Enhancing Geochemical Precision by Analyzing the Clay Fraction in Till: Cost-Benefit Study from the TREK Project Area, British Columbia

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Abstract

This study presents new data obtained from the clay-sized fraction (< 2 µm) of previously collected basal till material from the TREK Project area. A total of 1,222 samples were analyzed by aqua-regia digest and ICP-MS finish, following a wet clay-separation process. Data-quality assessment assures reliable and reproducible results for most of the reported suite of 53 elements. The high-quality clay-fraction data are a valuable addition to the already available data for the TREK Project and can be readily integrated with other regional geoscientific information. Exploratory data analysis is used to compare the clay-fraction geochemistry to that of the previously acquired coarser fraction (< 63 µm). Higher trace-element contents ensure improved data precision and a more homogenous mineralogical composition leads to enhanced anomaly-to-background contrast, relative to the coarser size fraction. Particularly, trace elements such as Au, Hg, and Tl show significant improvements in precision, leading to better maps and greater confidence in the interpretation of spatial patterns and relationships. Large-scale features such as the Tatuk and Nechako Fault zones are recognized in trace-element distributions, as well as multi-element anomalies related to known mineralization.

Introduction

Geoscience BC's TREK (Targeting Resources for Exploration and Knowledge) Project is focused on British Columbia's northern Interior Plateau region, which includes the Blackwater gold district and is considered highly prospective for mineral resources that are known to occur throughout the Stikine terrane (Clifford & Hart, 2014). Because of its thick overburden, the area is largely underexplored and its prospective bedrock geology is difficult to map. Large-scale geophysical surveys and regional mapping are generally beyond the scope and capacity of individual exploration projects, and this is where the data generated through Geoscience BC can add the most value. In addition to large-scale airborne geophysical surveys and geological mapping, the TREK Project included an extensive till sampling program to provide regional-scale surficial geochemical data (Jackaman & Sacco, 2014; Sacco *et al*., 2014; Sacco & Jackaman, 2015) (Fig. 1). The size fraction that was selected for the till geochemistry in the TREK Project was < 63 µm (230 mesh). Although this is standard practice in till analysis because dry sieving to finer fraction would be impractical, the geochemical response may still be overwhelmed by matrix components such as carbonate minerals and organic matter. To remedy the effect of matrix materials on the analysis of till and reduce its heterogeneity, the clay-sized fraction (< 2 µm) can be extracted and analyzed, which has been known to provide superior results for over several decades (Nichol & Bjorklund, 1973; Slavek & Pickering, 1981; DiLabio, 1995; van Geffen *et al*., 2012), albeit at greater cost per sample.

Ferbey *et al*. (2009) reported the reanalysis of the clay fraction of archived till samples from the Babine Porphyry Belt, which were sampled in the earlier NATMAP sampling program (2002). The BCGS Open File 2009-4 (Geoscience BC Report 2009-10) describes the relative abundances of a range of elements but does not proceed with interpretation, such as comparing the clay-fraction data to the original coarse-fraction results. Sacco *et al.* (2014) summarized all previous geochemical campaigns that generated public data from areas within the TREK Project area, and we refer to their work as it provides most of the context required for further interpretation of the data presented here.

The purpose of this study is to establish whether the extra expense of extracting the clay fraction from tills preceding chemical analysis is justifiable, given the expected improvement in the precision of assay results, increased trace-element contents, and enhanced anomaly-to-background contrast. Basic exploratory data analysis, as well as data-quality assurance techniques are used to accomplish this objective. The geochemistry of the two size fractions is compared, in both data space and geographic space. Trace-element anomalies are correlated with known mineral deposits and major structures mapped in the area (Angen *et al*., 2017).

Geology and Surface Materials

Although this study did not involve additional fieldwork, the geological setting is summarized here to provide geospatial context to the obtained results. The processes involved in the distribution of elements in surface sampling media are controlled by the structural geologic framework that forms the principal conduits for fluid transport, and the fluid composition is modified by the transected geology. The ultimate till geochemistry is thus a function of the provenance rock composition, modified by weathering and erosional processes, and the influx of hydromorphic species from groundwater and formation water.

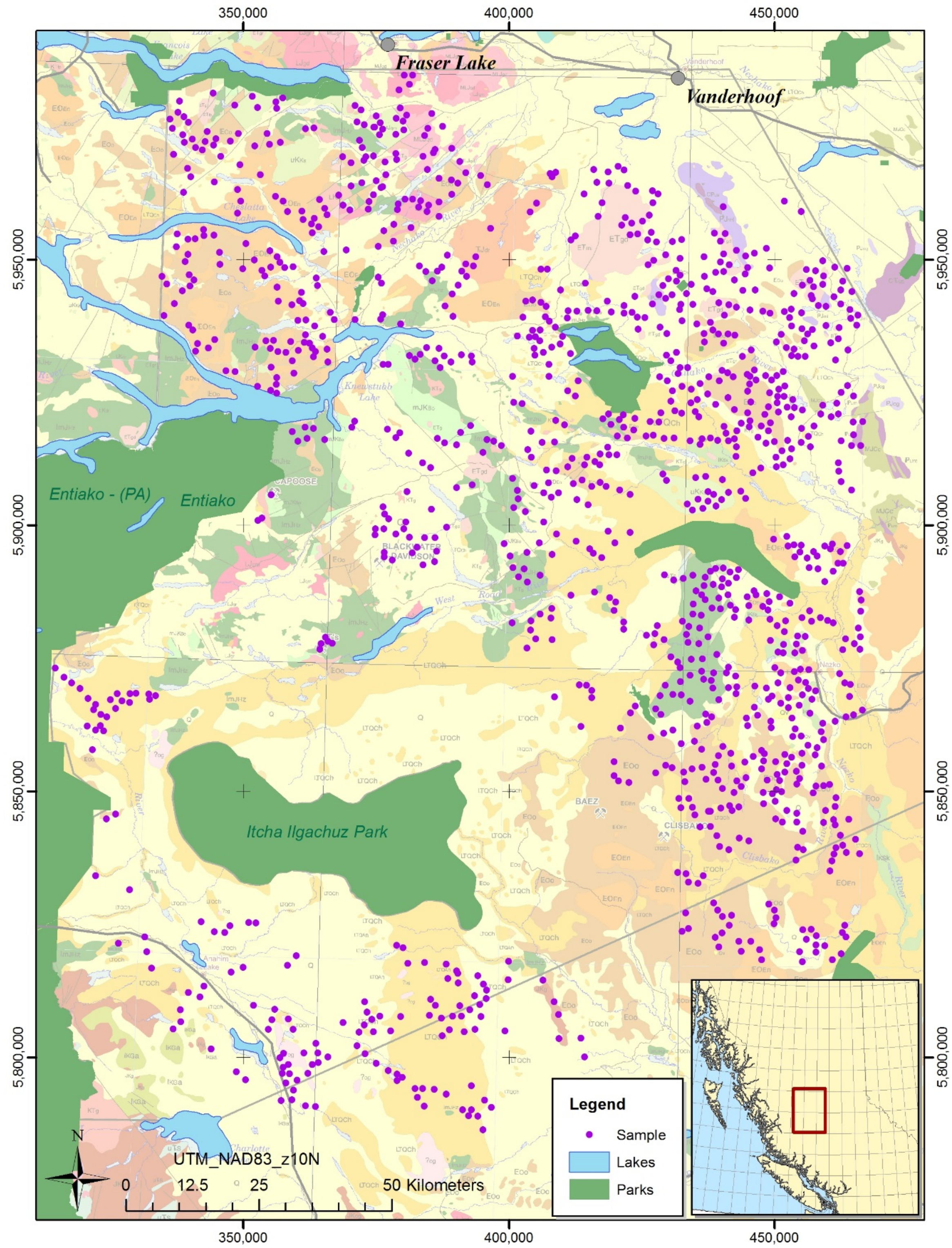

Figure 1: TREK Project 2013 and 2014 till sample locations, Central British Columbia, superimposed on 2014 Geoscience BC geological map of the TREK Project area (after Massey *et al*., 2005).

