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DISTRIBUTION OF ORE ELEMENTS IN ROCKS FOR EVALUATING ORE POTENTIAL: NICKEL, COPPER, COBALT AND SULPHUR IN ULTRAMAFIC ROCKS OF THE CANADIAN SHIELD

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ABSTRACT

For large deposits of nickel-copper sulphides to separate from an ultrabasic magma, the sulphur content of that magma must be relatively high at an early stage in its crystallisation history. If the sulphur content is high the solubility products of metal sulphides will be exceeded and sulphide liquid or crystals will separate and have the opportunity to coalesce before they can be trapped and diluted by crystallising silicate minerals. The purpose of this study was to discover whether there was indeed enrichment of sulphur or the sulphides of copper, nickel, and cobalt in ultramafic rocks associated with copper-nickel deposits and, if this were so, to determine the best combination of chemical variables for predicting the ore potential of an ultramafic body.

1,079 samples of ultramafic rock were collected from 61 widely scattered localities across the Canadian Shield and the Eastern Townships of Quebec. 372 of these samples come from ultramafic bodies associated with moderate to large deposits of nickel-copper sulphides; 91 are from bodies associated with small deposits or significant showings of sulphides; while the remaining 616 samples come from barren ultramafics or those containing only minor quantities of sulphides. Copper, nickel and cobalt present as sulphides were determined on these samples by atomic absorption spectrometry, following leaching with a mixture of ascorbic acid and hydrogen peroxide. Sulphur was determined by a combustion method.

Sulphur and leachable copper, nickel, and to a lesser extent cobalt are considerably enriched in the ore group of ultramafic rocks compared to the barren samples. Discriminant equations have been derived to best distinguish between the ore and the barren groups. Copper and sulphur contribute the most information to these discriminant equations, with nickel less and the contribution of cobalt negligible. Differences in the ratios of the above elements between the different

groups of ultramafic rocks and between these rocks and the associated mineral deposits contribute to an understanding of the processes of chemical adjustment between sulphides and silicates that take place after crystallisation of the primary minerals.

DISTRIBUTION OF ORE ELEMENTS IN ROCKS FOR EVALUATING ORE POTENTIAL: NICKEL, COPPER, COBALT AND SULPHUR IN ULTRAMAFIC ROCKS OF THE CANADIAN SHIELD

by

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INTRODUCTION

In a paper by Cameron and Baragar (this volume) the frequency distribution of copper in two volcanic groups was compared. One of these groups contains copper mineralization that appears to have been derived by the segregation of copper sulphides within the basaltic magma. The other group is barren of such mineralization. Another type of mineral deposit that is considered by many to have been derived by the segregation of sulphides within a magma are the nickel-copper ores that are associated with ultramafic rocks. These form an economically important class of mineral deposits. In this present paper the distribution of the elements forming the sulphide component are compared for ore-associated and barren ultramafic rocks.

The approach taken in the investigations reported in the two papers has been quite different. In the case of the study of volcanic rocks by Cameron and Baragar three belts of volcanic rock were sampled with a moderate to high degree of intensity. For the study described below a very large number of ultramafic bodies scattered across much of the Canadian Shield and adjoining areas have been sampled in reconnaissance fashion. This approach has, in part, been determined by geological considerations. Within the Coppermine River and Yellowknife volcanic belts, those rocks that are presently exposed at the surface probably form a fairly representative sample of the original magma. In turn, the samples collected for analysis are reasonably representative of the rocks exposed at the surface. In contrast, many of the ultramafic bodies of the Canadian Shield are small in extent and are poorly exposed. There are, for instance, over two hundred ultramafic bodies along the Waboden-Moak Lake portion of the Thompson Belt, of which only three outcrop (Kilburn et al, 1969). Thus the samples that we can collect without great expense from these bodies are unlikely to be representative of the original magma. We cannot, therefore, hope to construct distribution curves as we did for the Coppermine Group and expect to find a depletion of the sulphide component in some parts of the magma, compensated by an enrichment in others. The questions posed in this study cannot therefore be strictly quantitative. Rather, we have broadly compared the sulphide content of ultramafic bodies which appear to have or have not an association with nickel-copper ores.

There has been a substantial amount of work carried out in the Soviet Union on the geochemical differentiation of nickel-bearing mafic and ultramafic sequences from barren ones. The results reported by Godlevskiy (1959), Polferov (1962), Polferov et al (1965), Polferov and Suslova (1966) and Volkov (1963), particularly in the enrichment of trace metals within the mineralized sequences, have encouraged the writers to undertake this work in Canada.

Copper, nickel, cobalt and sulphur were determined in the samples. Since the purpose of the study was to examine the frequency distribution of the sulphide component of the magma, particular emphasis was given to employing an analytical method that separated the fraction of the copper, nickel and cobalt present as sulphide from the silicate component of these elements.

NICKEL - COPPER DEPOSITS IN ULTRAMAFIC ROCKS OF THE CANADIAN SHIELD

Because of their economic importance, nickel-copper sulphide ores associated with ultramafic bodies within the Canadian Shield have received a considerable amount of study, much of which is unfortunately not published. Recent summary reports have been given by MacKenzie (1968) and by Kilburn et al (1969). A report covering all North American deposits was written by Cornwall (1966).

