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THE APPLICATION OF DIFFERENT ANALYTICAL EXTRACTIONS AND SOIL PROFILE SAMPLING IN EXPLORATION GEOCHEMISTRY

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ABSTRACT

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This paper deals briefly with the principles of geochemical migration in the secondary (soil, sediment) environment, a knowledge of which is essential to a correct interpretation of exploration geochemical data. Examples are given which illustrate that the principles which apply in the more easily interpreted tropical areas, also apply in the more complicated glaciated regions. Any person employing exploration geochemistry in geomorphologically complicated areas, is well advised to study data from strictly residual soil areas where the fundamentals of geochemical migration are more easily observed. From this base it is easier to understand the additional complications of geochemistry in mountainous and glaciated terrain. Of the variety of exploration geochemical techniques which can be used, this paper deals specifically with two: soil profile sampling, and different strengths of acid extraction of metal from samples. Examples from the different environments are compared and contrasted.

INTRODUCTION

In the secondary environment, anomalous concentrations of metal may be derived from a mineralized source or non-mineralized country rock. The success of the interpretation of geochemical responses is gauged on the extent to which metal distribution patterns can be correctly assigned to mineralized or non-mineralized sources. Two criteria are of fundamental assistance in the interpretation of soil and sediment anomalies. The first criterion is a knowledge of whether the metal as detected has moved mechanically or in solution to the sampling point. At the present time the most effective way of making this distinction is by the use of different chemical extractions. The second criterion is a knowledge of the distribution of the metal in the soil profile. By the use of these two criteria it is normally possible to distinguish between soil anomalies directly over mineralization, displaced from mineralization, and anomalies unrelated to the mineralized source. Similarly, in sediments it frequently is possible to discriminate between anomalies related to sulphide mineralization and those due to changes in lithology.

The purpose of this paper is to show that these criteria are applicable in a residual soil tropical environment and also in the more complex Alpine glaciated and continentally glaciated areas. Geochemical exploration techniques must be altered in detail, however, as the environment changes (and on occasion even within one environment). There are in addition, a large number of practical parameters, such as sampling density, sampling depth, and mesh size to be used. These, however, are in the large part considerations which can only be decided for individual areas on the basis of such factors as geology, land form, drainage characteristic, logistics, and the estimated size and form of the target being sought.

DISPERSION MECHANISM

The mechanism of geochemical migration from bedrock through a soil profile or to stream sediments has been outlined in a number of publications of which the textbook by Hawkes and Webb (1962) is the most comprehensive. Therefore, only the most pertinent features related to an understanding of the examples given in this paper are repeated here.

In a residual tropical environment (Fig.1) geochemical anomalies are

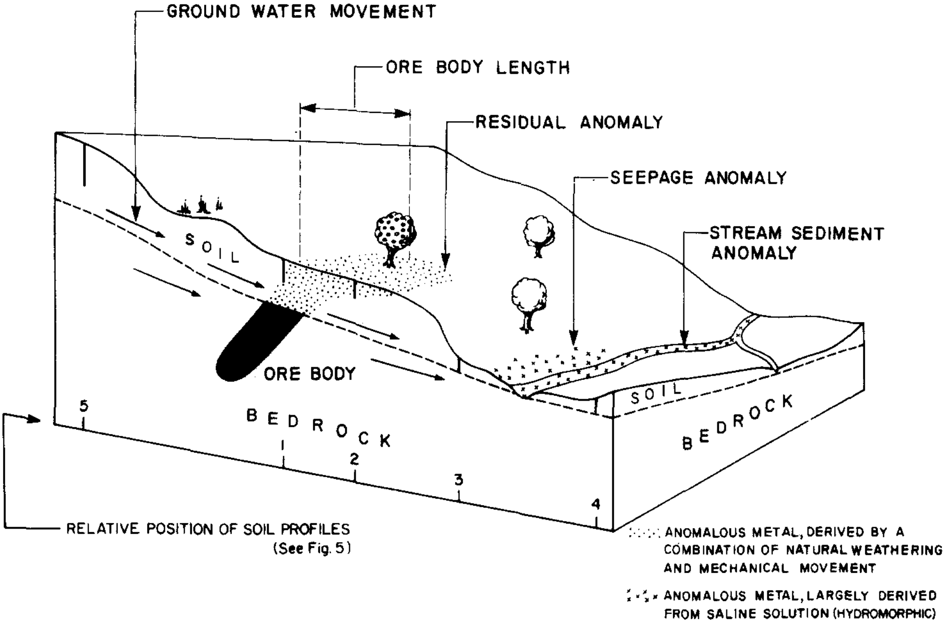

Fig.1. Idealized profile: geochemical anomalies in a residual soil, tropical environment, youthful topography.

characteristically formed directly over mineralization during the normal processes of soil formation (Webb et al., 1963; Tooms et al., 1965; Cole et al., 1968). That is, as the bedrock weathers and the soil forms from the weathering bedrock, the metal derived from mineralization is incorporated in the soil horizons. This anomaly is consequently derived "in situ" by normal weathering processes involving both mechanical and chemical modification. Such anomalies are characterised by relatively strong metal bonding (Tooms and Webb, 1961; Govett and Hale, 1967). The anomaly may be roughly the dimensions of the mineralization. Lateral spreading due to natural causes such as slumping, spreading during the normal process of rock weathering, and soil compaction, may result in an anomaly severalfold larger than the bedrock expression. This is a purely mechanical action and does not chemically alter the metal bonding. In addition to the soil forming process, metal is taken into solution in the generally reducing, slightly acidic, environment below the water table. Furthermore, metal is contributed at a relatively higher rate from weathering sulphide deposits than from barren rock due to the production of sulphuric acid as a component of sulphide weathering. This metal remains in solution until a change in the chemical environment occurs. Such a change is encountered when the groundwater enters the relatively oxidizing and generally less acid conditions of the surface environment, either as seepage areas at the break of slope, or in streams. Here the metal is precipitated on clay minerals, organic matter, hydrous oxides or as the salts of the metals giving rise to hydromorphic anomalies (Woolf et al., 1966; Coope and Webb, 1963). When moved in solution and precipitated in this manner, the metals are generally very loosely bonded.