Much of the prospective Jurassic bedrock of the Stikine Terrane in the central part of British Columbia's Interior Plateau is obscured by Miocene to Pleistocene volcanic rocks and extensive deposits of glacial sediments (Jackaman & Sacco, 2014; Angen *et al.*, 2015). Some major WNW thrust faults run oblique to the dominant NNW fabric of the Western Cordillera, including the Tatuk and Tasa Fault zones, with sinistral offsets along NE-trending structures such as the Natalkuz and Hallett Lake Faults (Angen *et al.*, 2017). Younger volcanic rocks of the Chilcotin and Anahim Groups form a significant obstacle to the direct geochemical detection of underlying bedrock and its mineral potential.

Most of the overlying glacial sediments are classified as till, with two dominant variations: basal till, the closest proxy to eroded bedrock with minimal glacial transport, and ablation till, deposited from the glacial load during stages of melting and glacial retreat (Sacco *et al*., 2014). Basal till, as the primary target of the 2013 and 2014 sampling campaigns, is characterized as a "dense, over-consolidated, matrix-supported diamict", conforming to the underlying topography and reaching thicknesses of up to tens of metres (Jackaman & Sacco, 2014). The moderate transport distances of basal till give this sample medium the potential to enlarge the surface footprint of geochemical anomalies, providing easier targets for regional exploration. The dominant ice-flow direction of the Fraser Glaciation that deposited most of the till across the northern part of the TREK Project area, was generally towards the east to northeast (Ferbey *et al.*, 2013; Sacco *et al*., 2014).

Methods

Basal till samples that were collected over the TREK Project area during 2013 and 2014 were archived by the British Columbia Geological Survey (BCGS) in Victoria, BC (Jackaman & Sacco, 2014; Sacco *et al.*, 2014; Sacco & Jackaman, 2015). For this study, a total of 1,222 'character splits' of the archived samples were retrieved for clay extraction and analysis, with help from Ray Lett and Wayne Jackaman. These samples were submitted to Bureau Veritas Mineral Laboratories' facility in Vancouver for preparation and analysis (BV Minerals, 2017). The analytical method is a clay-fraction separation by suspension in deionized water and high-speed centrifuging (CLYSP), followed by a modified aqua-regia digest and ICP-MS finish for 53 elements on 0.5 g aliquots (AQ250-EXT).

Previous geochemical campaigns run by the Geological Survey or Canada (GSC) and BCGS within the TREK Project area provided till samples that were recovered and submitted for reanalysis of the < 63 µm fraction at Bureau Veritas, as part of the TREK Project in 2015 (Jackaman *et al*., 2015). Unfortunately, the sample volumes remaining at the BCGS archives after reanalysis were considered insufficient for additional clay extraction. The GSC published till geochemical data from the area in 2001 (Plouffe *et al*., 2001), including ICP-AES data of both the < 63 µm and < 2 µm fractions. These studies are used for reference and, where appropriate, included in data analysis.

Results

Quality Assurance

The analytical results are of very good quality, and most analytes show excellent precision. Exceptions are B and Ta that are entirely below their analytical detection limits, as well as Ge, Pd, Pt, Re, S, and W, of which more than half the data are below detection. Other elements with relatively poor precision, either because of low abundances in the sample material or poor dissolution in the modified aqua-regia extraction, include Hf, In, Nb, Sn, Te, and U. The data distributions of the < 2 µm fraction analyses are presented in normal-probability plots (Fig. 2). The raw analytical data in CSV format and individual element maps in KML (Google Earth) format are available via the website of Geoscience BC's TREK Project: http://www.geosciencebc.com/s/TREK.asp.

Reference Materials

Under ideal circumstances, the accuracy of the analytical procedure would be tested and monitored through the inclusion of certified reference materials (CRM) of known composition and similar matrix to the samples, inserted randomly and blindly throughout the sample sequence submitted to the laboratory. However, as the analytical method involves only a partial digest (modified aqua regia), no reference materials could match the exact fraction of dissolution. Moreover, as the produced data are more sensitive to precision than accuracy, the exact contents of each analyte are less relevant. In other words, since we are looking for relative contents that may display relevant patterns over the sampled area, the precision is more important than the accuracy of the exact values. Four certified reference materials were included in the sample sequence after the clay extraction to measure the analytical consistency with time. Over the entire sequence, 18 aliquots of each CANMET reference material, TILL-1, TILL-2, TILL-3, and TILL-4, were inserted and analyzed. The results of seven analytes are presented in analytical order in line plots (Fig. 3). These CRM data serve as a monitor for instrument drift and analytical precision, rather than accuracy. No significant drift was observed for any of the measured elements and the few erratic numbers in the Au data are considered acceptable and to be expected at such low Au contents following an aqua-regia digest.