Deposits of nickel-copper sulphides within the Shield show all stages of segregation and migration of their sulphide component. In some the sulphide remains thinly disseminated through parts of its ultramafic host. In other deposits the sulphides have been segregated into zones of high grade ore and in some cases the sulphide has moved away from its original ultramafic host to be intruded into the adjoining country rocks. The degree of segregation and migration experienced by the sulphides is, of course, influenced by the type of ultramafic body and its geological history. Kilburn et al (1969) have grouped ultramafic host rocks into three broad types, two of which are of importance in Canada. In the first type, the ultramafic rocks are intruded along belts of major crustal faulting. The Thompson Belt, running for 100 miles along the boundary between the Churchill and Superior sub-provinces of the Canadian Shield, is a good example of this orogenic type. The second or volcanic type consists of concordant sills or dikes distributed through volcanic belts. There is an extensive development of this type of ultramafic rock through the Archean greenstone belts of northeastern Ontario and adjacent parts of Quebec. Nickel-copper sulphides in the ultrabasic sills of the Shield have often settled by gravity as bands and lenses along the bases of the sill and in some cases may have penetrated the wallrock of this lower contact. Segregation in this form is less common for the bodies that have been intruded along orogenic belts. These bodies more often tend to contain disseminated ore, although the Thompson ore body is a prime example of an ore body that has been injected into the wall-rock. Further reflecting the relative minimization of segregation processes within the bodies of the orogenic belts, the ultramafic host rock is more homogeneous than that of the bodies intruded along volcanic belts. These latter bodies may often be differentiated into mafic as well as ultramafic rock types. The ultramafic rocks of both associations have usually been serpentinized to a greater or lesser extent.

While a magmatic segregation hypothesis cannot be doubted for many of the nickel-copper deposits of the Canadian Shield, evidence has been presented by Sullivan (1959), Wilson and Brisbin (1961), Kullerud and Yoder (1965), and by Naldrett (1966) that some deposits may have formed by sulphurization of ultramafic rocks. In understanding this process it is important to consider the chalcophile character of the elements involved. Goldschmidt (1954, p. 19, Table 5) lists these elements in the following order of increasing chalcophile character: iron, nickel, cobalt, copper. During the crystallisation of the ultramafic rock the iron, nickel, and the cobalt may enter both the silicate and sulphide minerals; in the absence of sulphur they can be entirely contained within the silicate components. In contrast, copper is so strongly chalcophile that it shows little tendency to enter the ferromagnesian silicates and it may persist after crystallisation of the basic magma as a residual copper sulphide liquid (Wager et al, 1957). If sulphur is introduced into a partially or completely crystallised ultramafic rock that previously contained little sulphur, sulphides are derived by the reaction of sulphur with the iron, nickel and cobalt bearing silicates, principally olivine. In the presence of limited quantities of sulphur, the nickel and cobalt sulphides will tend to dominate the iron sulphides because of the more chalcophile character of these metals. After formation of the nickel-rich sulphides, which are dispersed through the ultramafic rock, there must be a mechanism for their concentration to ore grade. These ores, if such exist, will tend to have a low Cu/Ni ratio, a character which they are likely to share with the earliest precipitated magmatic sulphides (Wager et al, 1957; Chamberlain, 1967).

SAMPLING OF ULTRAMAFIC ROCKS

The samples utilized for this study were collected by G. Siddeley for a broader project on the total major and trace element content of ultramafic rocks from the Shield and adjoining areas. For this study, 1079 rock samples have been grouped into 61 locations. Some of these locations contain samples from but one ultramafic body; others are composed of samples from several bodies. Thus some locations represent a few hundred or thousand of feet of section of ultramafic rock; others may represent bodies scattered over several tens of miles. An estimate of the maximum distance between samples for each location is given, along with other data on the nature and sampling of the bodies, in Table 1. The geographic locations of the samples are shown on Figure 1.

Because of the often limited exposure of these bodies all possible types of sample had to be utilized. Thus the total sample is a mixture of outcrop hand specimens, hand specimens from mine workings, and drill core. In all cases as close to 1/2 lb. of sample was crushed and ground by the same procedures given by Cameron and Baragar (this volume).

For mineralized intrusions the ore zone itself was strictly avoided during the sampling, since the purpose of the work was to discover whether significant mineralization was scattered through the rest of the rock body. The samples are as representative as was possible of the total population of the ultramafic rock in the body. For inhomogeneous bodies, the different parts of the body were usually sampled.

CLASSIFICATION OF NICKEL OR NICKEL-COPPER DEPOSITS BY SIZE

An attempt has been made to discover the quantity of nickel and copper sulphides associated with each of the ultramafic localities that have been sampled. Unfortunately, the resulting data can only be an approximation of the truth, since the different bodies have been explored with a varying degree of thoroughness and the cut-off grades for ore vary from company to company and from locality to locality. Further, ore reserves are considered by some companies to be confidential information and are not published.

In Table 1, mineralization that is known to be associated with the different bodies has been noted. In Table 2, those localities that contain workable, potentially workable, or significant showings of nickel and copper sulphides are examined in more detail. Those localities that are not included in Table 2 are classed as BARREN, even though they may contain minor amounts of sulphide.

In order to have a non-subjective judgement as to which localities should be classed as ORE, all those which have produced, or have known reserves containing greater than 5000 tons Ni + Cu have been placed in this category. The remainder, with less than 5000 tons Ni + Cu, have been classed as MINORE (mineralized to minor amounts of ore grade material). Note that apart from important showings of sulphides this group contains one former producer (the Alexo Mine), so that inclusion within this group does not necessarily preclude commercial exploitatio The Preissac Township locality is classed as MINORE because these samples were collected several miles from the Marbridge Mine in what appears to be the same band of ultramafic rocks.

ANALYTICAL CONSIDERATIONS

Copper, Nickel, Cobalt: Since the purpose of this study was to examine the distribution of the sulphide fraction of certain metals in ultramafic rocks, it was important to use an analytical method that is specific for sulphides. This is of particular importance for nickel and cobalt because these elements may be several times more abundant in the silicate plus oxide component of a sample than in the sulphide component. The method used for copper, nickel and cobalt employs a cold leach with a mixture of ascorbic acid and hydrogen peroxide to selectively dissolve the sulphides. This method has been extensively used in the Soviet Union (Yegorova, 1938; Dolivo-Dobrovol'skiy and Klimenko, 1947; Dodin, 1963; and Smirnova et al, 1968). The details of this method are given in an appendix by John J. Lynch.