In Alpine glaciated areas (Fig.2) the dispersion mechanism is very similar to that in a residual tropical soil environment, with one principal difference. That is, the soil anomaly is modified to some extent by glacial dislocation of the anomalous material, although chemical action during soil formation also occurs. The glacial action generally results in a smearing of the anomaly in a down-ice direction (Bergey et al., 1971). Consequently, the surface expression and mineralization is frequently much more extensive than the source and, due to mechanical mixing, the soil anomaly is more discontinuous. Stream sediment anomalies tend to be similar to those in the residual tropical environment with the exception of the bonding. The hydromorphically transported material which discharges into the surface environment in glaciated areas is frequently more strongly bonded than in residual tropical areas. Laboratory experiments and field evidence discussed in this paper strongly suggests that in colder glaciated areas different metal ion complexes are formed than in warmer or non-glaciated areas. A more rigorous attack, such as cold or hot weak acid, may be required to break this bond.

There are a number of variations to the idealized profiles shown in Fig.2. Depending on glacial history, for example, in large U-shaped valleys the thickness of the fluvio-glacial material is such that no geochemical soil anomalies can be detected through it, and old glacial lake beds exist which

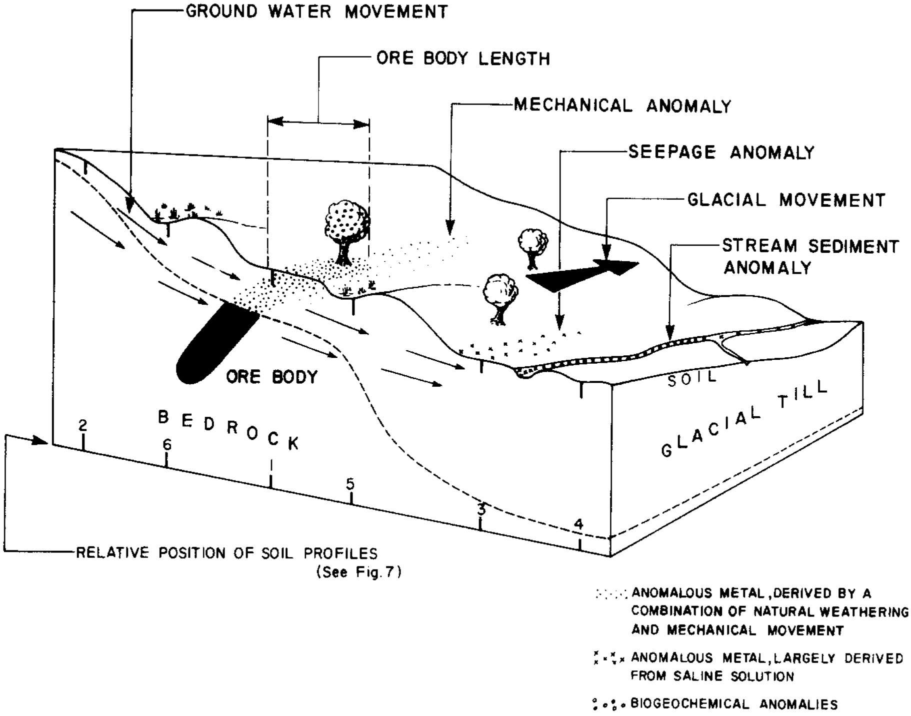

Fig.2. Idealized profile: geochemical anomalies in an Alpine glaciated area, youthful topography.

effectively mask all geochemical response when conventional techniques are used. Away from these areas, geochemistry in both soils and stream sediments can be used in the Alpine glaciated environment with confidence (Bradshaw et al., 1973, Part 6).

In the continentally glaciated environment, variations in glacial overburden type are extreme, resulting in very variable success of the use of geochemistry. The idealized profile shown in Fig.3 represents a thickness of glacial till covered by lacustrine clay. In an environment as variable as the continentally glaciated terrain, no model can be said to be typical, although the model given here is quite commonly encountered. A fan-shaped zone of anomalous material generally occurs in the till, a result of glacial action. This extends outwards and upwards in a down-ice direction from the mineralization (Garrett, 1971). If the till is not covered with any material of foreign provenance, soil sampling can be readily used to outline the geochemical anomaly (Band, 1969; Larsson and Nichol, 1971).

