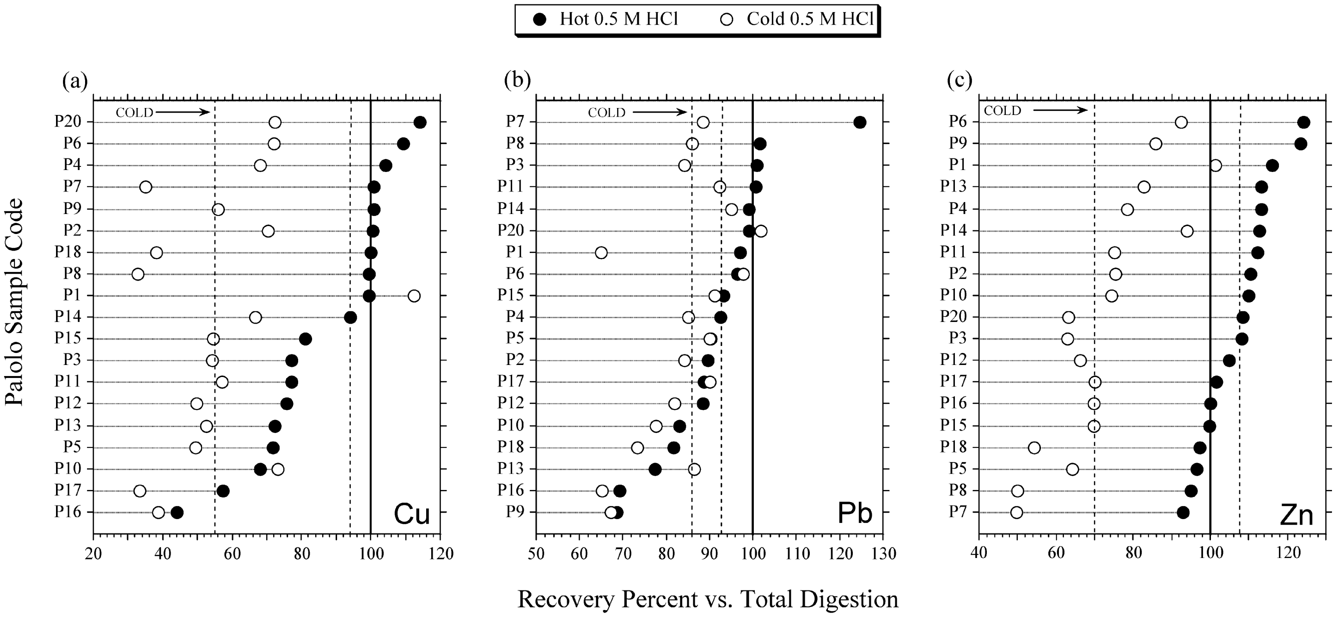


Fig. 8
a Individual Al, **b** Cr, and **c** Fe recoveries for 19 road sediments for hot and cold 0.5 M HCl treatments. *Dashed vertical lines* reflect overall medians for each treatment

when a synthetic mix of 2.5% quartz, 2.5 hectorite (Mg end member of the montmorillonite family) and 95% calcite was leached with 0.2 M HCl at room temperature or heated (80–85 °C) for 4 h. After the cold HCl treatment, the X-ray powder pattern showed no breakdown as a strong hectorite line was visible. However, by adding heat only a very faint hectorite line remained, indicating almost complete destruction. Snäll and Liljefors (2000) observed that the Al recovery from common silicate minerals, powdered to <20 μm, varied dramatically when leached with 1 M HCl (boiling) for 2 h. They found the following Al recoveries: biotite (107%) >Mg-chlorite (101%) >Fe-chlorite (65%) >hornblende (31%) >microline (8%) >muscovite (7%). These data and those from the present study indicate that if an investigator requires environmentally meaningful element data, heating of dilute HCl should be avoided. Recoveries for suspected contaminants (Cu, Pb, and Zn) were significantly higher for the hot HCl leach, especially for Cu and Zn. The hot HCl treatment released quantitative amounts of Cu and Zn when compared with a total digestion. This is important because this procedure would release both labile and residually bound fractions of Cu and Zn. Because the goal of many partial digestion

Fig. 9
a Individual Cu, **b** Pb, and **c** Zn recoveries for 19 road sediments for hot and cold 0.5 M HCl treatments. *Dashed vertical lines* reflect overall medians for each treatment. *Solid vertical line* reflects a recovery of 100%, i.e., partial digestion = total digestion



procedures is to release labile fractions, hot 0.5 M HCl would be less suitable than a cold leach for these two elements. Though Pb recovery was statistically higher for a hot HCl treatment compared with the cold treatment, the absolute median difference was only ~8%.

Conclusion

Single leach partial digestion procedures with dilute inorganic acids are potentially important for characterizing element bioavailability, lability, mobility, anthropogenic contamination, and anomaly assessment. Hydrochloric acid was selected for detailed examination of road-deposited sediments with a 0.5 M HCl solution applied cold (shaken for 1 h) and hot (90–95 °C for 1 h) with concentrations compared with a total digestion for 19 samples. Based on defined criteria, the cold 0.5 M HCl treatment was found to be superior. Median recoveries for Al, Cr, and Fe were =5% with cold HCl, compared with 16–29% for hot HCl. Thus, significant aluminosilicate matrix breakdown or release of refractory-associated fractions resulted with the application of heat. Though recoveries for the three suspected contaminants, Cu, Pb, and Zn, were higher for the hot HCl leach, this digestion was found to be quantitative for Cu and Zn, i.e., release of both labile and residual fractions.

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