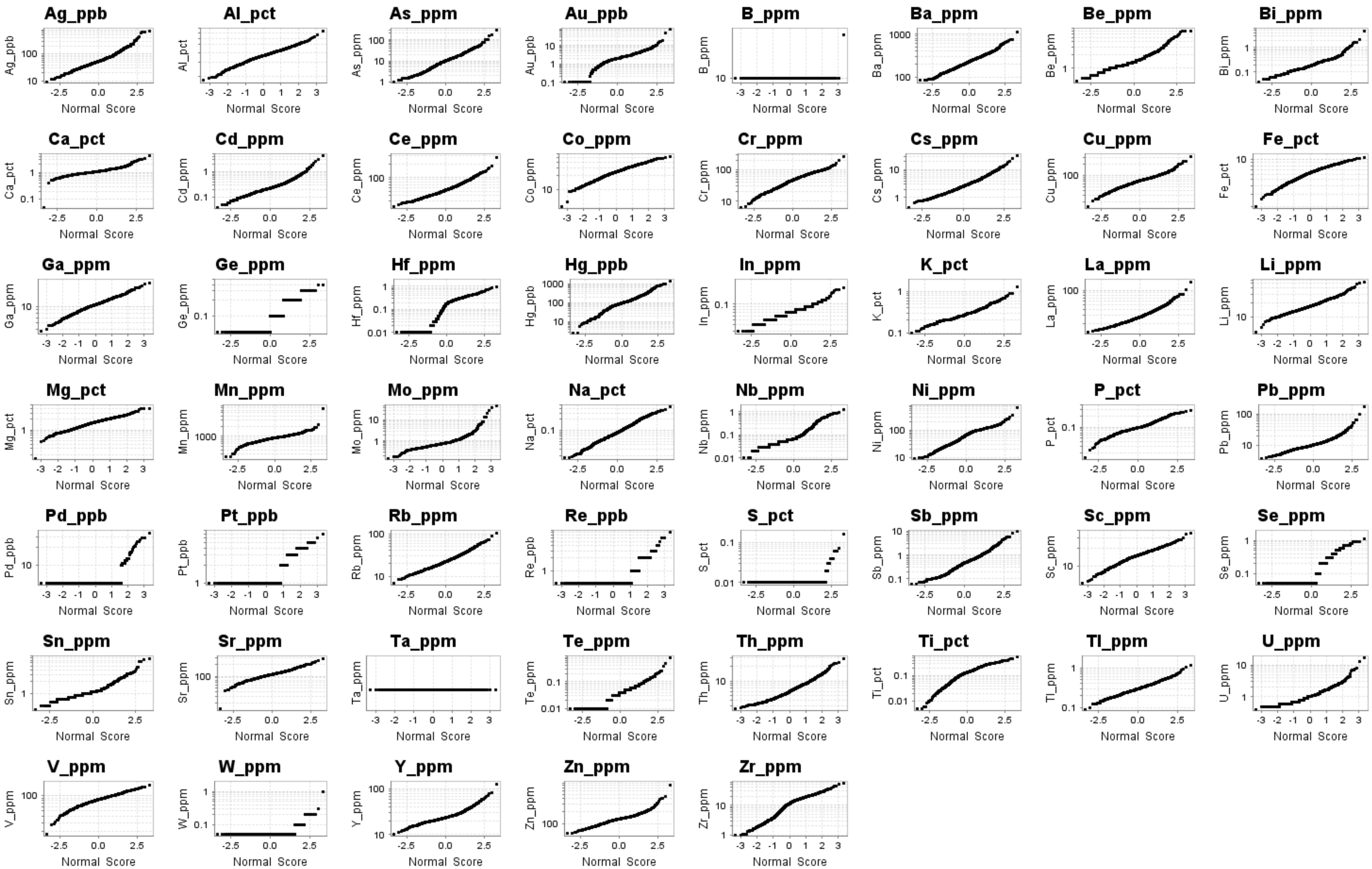

Figure 2: Normal-probability plots (logarithmic scale) for all 53 elements reported in the < 2 µm fraction of TREK tills, by aqua-regia digestion and ICP-MS analysis.

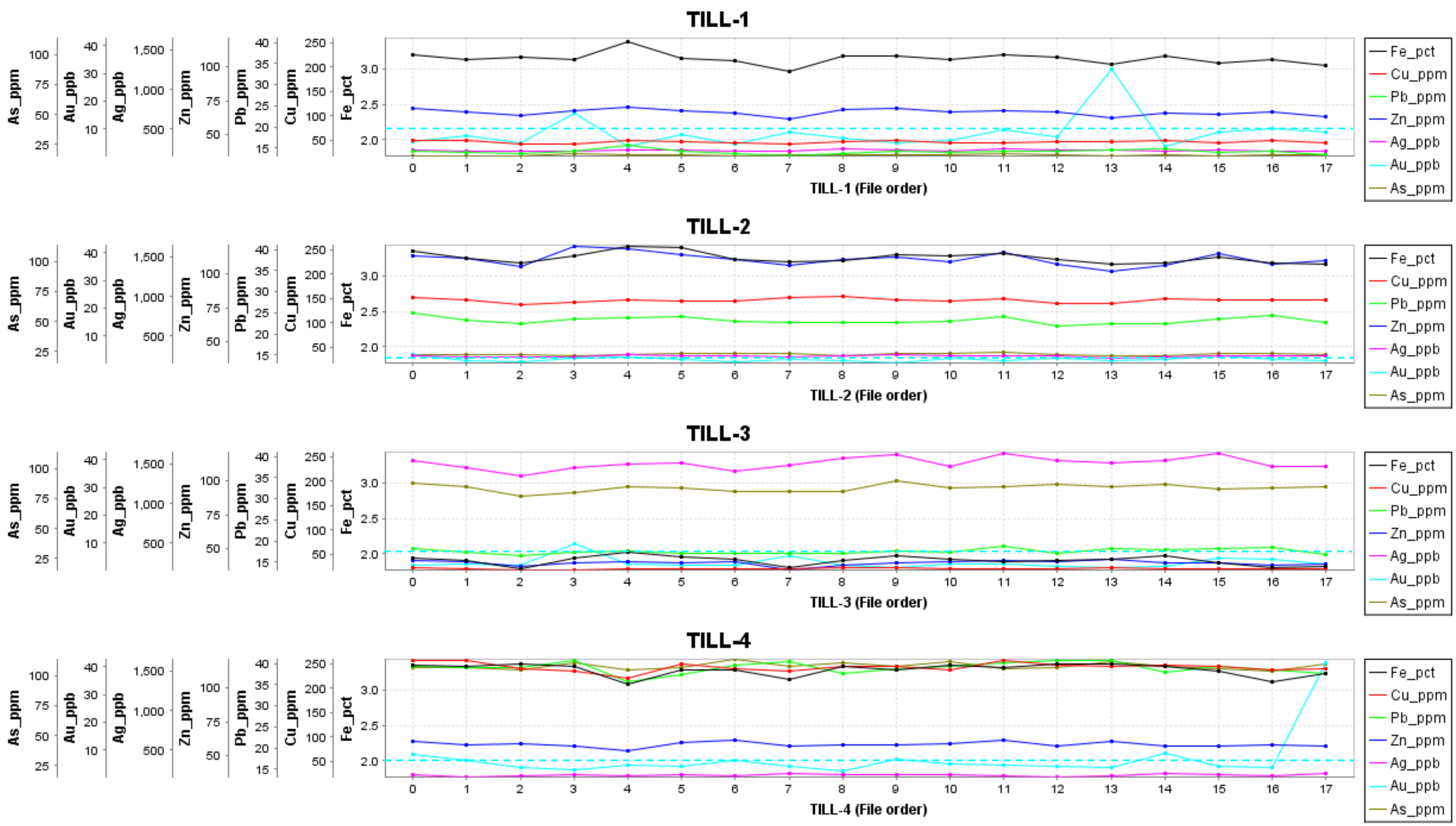

Figure 3: Element contents of Fe, Cu, Pb, Zn, Ag, Au, and As in 18 analyses of certified reference materials, TILL-1, TILL-2, TILL-3, and TILL-4, randomly inserted throughout the analytical sequence. Certified values for (total) Au content: 13 ppb, 2 ppb, 6 ppb, and 5 ppb, respectively (cyan dash).

Precision

To measure the precision of the sampling and analytical results, 64 pairs of "character splits", equivalent to field duplicates, recovered from the BCGS archives and Wayne Jackaman's sample storage, were analyzed. The results of these duplicate analyses are presented in x-y scatter plots of the 64 duplicate pairs (Fig. 4). The linear distributions along the y=x line indicate very good precision.