Although broadly similar to Alpine glaciated areas, the results of such a survey are generally even more variable and discontinuous. The variability of soil anomalies is dependent on variations in the thickness and provenance of the till, the relative movement of different periods of glaciation, and the

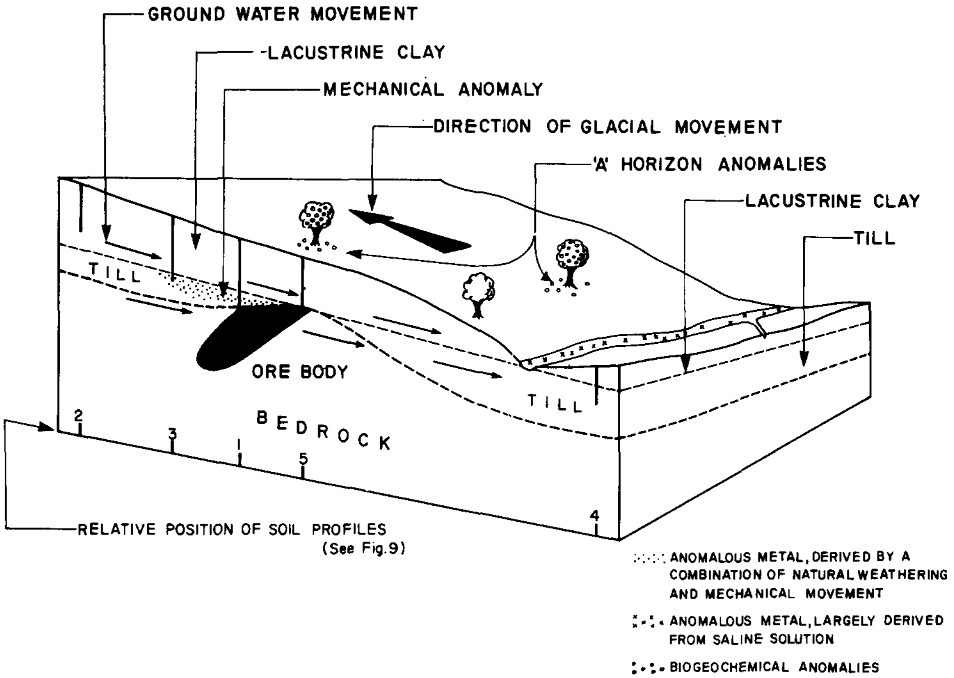

Fig.3. Geochemical anomalies in continentally glaciated areas, mature topography.

presence of fluvio-glacial or lacustrine deposits: factors that are generally less important in Alpine glaciated areas. If root systems can penetrate deep into the till, anomalous metal can be expected to rise into the plant and a soil anomaly will develop, even though the soil may be directly underlain with material of completely foreign provenance. Anomalies will also build up in stream sediments by the mechanism of hydromorphic movement already described for residual soil tropical areas (Boyle et al., 1966). However, anomalies in stream sediments in continentally glaciated areas are generally weaker and more erratic than under comparable situations in residual terrain. This is largely due to dilution by easily eroded barren till or fluvial and lacustrine clays. In varved clay environments groundwater circulation to the surface may be lost completely. In this case no geochemical surface expression will be obtained.

CHEMICAL EXTRACTIONS

"Total metal"-perchloric acid ($HClO_4$) extraction

A hydrofluoric-perchloric acid extraction evaporated slowly to dryness and taken back up in acid, is probably one of the few total wet chemical attacks. For example, Foster (1971) shows data obtained by a hydrofluoric-perchloricnitric acid attack that is comparable with emission spectrography for a number of pure minerals ranging from pyroxene to feldspar and limonite. A lithium metaborate fusion also has the same effect (Van Loon and Parissis, 1968). However, these total attacks are more expensive than less rigorous chemical attacks, which for all practical purposes give data of equal value to exploration geochemistry. One common attack is perchloric acid, without hydrofluoric acid, kept at the reflux temperature for 4 hours, then allowed to cool, and the metal analysed directly (when related to exploration geochemistry, this is commonly called a total attack). This attack extracts all loosely bonded or absorbed metal and metal from alkali silicates such as feldspar, layered silicates such as mica, and sulphides. However, some of the iron-manganese silicates, such as pyroxenes, amphiboles and sphene have only 20—80% of their metal extracted (Foster, 1971, 1973). This extraction is capable of removing between 80% and 100% of the total metal from a soil, or sediment. For all practical purposes this provides a geochemical total analysis sufficient to interpret contrasting geological units, and will extract all metal in the sulphide form or its weathered products.

"Weak acid"-hydrochloric acid (HCl) extraction

Weak acid extractions encompass a large number of attacks of quite variable nature and the specific attack to be used should be chosen by orientation. A weak acid attack will remove loosely bonded and absorbed metal, precipitated salts and possibly attack some of the less resistant silicates such as layered silicates. In some areas (notably residual and tropical environments) an organic acid (e.g., EDTA) is quite sufficient to remove this type of metal. In other areas, however, this attack is not sufficiently strong since, due to some natural process, the adsorbed metal, although having moved in solution, is bonded more strongly. From experience, hot 0.5*N* HCl extraction proves quite effective, but the successful use of the hot weak acid extraction is by no means confined to this strength and type of acid alone.

"Cold-extractable" (EDTA) extraction

As for the hot weak hydrochloric acid this extraction is intended to remove all loosely bonded material adsorbed only on clay particles or other minerals of large surface area, and metal precipitated as its salt. EDTA is a weak organic acid that has proved very effective for this purpose. This extraction has been found to be effective in detecting hydromorphically moved material, particularly in tropical environments, and has been used, with variation, for some considerable time (Webb et al., 1963). Other weaker extractions, such as sodium or ammonium citrate buffer, have been used with success in different environments (Tooms and Webb, 1961; Coope and Webb, 1963; Cooper et al., 1964; Govett and Hale, 1967).