Smirnova et al (1968) have stated that the common sulphide minerals of ultramafic rocks are soluble in an ascorbic acid-hydrogen peroxide mixture within a few hours. In this study we have confirmed that pyrite, pyrrhotite, pentlandite, chalcopyrite, tetrahedrite, sphalerite, galena, and arsenopyrite are soluble when present in the amounts commonly encountered in a mineralized rock sample.

Testing of the other facet of leaching efficiency--the extent to which these metals remain within the silicate and oxide minerals of the rock--is more complex. Leaching of a monomineralic concentrate of a silicate or oxide mineral may give misleading results because of possible sulphide inclusions within these minerals. By leaching samples of olivine, clinopyroxene, and titanomagnetite with ascorbic acid-hydrogen peroxide, Smirnova et al (1968) found quite substantial amounts of nickel and cobalt to be dissolved. For olivines, the average percentage that were dissolved was 41.7% for Ni, and 51.8% for Co; for clinopyroxenes 34.8% for Ni and 38.8% for Co; and for titanomagnetites 54.9% Ni and 60.0% Co. They attributed these soluble amounts to sulphide inclusions. For this study we have tested two samples of olivine, a clinopyroxene, and an orthopyroxene. The resulting data (Table 3) are very satisfactory, for they indicate that the amounts of nickel and cobalt leached from the silicates are quite minor, in every case being less than 5% of the total. For all samples but the orthopyroxene the total copper contents are quite trivial. The orthopyroxene contains 33 ppm Cu which is due to copper as sulphide (T.N. Irvine, personal communication). This amount has been almost totally leached from the powder.

For ultramafic rocks which have been serpentinized or altered in other ways, the matter is less clear. Are the metals bonded within silicate alteration products also stable to ascorbic acid-hydrogen peroxide attack? Changes that accompany serpentinization include the reduction of metal sulphides or of metals bound within the primary silicates (Ramdohr, 1967) to metal alloys such as awaruite, Ni_3Fe. A sample of josephenite, composed of a mixture of awaruite and native copper, tested by Mr. Lynch decomposed when attacked for several hours with ascorbic acid-hydrogen peroxide. But does this hold for other alloys and other mixtures?

The detection limit of the analytical method is 1 ppm for Cu, 2 ppm for Co and 3 ppm for Ni. Copper and cobalt could not be detected in some samples. For the purposes of calculation, values of 1 ppm Cu or 2 ppm Co were given to these samples. Productivity of the analytical method for copper, nickel and cobalt were 100 samples per man day using an automatic sampling attachment, punch tape output, and calculation of the ppm values by computer.

Sulphur: Sulphur was determined by a rapid combustion method described by Sen Gupta (1970). Detection limit of the method is 100 ppm S. Quite a considerable number of the samples contained less than this amount and again, for purposes of calculation, a value of 100 ppm S has been given to these samples. Productivity of the method is 18 samples per man day.

INTERPRETATION OF THE DATA

In Table 4 statistical data for the 1079 samples classified into 61 localities and three groups are given. For each locality and for each group the mean, standard deviation and geometric mean are given for each of the elements Cu, Ni, Co, and S. Correlation coefficients for all possible combinations of these variables are also given. The correlation coefficients have been computed from logarithmically transformed data. Values for the discriminant equation best separating the barren from the ore ultramafics have been computed from the logarithmically transformed values of Cu, Ni and Co. This will be discussed in some detail later.

The data for each element at every locality are plotted in Figures 2-5. The plots are on a logarithmic scale with the interval for each 10^1 increment being constant from figure to figure. This allows the relative dispersion of the different elements to be visually compared. The geometric means of the elements at each locality are shown by an upward facing arrow. If two or more samples from one locality have the identical content of a given element, the '+' signs marking the second and succeeding samples with this element content have been successively incremented to the right of the figure. Thus a number of samples with the same content of an element (for example at the 0.01% detection limit for S) appear as a band stretching to the right of the given value. In Figure 6 the following ratios are plotted for each locality: $(\overline{Cu} + \overline{Ni} + \overline{Co})/\overline{S}$; $\overline{Cu}/\overline{Ni}$; $\overline{Co}/\overline{Ni}$; where $\overline{Cu}$, $\overline{Ni}$, $\overline{Co}$, and $\overline{S}$ are the arithmetic mean contents of these elements at each locality.

It is at once apparent from the data listed in Table 4 and shown in Figures 2-5 that there is a distinct enrichment of copper, nickel and sulphur, and to a lesser extent cobalt, in the ultramafic rocks associated with ore deposits, compared to barren ultramafic rocks. On each of Figures 2-5 a line has been drawn at an arbitrary position which best appears to separate the distribution of the sulphide component of the barren ultramafics from that of the ore bearing ultramafics. For the barren ultramafics, few samples contain a greater concentration than the value represented by this line. Conversely, for the ore bearing ultramafics, a substantial proportion of the samples contain a greater amount of one or more of these elements. For copper the line is drawn at $\log_{10}$ 2.00 (100 ppm Cu); for nickel at $\log_{10}$ 3.25 (1780 ppm Ni); for cobalt at $\log_{10}$ 2.20 (158 ppm Co); and for sulphur at $\log_{10}$-1.25 (0.178% S).