The precision, as a measure of reproducibility and repeatability of analytical results, is best expressed as the coefficient of variation (CV), which is equivalent to the relative standard deviation (RSD). The CV for each element was calculated using the root-mean-square deviation (RMSD) method and charted for comparison of individual analytes (Fig. 5). Lower CV values correspond with greater precision. Values below 10% are very good, considering that most commercial laboratories won't guarantee greater precision than that for lab duplicates, and up to 20% is generally considered acceptable for field duplicates in most surface geochemical surveys. The lesser precision of Zr can be ascribed to the incomplete dissolution of zircon in aqua regia, and that of Sb and Au to their very low abundances. The precision naturally decreases at lesser contents, and one definition of the analytical detection limit is the content at which CV = 100%.

Discussion

Comparison of Two Size Fractions

The < 2 µm and < 63 µm fractions of the TREK Project tills have fundamentally different mineralogical compositions. The proportion of "blocky" minerals such as carbonates, feldspars, quartz, and apatite is much smaller in the clay-sized fraction than in the coarser fraction, relative to "flaky" hydrated aluminosilicates such as clay minerals and micas (Liu & Gonzalez, 1999; van Geffen *et al.*, 2012). The < 2 µm fraction is also known to host a greater proportion of transition elements and other metals that occur as positively charged ions adsorbed onto negatively charged clay-mineral surfaces and organic compounds (Rose *et al.*, 1979; Reimann, 1998; van Geffen *et al.*, 2012). However, the main cause for greater element contents in the finer fraction is the increased surface area of the mineral grains, which facilitates more rapid breakdown and a greater proportion of the sample being dissolved than an equal amount of coarser material exposed to the same process.

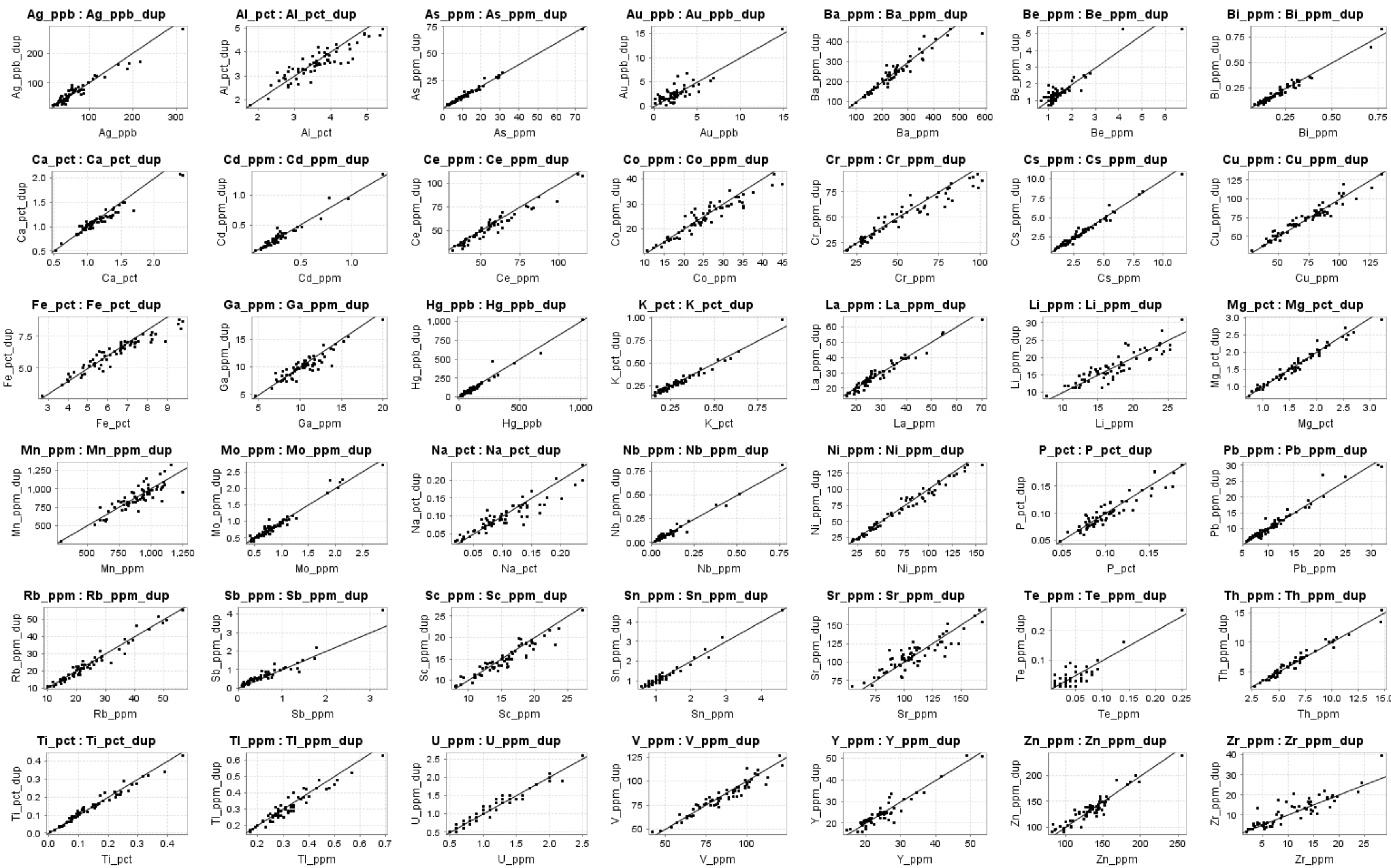

Figure 4: Scatter plots showing the analytical data of field-duplicate pairs in the < 2 µm-fraction. The y = x line is plotted for reference in each graph.

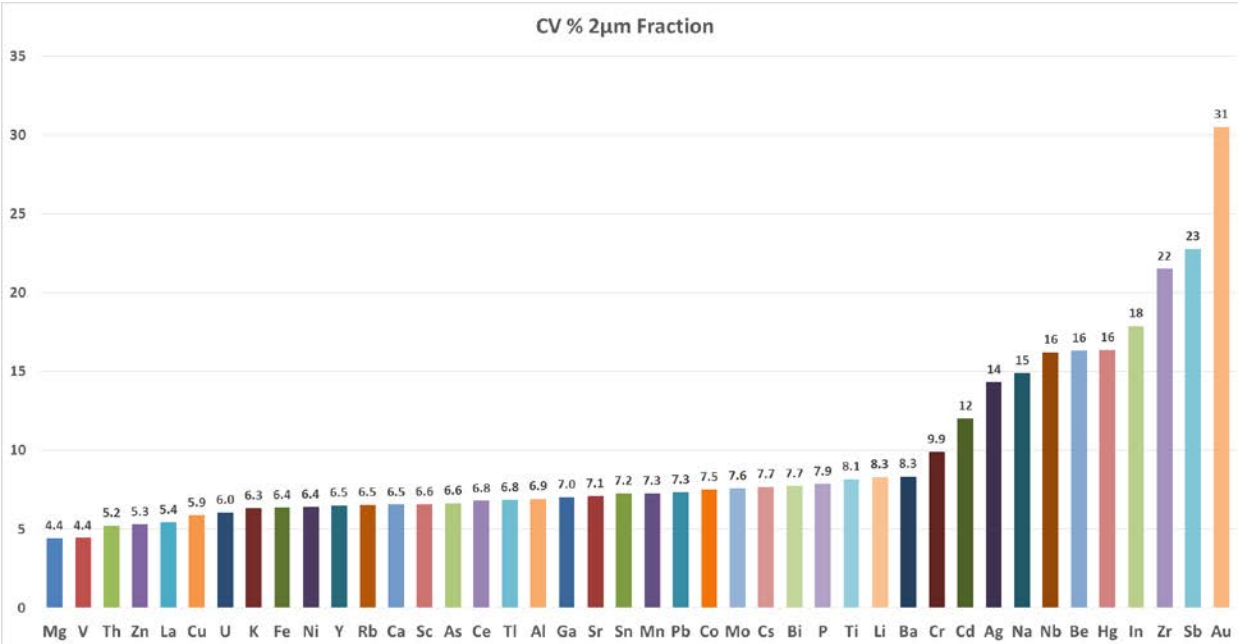

Figure 5: Field-duplicate precision of each element in the < 2 µm fraction, ordered by increasing CV %, or decreasing precision.