Fig.4 shows $HClO_4$ and EDTA-extractable copper in stream sediments from an area in Fiji containing porphyry copper mineralization. The area is of rugged mountainous rainforest country underlain by intermediate volcanics intruded by a number of granodiorite stocks.

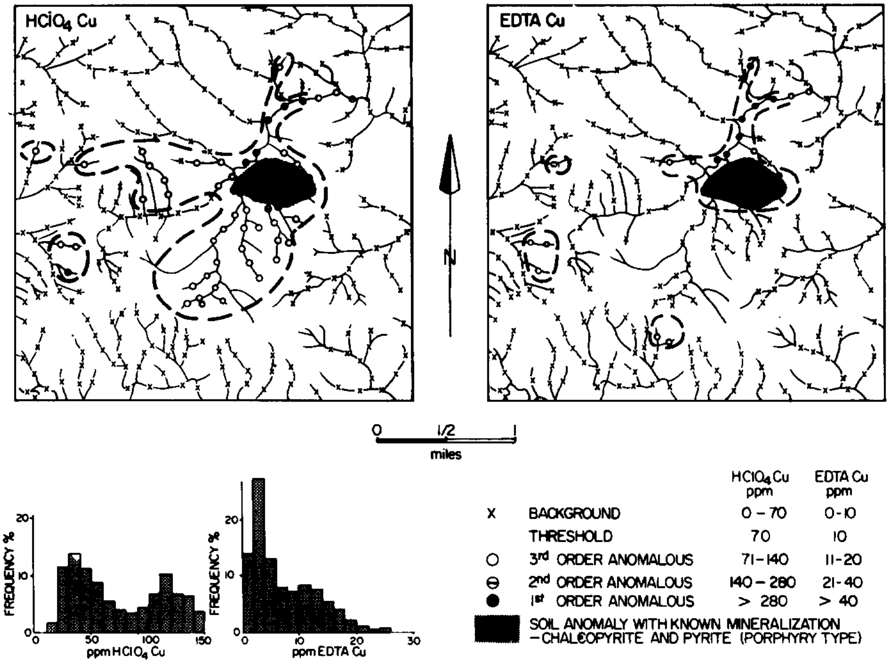

Fig.4. Copper in stream sediments, Fiji.

Both analytical techniques outlines the area of anomalous soils developed over known mineralization, but the patterns observed are somewhat different. The figure also gives the histograms of the metal concentration for background and weakly anomalous values (strongly anomalous values have been omitted in order to conserve space). In the case of the total extraction, the histogram shows two very distinct and well-separated populations. It is evident from the accompanying map that the break at approximately 70 ppm is probably geologically significant since the values from the two populations plot in discrete areas. Of particular interest are the third-order anomalous values upstream from known mineralization which cannot have been affected by dispersion from the area of the soil anomaly. Reconnaissance mapping has shown that this high copper area contains numerous outcrops of unmineralized intrusive rocks which contain slightly raised copper contents. Thus, it is concluded that the total analysis for copper is providing not only an anomaly related to mineralization (with a contrast between threshold to the most strongly anomalous sample of × 12), but also an anomaly pattern related to a change in rock type. The cold-extractable (EDTA) results, on the other hand, show only a very weak separation between the two populations. When this division, at 10 ppm, is chosen as threshold, it is evident that the vast majority of anomalous results are related to the area of known mineralization (with a maximum contrast of × 16). The effect of different bedrock types has been virtually obliterated by this weak extraction. At the time of writing, the three satellite anomalies to the south and west had not been investigated but are interpreted as probably related to mineralization.

From these sediment data, therefore, it can be seen that cold-extractable results, from a mineral exploration point of view, provide data which are easiest to interpret as the anomalies have the best contrast, natural variations due to changes in bedrock have been virtually eliminated, and the known mineralization has been outlined most concisely.

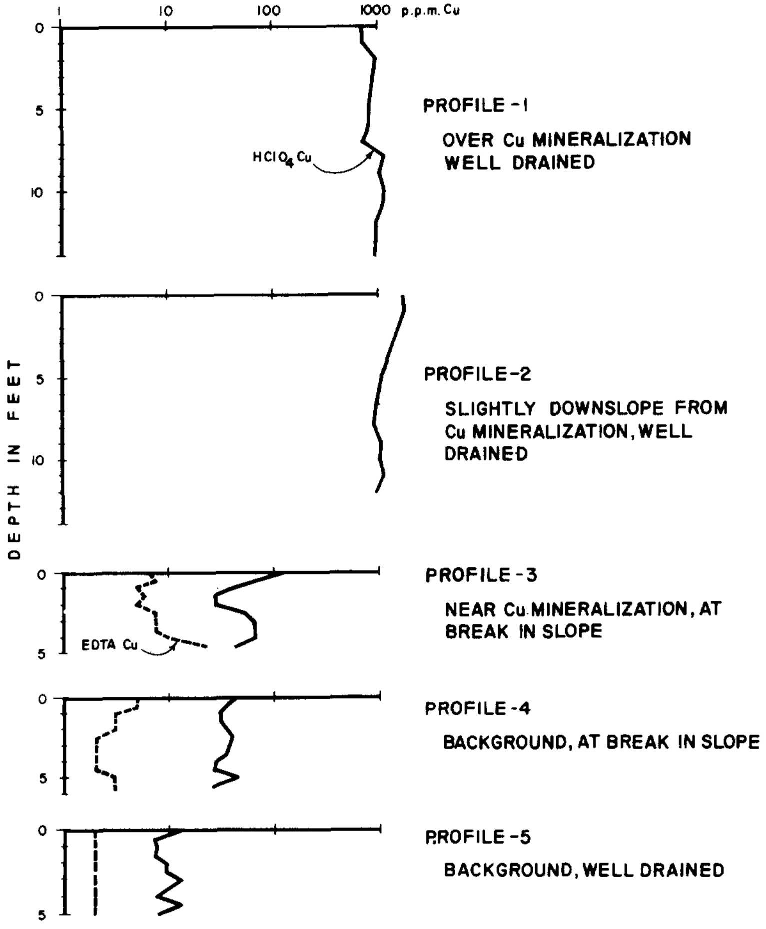

Fig.5. Variation of copper in soil profiles, Fiji.