At this point it is necessary to digress in order to discuss the physico-chemical mechanisms which may have given rise to the above element values. In a basic or ultrabasic magma an immiscible sulphide liquid and/or sulphide crystals will separate when the solubility product of one or more of the different metal-sulphide combinations is exceeded. Until this occurs all of the metals and the sulphur are held in solution within the silicate liquid. The point at which sulphide separates from the silicate liquid is dependent on the relative amount of metals which are available to enter the sulphide fraction, upon the content of other components within the silicate melt, and upon temperature and pressure. However, the dominant, gross control on the separation of sulphide is the sulphur content of the magma. For magmatic segregation ore deposits of nickel-copper to form, an immiscible sulphide liquid or crystalline sulphides must separate at an early stage in the crystallisation of the silicate liquid, in order that these sulphides are not trapped between silicate crystals. This implies a high sulphur content for the silicate liquid whether it be the original magma or a differentiate of this magma. After the initial separation of sulphides the silicate liquid will still contain sulph which will continue to separate as sulphide liquid or crystals from the silicate liquid as the latter crystallizes. These later sulphides will tend to be dispersed through the resulting ultramafic rock because they are precipitated with and thus trapped and diluted by silicate crystals.

There are a limited amount of data on the solubility of sulphides in basic magmas. Wager et al (1957) for the Skaergaard intrusion estimate that copper-rich sulphides were separating at different times when the sulphur content of the magma ranged between approximately 100 to 300 ppm S. Later, iron sulphide separated when the silicate liquid contained approximately 600 ppm S. Skinner and Peck (1969) found that liquid and crystalline sulphides separated from a basaltic magma when that magma reached a content of 380 ppm S at a temperature of 1065°C.

Examining the data for sulphur given in Table 4 and in Figure 5, it may be seen that the mean content of sulphur in the BARREN class of ultramafic localities is mostly in the range 0.01 - 0.1% S. The mean sulphur content of the 616 samples from this group is .059% S and the geometric mean content 0.031% S. Relating these data to the above values for sulphide solubility in basic magmas, it appears that the majority of bodies classed as BARREN may have been too low in sulphur to allow early separation and segregation of sulphide crystals or liquid. For most of the bodies or localities classed as ORE the converse is true. Here the range in mean sulphur values is in the range 0.11 - 1.92% and the mean content for the 372 samples 0.582% S, almost exactly an order of magnitude higher than for the BARREN group.

It is pertinent here to ask how representative of the ultramafic bodies are these mean sulphur values, particularly for the ORE group. Fortunately, some of the bodies that are richest in sulphides are also among the most representatively sampled. The Werner-Gordon Lake belt mean value of 1.92% S and geometric mean of 1.09% S are derived from 29 samples spaced along the seven mile length of the belt. The values for the three ORE localities in Ungava (Katiniq, Raglan, and Expo) are derived from drill core or outcrop specimens that representatively sample these ultramafic sills from top to bottom, avoiding of course the ore zone at the base. In the case of the Katiniq locality we may compare our results for 16 drill core samples of 1.92% S arithmetic mean and 0.71% S geometric mean with a mean of

1.21% S and a geometric mean of 1.01% S for 37 serpentinite samples listed by Wilson et al (1969, Table 2). The latter samples come from a complete drill core section through the sill whose location relative to the core that we have sampled is not known. In compiling these data the basal massive sulphide zone has also been omitted The mean values derived from Wilson et al's data are likely to be more precise than that for the data reported here, since they have analysed the entire length of the core as composite samples of each 10 feet. It is therefore not surprising to find a much lower variation for their data - expressed as a small difference between the geometric and arithmetic means--compared to our analysis of 1/2 lb. samples taken at intervals of approximately 20 vertical feet. Considering the different parts of the sill were sampled by quite different sampling methods, the agreement for the two sets of data from Katiniq are excellent.

We may conclude that those ultramafic bodies that are associated with nickel-copper deposits are enriched in sulphur, often very considerably. For those deposits that have formed by magmatic segregation processes, this enrichment of sulphur in the parent ultramafic magma will have caused early separation and concentration of sulphides. It may be of significance that the small nickel deposits of the Porcupine area (Alexo, Texmont, Langmuir), where Naldrett (1966) has suggested formation of the nickel sulphides by sulphurization, together with other deposits from this general region (Sothman Township, Marbridge), are lower in sulphur than the average for the ORE group. The 157 samples that were taken from these five locations average 0.161% S, with a geometric mean of 0.064% S.

Enrichment of sulphur in the ORE group of samples is, of course, paralleled by an enrichment in one or more of the elements copper, nickel, and cobalt in the same samples. For most of the ORE localities the enrichment in nickel and cobalt is not very high in relation to the total nickel and cobalt content of typical ultramafic rocks (Turekian and Wedepohl, 1961: 2000 ppm Ni, 150 ppm Co, 10 ppm Cu). For copper this enrichment is often very considerable. This observation poses some interesting questions. Was this copper introduced with the sulphur either from an external source or by concentration from other parts of the primary magma? If it were not introduced with the sulphur what occurs during the crystallisation of sulphur deficient but copper enriched ultrabasic magmas? Can considerable volumes of copper-rich residual solution result, which then can migrate to deposit copper ores apparently unrelated to the ultramafic material? For the total set of samples, the correlation between copper and sulphur is, at 0.65 (Table 4), higher than the correlation of nickel or cobalt with sulphur at 0.49 and 0.59 respectively.

Although copper and nickel are so often associated in ore deposits derived from ultramafic rocks, their chemical properties are rather dissimilar. The very low correlation of 0.19 between copper and nickel is therefore not unexpected. Cobalt's correlation with copper of 0.40 and with nickel of 0.71 indicates that cobalt is intermediate in character between the other two elements, but more closely resembles nickel. For the ORE group the Co-Ni correlation rises to 0.89.