The data distributions of all measured elements provide a clear picture of the differences between the two fractions in split normal-probability plots (Fig. 6). For most elements that are above analytical detection levels, the clay-sized fraction produced much greater contents than the coarser fraction, providing a proportional improvement in precision. To assess the magnitude of the increased element contents in the clay-sized fraction, enhancement factors were calculated as ratios of the median values in the < 2 µm over the < 63 µm fraction (Table 1). These factors range between 0.9 and 3.3 and are mostly well above 1, except for Nb, P, and Ti, which are only marginally lower in the finer fraction, indicating their affinity with more stable mineral compounds. The greatest enhancements are observed in the volatile and chalcophile elements Hg, Tl, and Cu, as well as the principal clay-mineral constituents Al and K, with the alkali metals Rb, Cs, and Li substituting for K, and tri-valent Sc for Al, displaying similar preference for the clay-sized fraction.

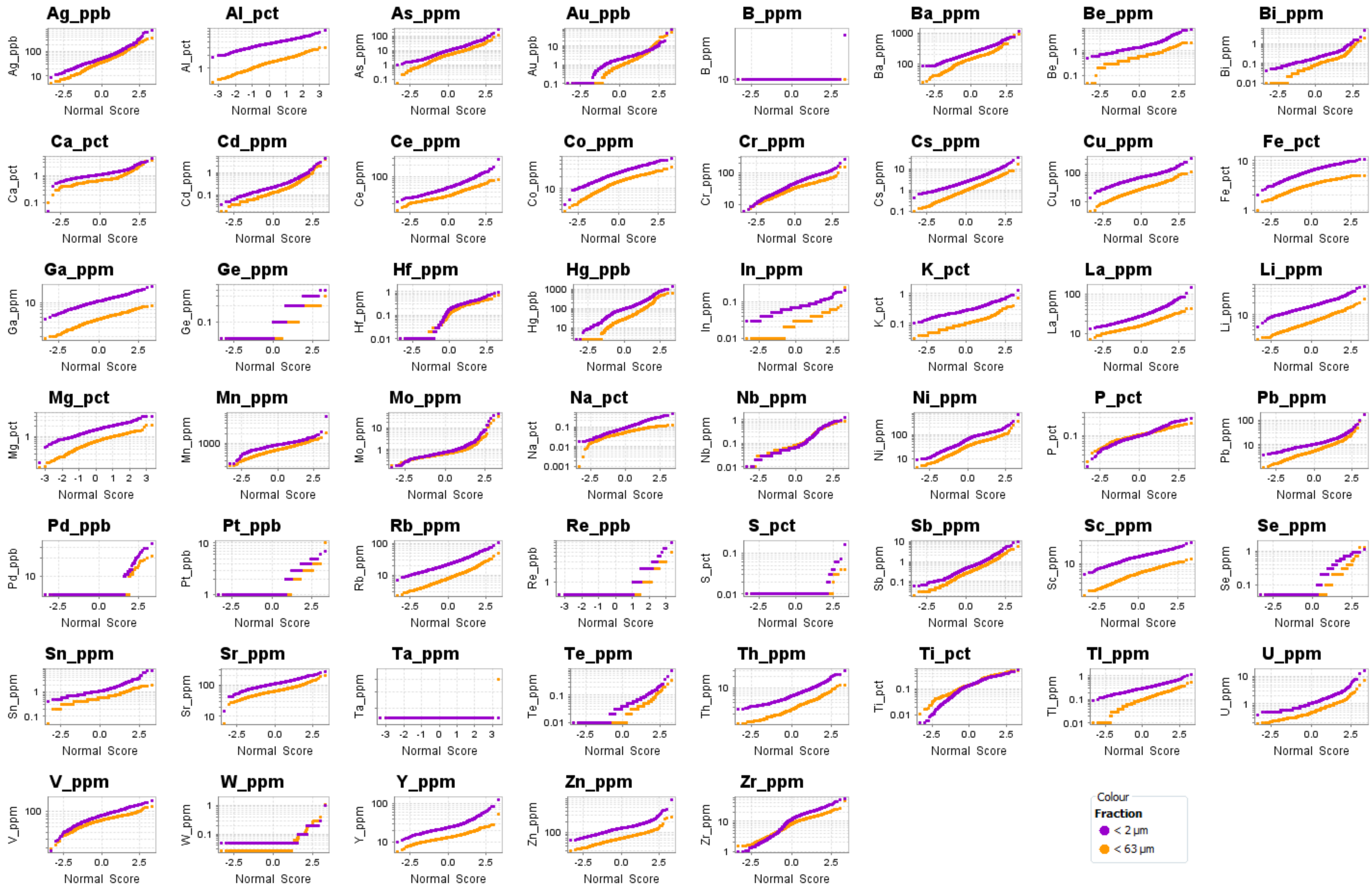

Figure 6: Split normal-probability plots with logarithmic y-axis of the < 2 µm (purple) and < 63 µm fraction (orange) data for all 53 measured elements, by aqua-regia digestion and ICP-MS analysis.

Table 1: Enhancement factors of the < 2 µm versus < 63 µm-fraction data, in decreasing order of median element-content ratios.