On the other hand, should bedrock information be desirable, then total analysis is adopted in an environment such as this, many of the anomalies will be solely a reflection of unmineralized rock types and they must be very carefully interpreted in this light.

Fig.5 shows five soil profiles collected both over and away from known copper mineralization in Fiji. The relative position of the soil profiles is shown diagrammatically in Fig.1. Profile 1, which is located over known mineralization, maintains a more or less uniform anomalous level of total copper (greater than 800 ppm), but shows a gentle increase with depth as the mineralization is approached, up to 1500 ppm. Profile 2 is located slightly downslope from mineralization and is anomalous throughout its length. The profile is most strongly anomalous in the upper part showing a more or less continuous decrease in metal concentration with depth, indicating that the source of the mineralization is to one side of the profile. EDTA data are not available for profiles 1 and 2. Profile 3 data were collected downslope from known mineralization at the break in slope. It is weakly anomalous in both EDTA and total copper with the percentage of cold-extractable copper high (10—50%), compared with background profiles (5—10%). This indicates that the anomalous metal has been derived in large part by hydromorphic dispersion and consequently is probably downslope from mineralization. Data in profiles 4 and 5 were collected from background areas; profile 4 at the break in slope and profile 5 from well-drained ground. Both profiles are fairly uniform throughout, with profile 5 not exceeding 20 ppm and profile 4 not exceeding 50 ppm. The percentage of cold-extractable copper is low in both. (It cannot be calculated for profile 5 as all samples were below the detection limit of 2 ppm.)

ALPINE GLACIATED AREAS

Results of three different extractions on the same stream sediment samples collected during an exploration programme in the southwest part of the Northwest Territories are shown in Fig.6. Although the geology is imperfectly known at this time, their strata-bound mineralization has been located in the eastern portion of the surveyed area. The $HClO_4$-extractable copper results show a clear-cut difference between stream *D* to the northwest and the remainder of the area. This probably reflects changes in bedrock. The same attack also picks up a moderate anomaly at the headwaters of creek *A*. These anomalous values do not, however, reflect known mineralization with any degree of reliability as the tributary samples closest to the known mineralization are all third-order anomalous, as are virtually all the results over the south and eastern two-thirds of the area. The cold $0.5N$ HCl-extractable copper gives little apparent reflection of rock types. Using this extraction five of the eight tributaries draining known mineralization are second- or first-order anomalous. In addition, anomalous values are found near the headwaters of creek *B*, and two tributaries draining from the western side of

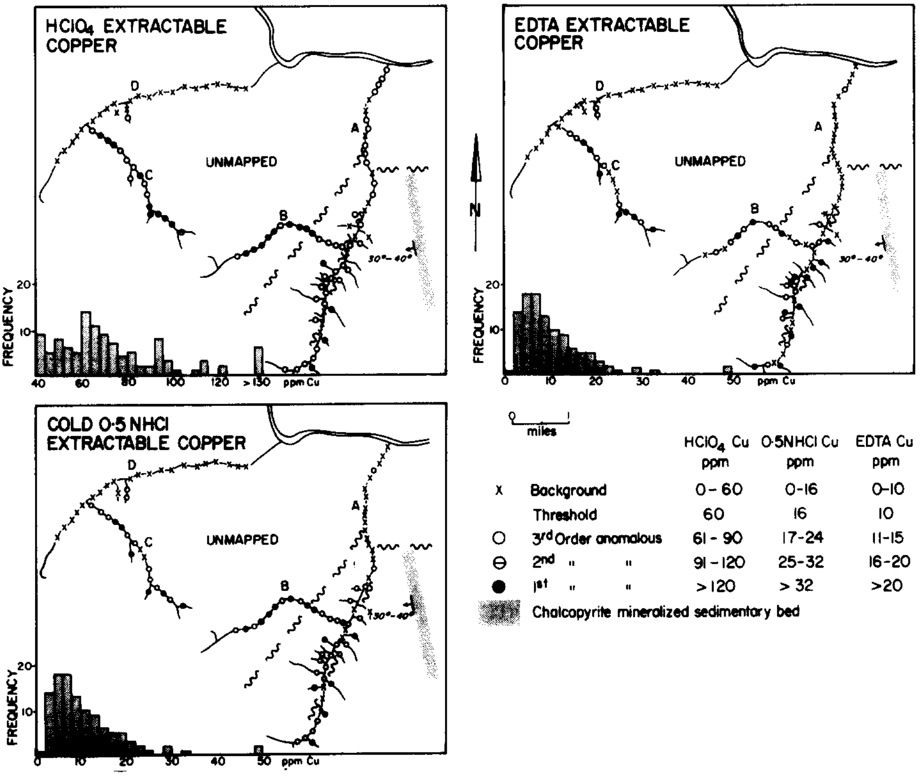

Fig.6. Copper in stream sediments, southwest part of the Northwest Territories.

creek *A*. The contrast between threshold and the most strongly anomalous samples is X 4.7. The EDTA-extractable copper shows virtually no association with bedrock as the downstream portion of creek *A* has the same range of metal concentration as the area to the northwest. In this case, however, seven of the eight tributaries draining from areas of known mineralization are second- or first-order anomalous with a maximum contrast of X 5.0. In addition, the EDTA-extractable copper also shows as anomalous the headwaters of creeks *B* and *C* and two tributaries draining from the western side of creek *A*. These anomalies most probably relate to further mineralization.