As might be expected from their low correlation, the Cu/Ni ratio varies widely from locality to locality. Where data are available, the correspondence between the Cu/Ni ratio of the ore deposits and the Cu/Ni ratio of the sulphides contained in the associated ultramafic rocks is generally close (Table 5, Figure 7). Only for the Eastern Metals Prospect and for the Strathy Township deposit are there gross differences between the pair of ratios. For these two deposits this may be reason enough to question the genetic relationship between the sulphides of the ore and the sulphides of the adjoining ultramafic rock. It may be seen from Figure 7 and Table 5 that there is a general tendency for the sulphides of the ultramafic rocks to have a lower Cu/Ni ratio than the associated ore body. At first sight this is surprising since the sulphides disseminated through the ultramafic rocks should, in part, have separated later than those that went to form the ore deposit. Chamberlain (1967) has shown, for the Muskox intrusion, that nickel-rich sulphides separate before nickel-copper sulphides. A possible explanation for this anomaly is that the disseminated sulphides of the ultramafic rock will have had a greater opportunity to re-equilibrate with nickel-rich,copper-poor silicates during serpentinizat than will the massive sulphides of the ore zone. During the re-equilibration processes nickel may become enriched in these disseminated sulphides because it is more chalcophile than iron, thus decreasing the Cu/Ni ratio. If this explanation is correct one might expect the BARREN ultramafics to have a lower Cu/Ni ratio than the ORE group, since enrichment of nickel by this mechanism will be relatively greater the lower the sulphide content. This is indeed the case. Based on the mean copper and nickel contents of the three groups, the Cu/Ni ratio for the ORE group is 0.234; for the MINORE group 0.070; and for the BARREN group 0.045. It should be pointed out that a non-geological factor will influence this ratio. As was explained in an earlier section, some nickel - hopefully small in amount - is leached from the silicate components of each sample. On average, this amount will remain constant over the range of sulphide contents dealt with here and will therefore contribute a built-in decrease in the Cu/Ni ratio of the samples as the sulphide content of the samples decrease.

If re-equilibration processes are significant in causing the ratio changes noted aobve, they should also influence other metal ratios. Slightly more chalcophile cobalt should be enriched relative to nickel in the low sulphide rocks. Both elements are available within silicates. Again this is the case. The Co/Ni ratio of the ORE group is 0.045, increasing to 0.052 for the MINORE group, and to 0.075 for the BARREN group. Another ratio which should change as a result of re-equilibration is the Ni/Fe ratio, increasing from the ORE group to the BARREN group.

Although iron has not been measured, we may examine the (Cu+Ni+Co)/S ratio. This increases from 0.41 in the ORE group to 0.53 in the MINORE group, to 1.10 in the BARREN group. A decrease in the amount of iron relative to copper, nickel, and cobalt will, in part, explain the increase in this ratio. Other causes are a loss in sulphur and the creation of sulphur-deficient mineral species such as heazlewoodite (Ni_3S_2), or of metal alloys such as awaruite (Ni_3Fe) which are at least partially soluble in the leach solution. The metals to form these alloys may have come from either sulphides or from silicates (Ramdohr, 1967).

A considerable amount of information of academic and economic interest is revealed by comparison of the metal contents and element ratios for the different localities as shown in Figures 2 to 6. It is not appropriate to detail such comparisons here. There are, however, some points which should be noted. For the BARREN group, samples with sulphur contents greater than 0.178% S (Figure 5) include some from Parry Sound - Huntsville, Malartic Township, Lac Bourbeau, Kenogamin Township, and the Thompson Belt (Hargrave River, Resting Lake, Bucko Barren Sill). The enrichment in the Thompson Belt is not surprising because of the abundance of nickel and copper sulphides in this belt. The Malartic ultramafic rocks were emplaced along a notably mineralized, major fault zone and the Lac Bourbeau samples come from an area of widespread copper mineralization. Minor pentlandite mineralization is known within the rocks sampled in Kenogaming Township. The Parry Sound-Huntsville samples are peculiar in having a very high content of leachable cobalt. The mean value for this locality is 99.1 ppm Co, which is three times greater than the mean for the whole group, and is exceeded only by three localities of the ORE group.

APPLICATION TO MINERAL EXPLORATION

Since the four element components which we have studied are enriched in the ore-associated ultramafic rocks compared to barren ultramafics, we may now consider how best to use this phenomenon to predict the ore potential of any given ultramafic body.

One of the primary considerations must be the analytical productivity for measuring the different elements. For the methods used in this study, the sample productivity to determine copper, nickel and cobalt is approximately five times greater than that of the method used to determine sulphur. Thus, provided little information is lost, the practicality of this method in mineral exploration is considerably enhanced if sulphur need not be determined. We have, therefore, first examined what contribution is made by copper, nickel and cobalt to discriminating between the ORE and the BARREN groups; we have then considered what additional information is contributed by sulphur.

The primary statistical method employed in this section is discriminant analysis. Discriminant analysis finds the best combination of variables to separate two or more groups of samples. In determining which of two groups a sample most resembles, one may compare the different variables one by one. It is, however, much easier and more efficient to combine these different variables into one measure (the discriminant score) for each sample, which may then be compared with the discriminant score of every other sample. Discriminant analysis is more efficient the greater is the number of variables to be compared. For the three or four variable to be compared here, the advantages to be derived from employing discriminant analysis are obviously much less than when twenty or thirty variables are to be compared.

The discriminant equations described below are computed to best distinguish the 616 samples of <u>BARREN</u> ultramafic rock from the 372 samples of the <u>ORE</u> group. The method of discriminant analysis has been particularly well described by Cooley and Lohnes (1962), and has previously been applied to multivariate geochemical data by Cameron (1969). The element values were first converted to logarithms because of the high variance of the data. The matrices W and A were then computed from the transformed data:

$$w_{ij} = \sum_{k=1}^{g} \left[\sum_{n=1}^{N_g} (\bar{x}_{ikn} - \bar{x}_{ik})(\bar{x}_{jkn} - \bar{x}_{jk}) \right]$$

$$a_{ij} = \sum_{k=1}^{g} N_g(\bar{x}_{ik} - \bar{x}_i)(\bar{x}_{jk} - \bar{x}_j)$$

where g is the number of groups, N_g the number of samples in group 'g' and i and j run from 1 to p, with p being the number of variables.