Element	Median < 63 µm	Median < 2 µm	% Difference	Factor
Hg_ppb	29	97	108	3.3
Tl_ppm	0.10	0.29	97	2.9
Rb_ppm	7.8	22	94	2.8
Sc_ppm	5.4	15	94	2.8
K_pct	0.10	0.27	92	2.7
Al_pct	1.35	3.62	91	2.7
Li_ppm	6.3	16.6	90	2.6
Cu_ppm	27.37	70.64	88	2.6
Cs_ppm	0.99	2.52	87	2.5
Ga_ppm	4.5	10.6	81	2.4
Th_ppm	2.6	6.0	79	2.3
Bi_ppm	0.08	0.18	77	2.3
Mg_pct	0.70	1.57	77	2.2
Au_ppb	0.9	2.0	76	2.2
U_ppm	0.48	1.00	70	2.1
As_ppm	4.9	10.0	68	2.0
Zn_ppm	67.2	130	64	1.9
Fe_pct	3.29	6.31	63	1.9
Ni_ppm	33.4	63.5	62	1.9
Pb_ppm	5.46	10.38	62	1.9
Sb_ppm	0.25	0.47	61	1.9
Sn_ppm	0.6	1.1	59	1.8
Cd_ppm	0.12	0.22	59	1.8
Y_ppm	12.85	23.4	58	1.8
Sr_ppm	63.7	110.7	54	1.7
La_ppm	15.6	27	54	1.7
Na_pct	0.051	0.088	53	1.7
Co_ppm	14.7	24.2	49	1.6
Ce_ppm	32.0	52.4	48	1.6
Ca_pct	0.67	1.09	48	1.6
Ba_ppm	144.1	227.7	45	1.6
Hf_ppm	0.12	0.18	40	1.5
Cr_ppm	32.7	46.7	35	1.4
Mn_ppm	634	897	34	1.4
Zr_ppm	8.1	11.4	34	1.4
Ag_ppb	37	52	34	1.4
V_ppm	68	83	20	1.2
Mo_ppm	0.62	0.74	18	1.2
Ti_pct	0.138	0.135	-2	1.0
P_pct	0.103	0.096	-7	0.9
Nb_ppm	0.08	0.07	-13	0.9

Spatial Distribution

Before interpreting any surface geochemical dataset, it is important to evaluate the influence that topography and drainage patterns may have on the distribution of elements in geographic space. It can be complicated to discern the influence of geological structures from topographic features, as many streams tend to follow zones of weakness such as faults. In this case however, the samples were deliberately taken from the basal till, or its closest equivalent, which was not washed out by meltwater (Jackaman & Sacco, 2014). As such, the observed spatial distributions reflect the composition of the underlying lithology, up to a few km west-southwest in up-ice direction, combined with the potential influx of elements from groundwater. The sample sites can be verified by plotting the KML files in Google Earth or any GIS software platform.

When we view the data in geographic space, the difference in data precision between the two size fractions is most pronounced in those trace elements that are near their detection limit in the < 63 µm fraction. The enhanced precision, as a result of the greater trace-element contents in the finer fraction, translates into improved spatial continuity of element trends, as observed in the interpolated grid maps of Au, Tl, and Hg (Fig. 7). For example, the median Au content in is 2.2 times greater than in the coarser fraction (Table 1), producing a much less erratic distribution pattern on the gridded map. This emphasizes the importance of data-quality assessment: although most of the Au data in the < 63 µm fraction are above the analytical detection limit, the poorer precision produces a more erratic spatial distribution with many more false positives on the map. If any of these false positives were to be interpreted as true anomalies and used as exploration drill targets, much of an exploration budget could be wasted on barren ground. Less obvious but more important on these maps are the false negatives: seemingly barren ground in the coarse fraction that would be dismissed in lieu of the subtler anomalies in the clay-sized fraction. Enlarged probability plots of these elements are included to illustrate in detail the difference in precision as it is observed in data space (Fig. 8). Other trace elements such as Cu, Pb, and Zn are also significantly higher in the clay-sized fraction, but as these are more abundant in the bulk till material, the contents in the coarser fraction are all well above their detection limit and they don't demonstrate such a significant improvement in analytical precision (Fig. 6).

When compared to the most recent geological map of the area (Angen *et al.*, 2017), some major structures stand out that may control the observed trends in Au, Tl, Hg, and other elements: the southeast-northwest Tatuk and Tasa Fault zones across the central part of the TREK Project area, as well as the northeast trending Natalkuz Fault along the Nechako river to the north (Fig. 7). Other elements with similar elevated trends along the Tatuk Fault include Ag, As, Cd, Mo, Sb and Zn. This suite of chalcophile elements can be interpreted as pathfinders towards buried gold or base-metal mineralization in the basement, provided that all relevant transport mechanisms are considered. The most prominent and spatially continuous multi-element anomaly of Cu, Au, Ag, As, Cd, Hg, Mo, Sb, Tl, and Zn occurs along the Tatuk Fault, just northeast (down-ice) from the Chu molybdenum-copper porphyry deposit. Two mineral showings are reported directly southeast of the most anomalous till samples as well: one containing Au, Ag, and Zn, and one carrying Mo-Cu mineralization (BCGS, 2017). The Capoose gold-silver and 3T's gold-vein deposits are recognized as individual anomalies but lack coverage to identify spatial trends around them. The Blackwater gold deposit only shows a subtle Au anomaly in the clay-sized fraction. Readers are encouraged to download the data from Geoscience BC's website for further interpretation of the clay-fraction geochemistry in relation to other spatial data.