In conclusion, therefore, it is noted that the three extractions give different patterns and, therefore, the interpretation of each will lead to different conclusions. Limited knowledge of "ground truth" in the area makes exact interpretation difficult but several points are apparent. The total metal content reflects bedrock composition and at best only imperfectly the presence of the known mineralization.

EDTA-extractable copper reflects the known mineralization very well, and also accentuates two other anomalies in the area, but does not give any indication as to rock type changes. Cold 0.5*N* HCl copper gives a picture intermediate between total and EDTA-extractable copper. Further indication of the differences between the three attacks can be seen by comparing the histograms in Fig.6. The $HClO_4$-extractable copper shows a polymodal distribution indicating that it is reflecting more than one parameter, in this instance different lithologies. Consequently, the task of relating the anomalous values only to mineralization is difficult. EDTA-extractable copper on the other hand has a far more nearly modal distribution. For such a distribution it is possible to choose an unambiguous threshold and anomalous samples may be more confidently related to mineralization.

Six profiles located over the Cariboo-Bell deposit in the McLease Lake area of British Columbia are shown in Fig.7. The relative positions of these profiles are shown in Fig.2, the idealized profile for Alpine glaciated areas. The six profiles form three pairs of anomalous and background profiles from three different situations; well-drained soil on a slope, soil at the break of slope,

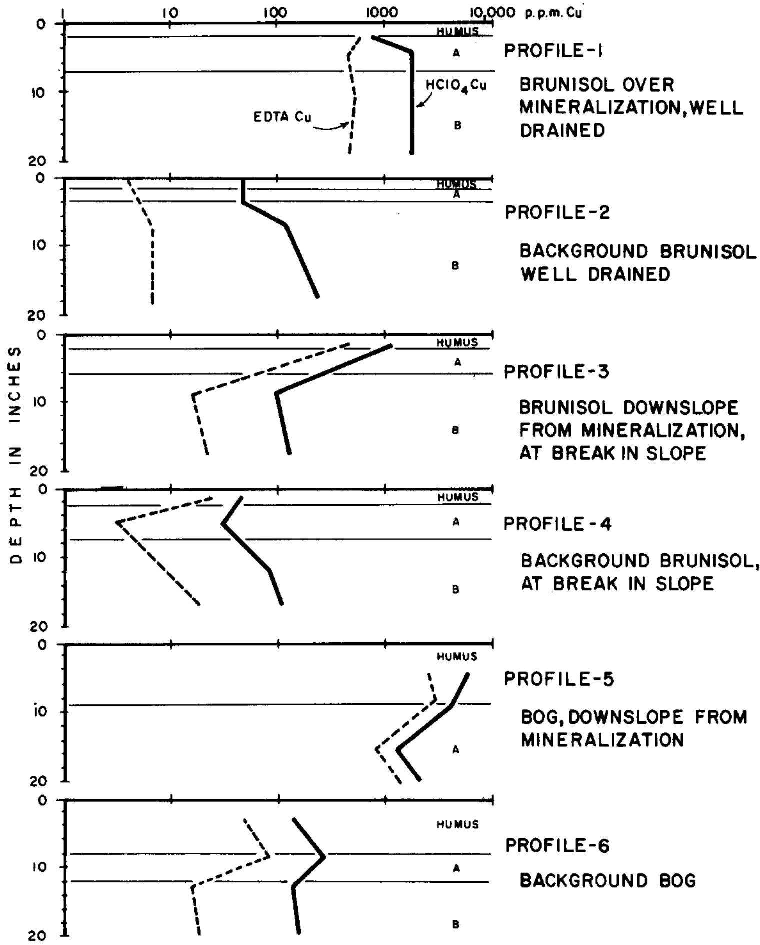

Fig.7. Variation of copper in soil profiles over the Cariboo-Bell deposit, McLease Lake area, British Columbia.

and material from boggy ground. All the background samples are characterized by copper values lower than 300 ppm, while the profiles related to mineralization are generally greater than 1000 ppm. Profile 1 collected from well-drained soil over mineralization shows a uniform level of copper content with depth in the mineral soil, while the uppermost A horizon is slightly lower. It is, however, anomalous throughout its depth and this uniformity is a good indication that mineralization lies directly below the sampling point (compare, for example, profiles 3 and 5). Approximately 20—25% of the metal is cold extractable except in the mineral soil, while in the organic-rich A horizon it is approximately 70% cold extractable. The equivalent background profile (profile 2) is not anomalous and shows 5—10% cold-extractable metal.

Profile 3 is collected 2000 ft downslope from the known mineralization at the break in slope where some seepage or spring water can be expected but bog conditions are not encountered. The total metal concentration decreases very sharply with depth by a factor of ten, falling into the background range below eight inches.