These matrices are then formed into the following equation, I being the identity matrix, and the eigenvectors and eigenvalues are extracted:

$$|W^{-1}A - \lambda I| = 0$$

The eigenvectors are the discriminant functions; for this two-group problem only one eigenvector is extracted. To test for a significant difference between the composition of two groups Λ is computed:

$$\Lambda = 1/(1 + \lambda)$$

where λ is the eigenvalue associated with the eigenvector that is the discriminant function. Lambda is tested by the F approximation of Rao (1952). For a two-group problem F is computed thus:

$$F_v^p = [(1 - \Lambda)/\Lambda]\,(v/p)$$

where $v = N - p + 1$, N is the number of samples, and p is the number of variables. Λ may also be computed as the ratio of within groups scatter to total scatter. Thus

Λ may be conveniently used to measure the discriminating power of different combinations of variables. The lower is the value of Λ, the greater is the discriminating power of these variables.

To show the relative contributions of the different variables to a discriminant equation a scaled discriminant vector may be obtained by multiplying the vector of discriminant coefficients by the vector of square roots of the diagonal of the W matrix.

Discriminating between the ORE and BARREN groups of samples using the variables copper, nickel and cobalt, the following values are obtained:

Discriminant Vector (1) 0.92 Cu, 0.37 Ni, 0.08 Co

Scaled Vector (1) 22.6 Cu, 6.85 Ni, 1.01 Co

$$\lambda = 0.42, \quad \Lambda = 0.70, \quad F^{3}_{984} = 137., \quad p < 0.0001$$

The F and p values indicate that there is a very high probability that the two groups are different. This information is almost superfluous, since this fact is visually observable in Figures 2 - 5. What is of more interest is that the scaled vector indicates that a considerable role is played by copper in distinguishing between the two groups, while cobalt plays a very minor role. That cobalt is of little help in discriminating between the two groups is not surprising. Comparing the plots for the four elements (Figures 2 - 5) or the group mean values (Table 4), cobalt shows the least relative difference between the ORE and the BARREN groups. Further, since it has moderate to high correlations with the other elements, much of t discriminating power of the data for cobalt is rendered redundant by these correlation

The very considerable difference between the ORE and the BARREN groups is also reflected in differences in their variance - covariance matrices (Table 6), which can be shown, by techniques described in Cooley and Lohnes (1962), to be markedly heterogeneous. Most multivariate analysis of variance and covariance methods and discriminant analysis methods assume a reasonable degree of homogeneity in the different variance - covariance matrices. Anderson and Bahadur (1962), have described a procedure for constructing a discriminant vector for unequal variance-covariance matrices. A Fortran coding of the procedure has been published by Reyment et al (1969). Using this method the following discriminant equation was obtained -

Discriminant Vector (2) 1.59 Cu, 0.63 Ni, 0.14 Co

Although, at first sight, this vector appears to be different from discriminant vector (1) above, converting the two vectors to percentage values shows that this is not so:

(1)	67 Cu,	27 Ni,	6 Co
(2)	66 Cu,	29 Ni,	5 Co

If we now include sulphur (as $\log_{10}$ ppm S) in the discriminant analysis the following values are derived:

Discriminant Vector (3) 0.51 Cu, 0.10 Ni, -0.18 Co, 0.84 S

Scaled Vector (3) 12.5 Cu, 1.87 Ni, -2.22 Co, 14.3 S

$$\lambda = 0.53, \quad \Lambda = 0.65, \quad F^{4}_{983} = 130., \quad p < 0.0001$$

The decrease in the value of Λ compared to the value associated with discriminant vector (1) indicates that a modest increase in the discriminating power of the data set is gained by including sulphur. If we again compute a discriminant vector by the method of Anderson and Bahadur (1962) the following coefficients are obtained:

Discriminant Vector (4) 1.04 Cu, 0.20 Ni, -0.37 Co, 1.75 S

Again, in terms of percentage values there is not a great deal of difference between discriminant vectors (3) and (4).

The value Λ has been computed for all possible combinations of variables:

Cu - Ni - Co - S	0.65
Cu - Ni - S	0.65
Cu - Co - S	0.65
Cu - S	0.65
Cu - Ni - Co	0.70
Ni - Co - S	0.70
Cu - Ni	0.70
Ni - S	0.70
Co - S	0.70
Cu - Co	0.71
Ni - Co	0.91

From this data it appears that copper and sulphur are of approximately equal value in discriminating between the <u>ORE</u> and the <u>BARREN</u> groups; that nickel is of lesser importance and that cobalt is of very little value. Copper and sulphur together are as powerful for discriminating between the groups as are all four elements combined.

The scalar discriminant score may be computed for each sample by multiplying a row discriminant vector by a column vector of element values (as $\log_{10}$ ppm in this case). This has been done for the sample data using discriminant vector (2). The discriminant scores are plotted on Figure 8 and the locality and group means are given in Table 4. In Figure 8 a line has been drawn at an arbitrary score value of 5.5 which best appears to separate the distribution of the <u>BARREN</u> group from the upper part of the distribution of the <u>ORE</u> group. On or to the right of this line score values are plotted as a larger cross. Mean values for the discriminant scores at each locality were computed and these are plotted as an upward facing arrow. It should be recalled that Figure 8 is essentially a logarithmic plot, since the element values have been transformed to logarithms prior to the discriminant scores being computed.

Only 16 of the 616 BARREN samples have discriminant score values equal or greater to 5.5. In contrast, a substantial proportion of the ORE group of samples have scores equal or greater to this value. On the basis of this plot many of the ore - associated ultramafic bodies could be identified as such, particularly those associated with greater quantities of ore grade material. The majority of MINORE localities more closely resemble those of the BARREN group. In practical exploration it would be worthwhile to follow-up any sample which gave a discriminant score greater than 5.5.