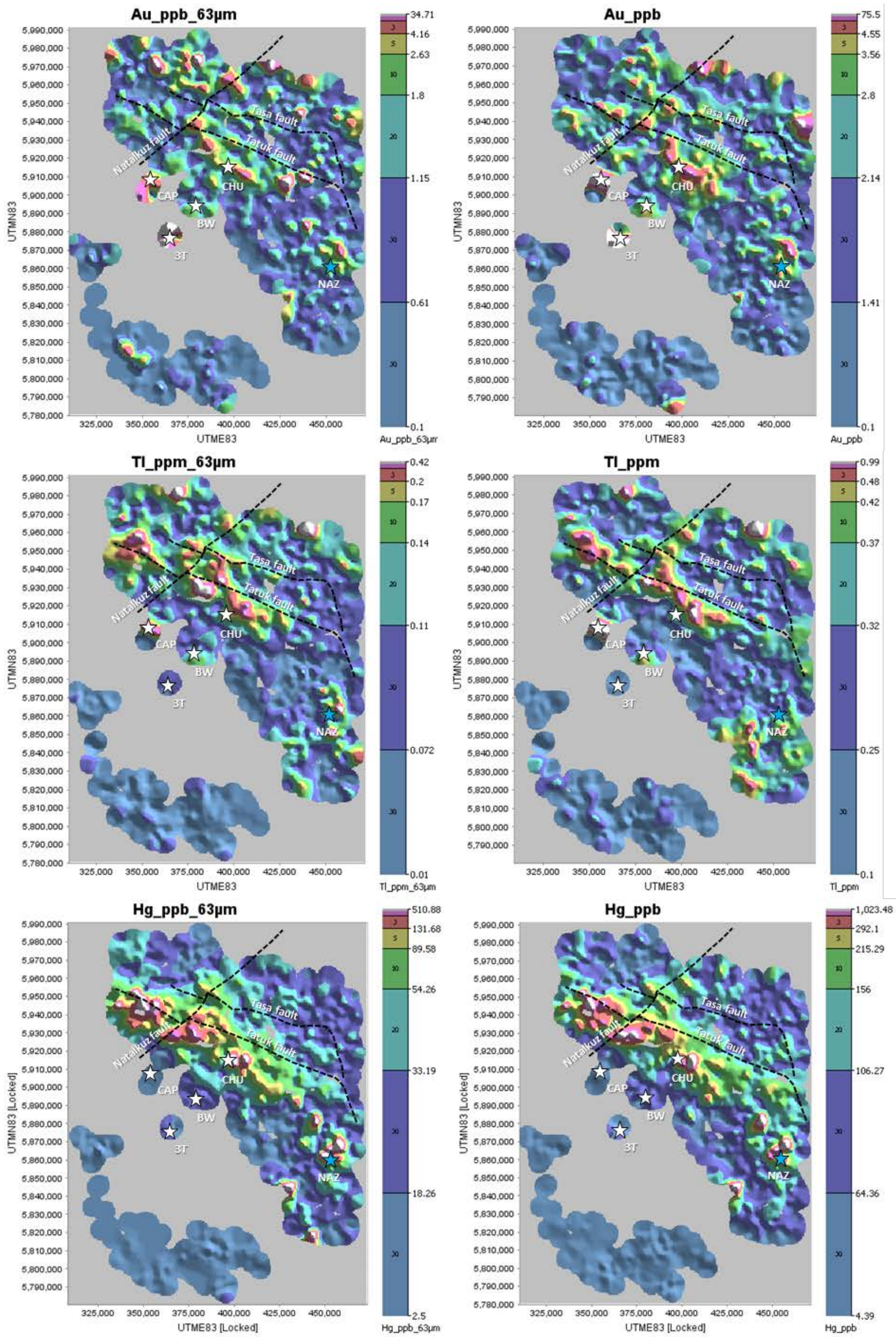

Figure 7: Interpolated grid maps of Au, Tl, and Hg in the < 63 µm (left) and < 2 µm fractions illustrate the improved spatial continuity and enhanced contrast because of greater data precision in the finer fraction. Overlay, major faults & deposits: CAP = Capoose Au-Ag, 3T = 3T's Au, BW = Blackwater Au, CHU = Chu Mo-Cu, NAZ = Nazko aggregate

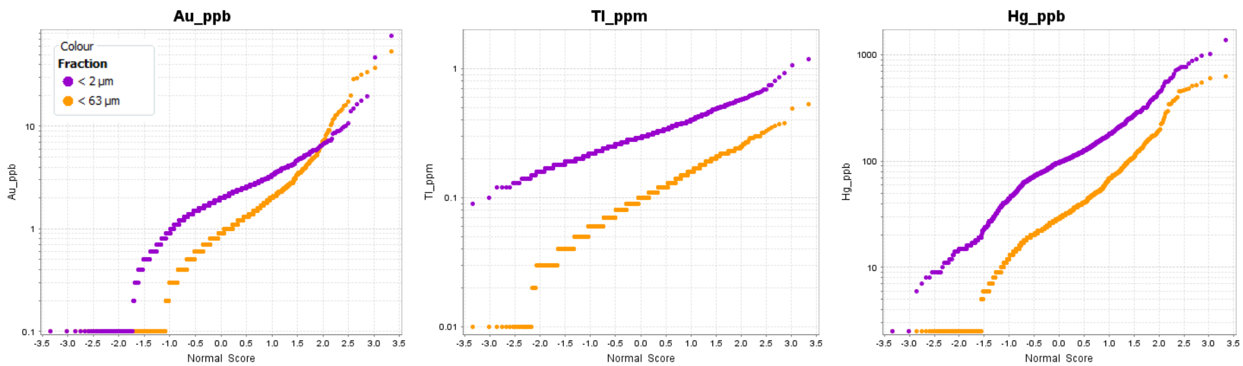

Figure 8: Detailed probability plots illustrating the improved precision in Au, Tl, and Hg data in the < 2 µm (purple) relative to the < 63 µm fraction (orange).

Homogeneity of the Clay Fraction

The greater element contents in the clay-sized fraction are not directly proportional to those in the coarse fraction, indicating the mineralogical differences discussed above (Fig. 9). Those elements that are proportional in each fraction, i.e. display linear trends in the x-y plots, tend to be the chalcophile and labile components (e.g. As, Bi, Cs, Ni, Sb, and U), whereas the elements that are preferentially partitioned into minerals favouring either size fraction show less linear, more scattered trends (e.g. Al, Ba, Mn, Sr, Y, and Zr).

The mineralogical differences between the two fractions can be emphasized in molar element-ratio diagrams of the major rock-forming elements (van Geffen *et al.*, 2012). Ratios of Fe, Mg, Ca, K, and Na over Al, even following the incomplete aqua-regia digest and in the absence of Si data, illustrate the presence of ferromagnesian and calcic minerals in the < 63 µm fraction, visible in trends that depart from a cluster of an Al-rich background composition (Fig. 10). The < 2 µm fraction is more homogenously clustered with a similar but more Al-rich composition. The ratios of K and Na over Al depict less of a difference between the size fractions, only a minor shift from Na to K-dominant compositions in the < 2 µm fraction.