Within the anomalous portion of the profile, the cold-extractable metal is 25—40% consistency, indicating a higher proportion of hydromorphic movement than was observed in profile 10.

Profile 4 is located at the break in slope away from the influence of mineralization and contains only background levels of copper. Profile 5 was collected from a bog downslope from mineralization and is the most strongly anomalous. The percentage of cold-extractable metal is between 45 and 50% throughout the profile providing a very valuable indication that the metal has been moved by hydromorphic dispersion and in all probability the source does not directly underlie that particular profile. The background bog profile, like the other background profiles, is not anomalous throughout its depth showing a quite variable degree of cold-extractable copper from 10 to 35%. If the contour map of a regular soil grid was provided, the boggy ground 600 ft downslope from mineralization would provide the area of highest geochemical response. If this zone was chosen as a geophysical or drilling target, mineralization would not be encountered.

These data demonstrate that profile sampling, even to the relatively shallow depth of 18 inches, can be very valuable in interpreting the location of the related source, either upslope or directly underneath. Because in most soil anomalies overlying mineralization the metal is less readily removed by a cold extraction technique than anomalous metal in seepage areas and bogs, it is essential to initially use a total attack. The cold-extractable or other weak attacks should only be used where further information regarding the nature of the anomaly is required.

CONTINENTALLY GLACIATED AREAS

The results of three different chemical extractions for copper on stream sediments collected in the Northwest Territories are shown in Fig.8. The area

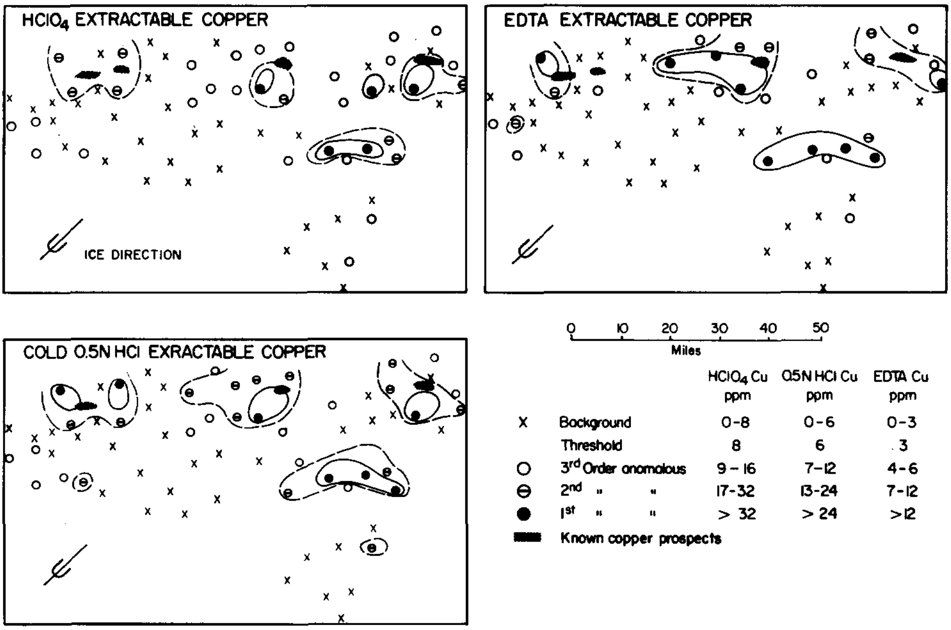

Fig.8. Copper in sediments, McDonald Fault Zone, near McLeod Bay, Northwest Territories.

is virtually completely drift covered and the geology is imperfectly known. The region to the north of the copper prospects is largely underlain by Precambrian metasediments (sandstone, siltstone and some limey sediments) and the area to the south by Precambrian granitic gneiss. The known copper prospects shown in the figure are massive pyrite/chalcopyrite and, as presently known, are sub-economic. They are apparently related to a major east—west shear zone and there is evidence of parallel shear zones elsewhere in the area. The area has a low relief with broad interfluves where no reliable sediment samples can be collected, consequently the sampling density is variable. It is also important to realize that the anomalies will be down-drainage from the source, the contouring of the results has taken this into account.

The $HClO_4$-extractable copper results show a reasonable correlation with the known mineralization, two of the prospects have associated first-order anomalies and the western pair of prospects are reflected by a second-order anomaly. In addition, a fairly strong anomaly in unprospected ground is found to the north. $0.5N$ HCl-extractable copper has first- and second-order anomalous samples associated with each known prospect and also indicates the same southern anomaly. EDTA-extractable copper reflects the two eastern prospects. This extraction also locates the southern anomaly. In general all three extractions give somewhat similar results but the $0.5N$ HCl extraction gives the best response to all known mineralization both in strength and extent. Away from these anomalies copper values in the sediments are similar and no significant reflection of the various rock types is seen with any of the attacks.

Five soil profiles, three collected over known mineralization and two from background areas, are shown in Fig.9, their relative position is also shown in Fig.3. Profile 1 is collected through lacustrine clay into underlying lodgement till which is approximately 4 inches thick. The lodgement till gives a strong reflection of the underlying mineralization for both cold-extractable and total copper. The till at this point contains iron oxide and shows signs of weathering, probably due to groundwater movement. The copper is more easily cold extractable than in anomalous profile 3 (10% vs. 1%), indicating that here some weathering in the till has occurred.