APPENDIX

THE DETERMINATION OF COPPER, NICKEL AND COBALT IN ROCKS BY ATOMIC ABSORPTION SPECTROMETRY USING A COLD LEACH

by

John J. Lynch

PRELIMINARY REMARKS

The method described in this paper is an application of the studies carried out by N. P. Smirnova, et al (1968) wherein certain trace elements occurring in rocks as sulphide minerals were preferentially dissolved by the action of hydrogen peroxide in the presence of ascorbic acid. The ascorbic acid lowers the pH of the solution to approximately 2.5 and provides a reducing medium which inhibits the oxidation of ferrous sulphate to ferric sulphate which in turn hydrolizes to hydrous ferric oxide. The ascorbic acid-hydrogen peroxide solution prepared as described below was found to decompose galena, arsenopyrite, chalcopyrite, pyrite, pyrrhotite, pentlandite, tetrahedrite, sphalerite, and awaruite.

Mixed standard solutions for the calibration of the atomic absorption spectrophotometer were initially prepared in the same concentration of ascorbic-acid hydrogen peroxide as was used for the decomposition of the samples. These standard solutions were found to be unstable over periods longer than three or four days. A fresh set of standard solutions contained in ascorbic acid and hydrogen peroxide were compared against a set of standard solutions contained in 1N HNO_3. There was no appreciable difference in percent absorbance readings between the standard solutions prepared in the two media. (Table 1). Standard solutions contained in 1N HNO_3 have been found to be stable for over a year and these are used for calibration.

TABLE 1

Comparison of Standard Solutions prepared in ascorbic acid-hydrogen peroxide and in 1 N HNO_3.

Concentration µg/ml	Cu, % Absorption		Ni, % Absorption		Co, % Absorption	
	H_2O_2 - Ascorbic Acid	1 N HNO_3	H_2O_2 - Ascorbic Acid	1 N HNO_3	H_2O_2 - Ascorbic Acid	1 N HNO_3
0.0	0.0	0.0	0.0	0.0	0.0	0.0
0.5	18.5	18.5	7.0	7.1	11.8	11.6
1.0	36.6	36.9	13.8	13.8	23.0	23.2
2.0	70.4	70.0	24.2	24.2	43.5	43.9
4.0	-	-	44.7	44.9	79.7	80.1

ANALYTICAL PROCEDURE

Decomposition of the Sample

1. Into a clean dry test tube (16 x 125 mm), calibrated at 10 ml., weigh a 100 mg sample of the ground rock. A larger sample weight may be used if low values are anticipated.
2. Add 7 ml. of ascorbic acid-hydrogen peroxide mixture and allow to stand approximately 18 hours (overnight) with occasional mixing. Do not heat.
3. Dilute to the 10 ml. mark with metal free water. Stopper the test tube with a clean cork and shake vigorously to mix.
4. Centrifuge for 5 minutes to obtain a clear supernatant solution.

General Procedure for Calibration of Instrument

1. The operating parameters described here refer to the Perkin Elmer Model 303 atomic absorption spectrophotometer. Other makes of instruments will require

changes in some of these parameters.

Parameter	Copper	Nickel	Cobalt
Meter Response	1*	1*	1*
Filter	out	out	out
Scale	x5	x2	x5
Slit	4 (7Å)	3 (2Å)	3 (2Å)
Source Current	15 ma	25 ma	30 ma
Range	U.V.	U.V.	U.V.
Wavelength	3247 Å	2320 Å	2407 Å
Burner	10 cm Single slot	Boling	Boling
Air Pressure	30 psi	30 psi	30 psi
Air Flow	9*	9*	9*
Acetylene Pressure	8 psi	8 psi	8 psi
Acetylene Flow	8*	7½*	7½*

* NOTE: Arbitrary units given by Perkin-Elmer

2. Aspirate 1N HNO_3 into the flame and zero the instrument.
3. Using the parameters listed above, the following standard solutions are used for calibration:

Cu: 0.5, 1.0 and 2.0 μg/ml
Ni: 0.5, 1.0, 2.0, 4.0, 6.0 and 8.0 μg/ml
Co: 0.5, 1.0, 2.0, 4.0 μg/ml

4. Calibration curves are plotted relating percent absorption and μg/ml.

General Procedure for Analysis of samples

1. After calibrating, aspirate 1N HNO_3 and readjust zero if necessary.
2. Aspirate sample solutions and record percent absorption. Care should be taken not to suck any of the sediment into the burner. Aspirate 1N HNO_3 between each sample and check a calibration standard after every tenth sample. If check standard changes more than 1 percent absorption, recalibrate and repeat previous ten samples.
3. Using calibration curves, interpolate percent absorption of samples to obtain µg/ml. Calculate the concentration in parts per million using the equation:

$$\text{ppm element} = \frac{\mu g/ml \times 10}{\text{sample weight (gm.)}}$$

4. Should the sample be above the top standard of the calibration curve, an aliquot of the sample solution may be diluted to 10 ml with the appropriate volume of ascorbic-acid-hydrogen peroxide mixture and metal free water. Alternatively the sensitivity of the instrument may be lowered by reducing the scale expansion and/or using a smaller burner. This permits use of standards up to considerably higher concentrations.