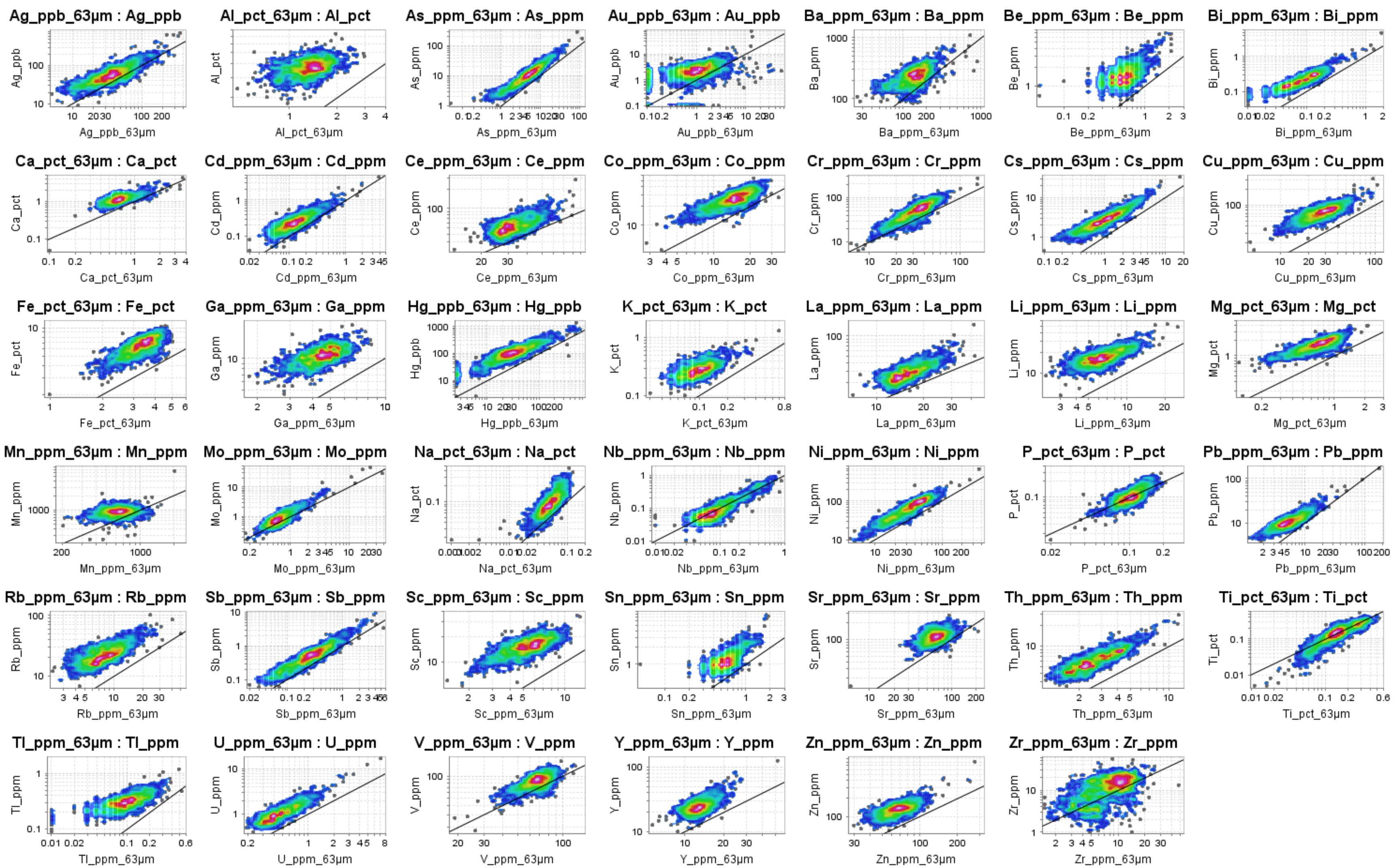

Figure 9: Scatter plots of the < 2 µm fraction against the < 63 µm fraction results, with data-density colour shading and y = x line for reference, logarithmic scale.

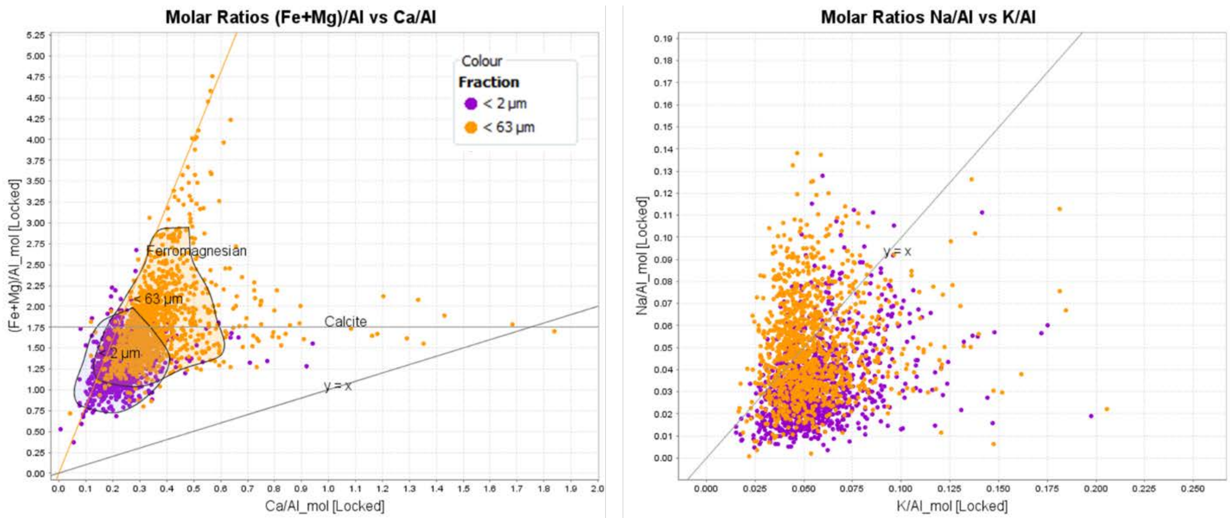

Figure 10: Molar element ratio diagrams of (Fe+Mg)/Al versus Ca/Al (left) and Na/Al versus K/Al, depicting greater mineralogical variability in the < 63 µm fraction (purple). Reference lines are included to highlight compositional trends departing from the background data cluster, as well as the 90th percentile contours of each size fraction in the Fe-Mg / Ca graph.

A major implication of a more homogenous mineralogical composition of the clay-sized fraction is not only a reduction of noise and increased precision with greater element contents, it also provides a more homogenous substrate for positively charged metal ions and ionic complexes that reflect the hydromorphic component of the overall trace-element content. In other words, variations in trace elements such as Au, Hg, Sb, As, Tl, Bi, and others are more likely the result of redistribution by groundwater and formation waters, rather than just a reflection of the mineralogical variations in their lithologic provenance. The spatial correlation with major structural breaks already implies the movement along fault zones, and the homogeneity of the clay-fraction mineralogy, as a substrate for ionic species, enhances the confidence that these patterns can indeed be ascribed to hydromorphic transport of these elements. The notion that trace-element anomalies in the clay-sized fraction are likely to reflect buried mineralization, particularly when suites of correlating pathfinder elements can be identified with confidence, has the potential to greatly benefit exploration efforts in terrain with extensive transported overburden.

Notes on Sampling and Implementation

Geochemical analysis of the clay fraction is not a new concept, and was in fact widely applied during the 1960's and 1970's (Nichol & Bjorklund, 1973), but fell out of favour because of cost considerations and handling requirements. However, the method described in this report does not require special sampling protocols or additional handling instructions, other than standard till sampling guidelines (e.g. Levson, 2001), and the cost difference is only marginal. The samples ('character splits') used in this study ranged in gross weight between 125 and 500 g, which was enough to extract sufficient clay-sized material for aqua-regia analysis of about 0.5 g. Whereas the list price of clay extraction at commercial laboratories can be in the order of $10-15 per sample (e.g. BV Minerals, 2017), it should be considered that no other preparation is necessary, such as sieving, which typically costs about $5 per sample. And because only a small aliquot of clay-sized material is required for aqua-regia digest and ICP-MS analysis (0.5-1.0 g), another $4-5 can be saved in comparison to the cost of analyzing 15-30 grams of coarser material. It is generally not advisable to switch sampling or analytical methods mid-program, but any new till sampling program should consider the clay fraction as a preferred medium for greater data precision over conventional size fractions.

Conclusion

The clay-fraction geochemistry of the TREK Project tills provides a valuable data layer that can be used in conjunction with other regional data types to assist exploration efforts in BC's Interior Plateau, as well as in other areas with similar terrain and cover materials. Large-scale geological features are recognized in trace-element maps and several multi-element anomalies can be correlated with areas of known mineralization. In particular, this study has demonstrated the following five benefits of extracting the clay fraction from basal till material, in comparison to the standard procedure of sieving to < 63 µm silt + clay fraction: 1) greater trace-element contents, 2) improved data precision, 3) enhanced anomaly-to-background contrast, 4) a more homogenous, clay-rich mineral composition that acts as a substrate for adsorbed ionic species, and 5) greater confidence that trace-element patterns observed in geographic space reflect the transported hydromorphic component, in addition to the inherent geochemistry of the till's provenance. Organizations undertaking future exploration programs will have to weigh these benefits against the marginal additional cost of clay extraction and evaluate whether it fits their purpose. Considering the demonstrated improvements in data quality, we recommend that it be given serious consideration before budget decisions preclude a potential discovery.

Acknowledgments

We would like to acknowledge Ray Lett, emeritus at the BC Geological Survey, for his help with sample retrieval from the BCGS archives in Victoria. Wayne Jackaman helped us recover a large portion of the TREK till "character splits" from his storage. And we would like to thank Jamil Sader at Bureau Veritas for securing preferred rates and services towards this public geoscience undertaking.

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