There is also an anomaly in the A horizon sample at the top of profile 1 which may be considered to reflect the underlying mineralization. However, when profile 2, collected several miles from known mineralization, is compared with profile 1, the A horizon material is found to have an identical

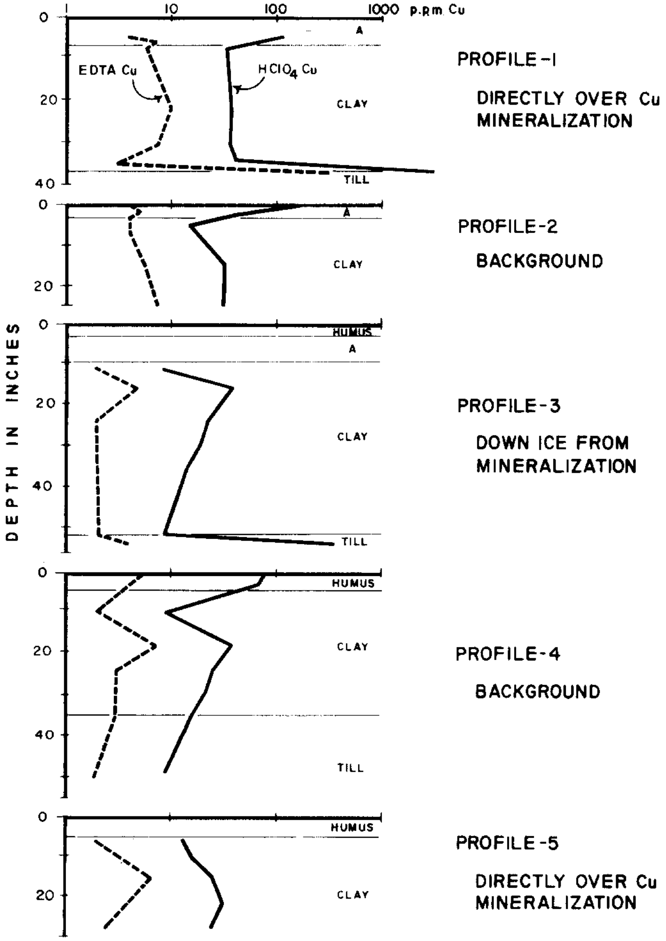

Fig.9. Variation of copper in soil profiles, Central Manitoba.

copper content. At the present time no reliable method has been found to separate "true" and "false" anomalies in the A horizon. Consequently, the only reliable reflection of mineralization is the till sample.

Profile 3 is located immediately down-ice from known copper mineralization and again only the lodgement till sample below the lacustrine clay reflects the mineralization. The fact that the copper is only 1% cold extractable is indicative of almost entirely mechanical movement at this point. This feature demonstrates that cold-extractable copper should not be used to analyse till samples.

Profile 5 is located over known copper mineralization, but the pit stopped in lacustrine clay. The copper content of the lacustrine clay, both cold extractable and total, is comparable with the background profiles and shows that in this area the copper anomaly does not penetrate into the clay (this is consistent with the results from the central portion of profile 1 and 3).

SUMMARY AND CONCLUSIONS

This paper relates entirely to metals which have at least a high component of hydromorphic dispersion such as copper, lead and zinc and is intended to demonstrate two principal points.

(a) That the choice of chemical contraction used on sediment and soil samples is very important to the character and significance of the results obtained. While few extractions can be regarded as giving results of no value to exploration, some provide far more meaningful information than others. The type of extraction used may well vary between reconnaissance and follow-up stages depending on the information required.

(b) The use of soil profile data, even when the profiles are collected from shallow depths, can greatly facilitate in distinguishing between anomalies overlying mineralization and those in seepage or bog areas downslope.

Some of the more pertinent conclusions which may be drawn from the data in this paper are:

(1) While surface geomorphological conditions may differ drastically in tropical residual, Alpine glaciated, and continentally glaciated terrain, the mechanisms of geochemical dispersion are essentially the same, although the surface expression may be different (compare Figs.1, 2 and 3).

(2) The stronger chemical attacks, when used on sediments, tend to accentuate the effects of changes in rock composition and consequently relatively reduce the effect of mineralization. In some cases the mineralization will be clearly seen by the use of a total attack, even in areas of varying geology. In other instances, however, the mineralization is not easily seen by the use of total attack on stream sediments because the variations in metal content of different rock types are large with respect to anomalies from mineralization.

(3) A particular weak extraction, for example 0.5N HCl or EDTA, will not produce the same effect when used to analyse sediments in all areas of the world.

In some instances, the metal is relatively tightly bonded even after hydromorphic movement for reasons that are not clearly understood at this time. In this case, EDTA-extractable metal will not detect the strongest anomaly with the best contrast and the use of a stronger, but still less than total attack should be considered (compare Figs.4, 5 and 8).

(4) The strongest surface soil anomalies do not necessarily relate directly to underlying mineralization but may be the result of hydromorphic movement downslope. Profile samples taken from anomalies overlying mineralization generally maintain the same metal value or increase in metal value with depth. In hydromorphically derived anomalies downslope the metal values decrease down profile.

(5) The percentage of cold-extractable metal is increased in mineral soils where the anomalies are a result of hydromorphic accumulation and increases very dramatically in organic soils under the same conditions.

(6) The depth of sampling during the soil programme may be very critical as variations with depth can be greater than a factor of ten within 6 inches, (see, for example, Figs.7 and 9).

(7) It is very important, even in relatively well explored areas, to undertake orientation surveys, in order to determine the variation in the parameters discussed above.

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