Preparation of Reagents

1. <u>1% Ascorbic Acid solution</u>: Dissolve 5.0 gm ascorbic acid in metal-free water and dilute to 500 ml with metal free water. This solution should be prepared freshly just prior to using.
2. <u>Ascorbic acid-hydrogen peroxide mixture</u>: Add 200 ml 30% hydrogen peroxide to 500 ml ascorbic acid and mixthoroughly. This solution is made up freshly each d

3. Mixed copper, nickel and cobalt standard solution: 1000 µg/ml. (Solution A) In a 1000 ml volumetric flask, dissolve 3.9297 gm copper sulphate ($CuSO_4 .5H_2O$), 4.0487 gm nickelous chloride ($NiCl_2.6H_2O$) and 4.0372 gm cobaltous chloride ($CoCl_2.6H_2O$) in about 900 ml of metal free water. Add 6 ml concentrated nitric acid mix and allow to cool to room temperature. Dilute to 1000 ml with metal free water and mix well. This solution is about 0.1N with respect to nitric acid.

4. Mixed copper, nickel and cobalt standard solution: 100 µg/ml (Solution B). Pipett 100 ml of solution A into a 1000 ml volumetric flask. Add 5.4 ml concentrated nitric acid, dilute to about 900 ml with metal-free water, mix and allow to cool to room temperature. Dilute to 1000 ml with metal free water and mix well. This solution is about 0.1N with respect to nitric acid.

5. Mixed copper, nickel and cobalt calibration solutions:

These are prepared according to the dilutions listed in Table II

TABLE II

Concentration (µg/ml)	Volume of Solution A(ml)	Volume of Solution B(ml)	Volume of Concentrated HNO_3(ml)	Final Volume (ml)
0.5	-	1	12.5	200
1.0	-	2	12.5	200
2.0	-	4	12.5	200
4.0	-	8	12.5	200
6.0	-	12	12.5	200
8.0	-	16	12.5	200
10.0	-	20	12.5	200
15.0	3	-	12.5	200
20.0	4	-	12.5	200
25.0	5	-	12.5	200
30.0	6	-	12.5	200
35.0	7	-	12.5	200
40.0	8	-	12.5	200

6. IN HNO_3: Dilute 125 ml concentrated HNO_3 to 2000 ml with metal-free water. Store in a polyethylene bottle.

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McWatters Gold Mines Ltd.
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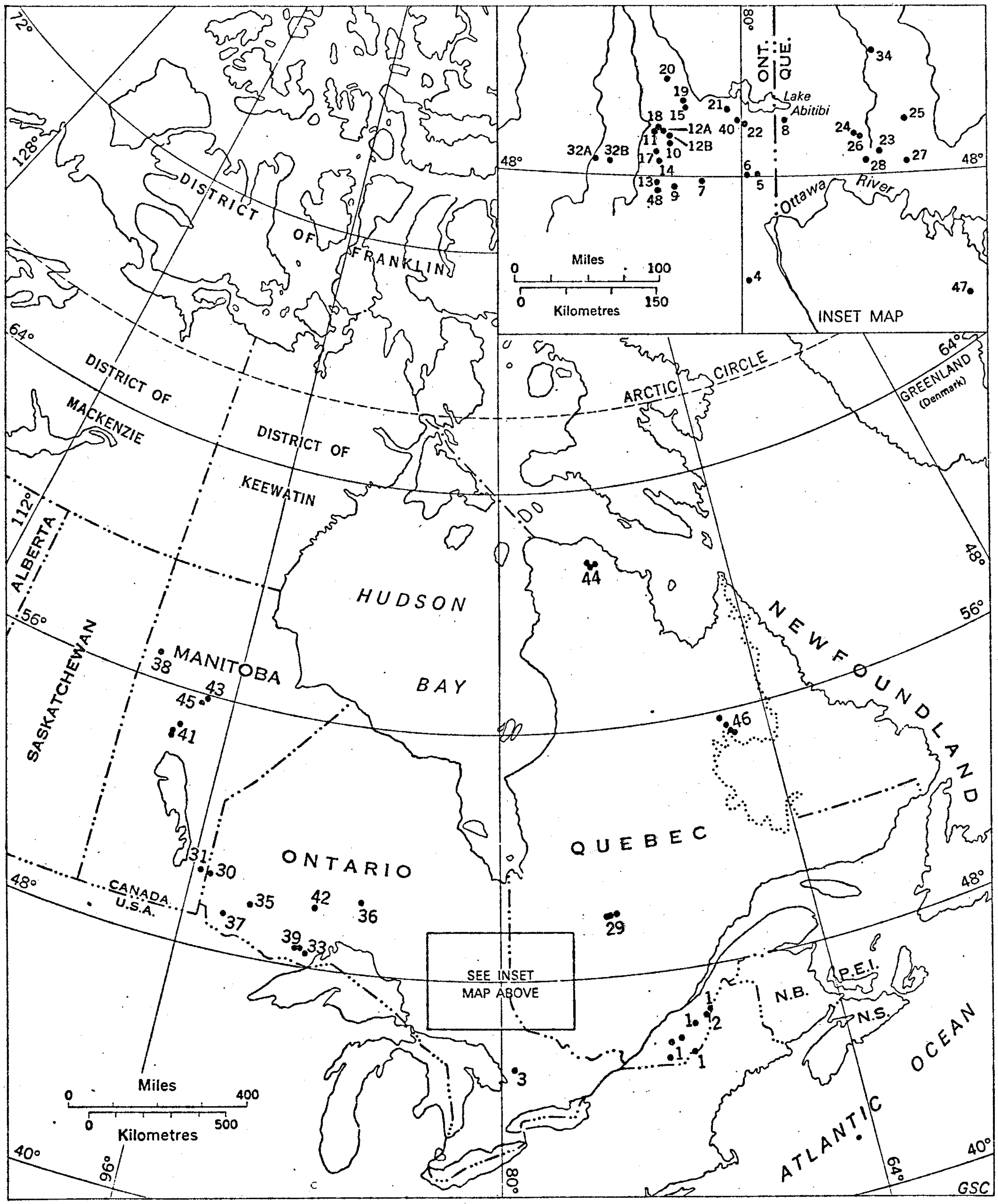
FIGURES

Figure 1. Map showing sample locations.

(Cameron et al)



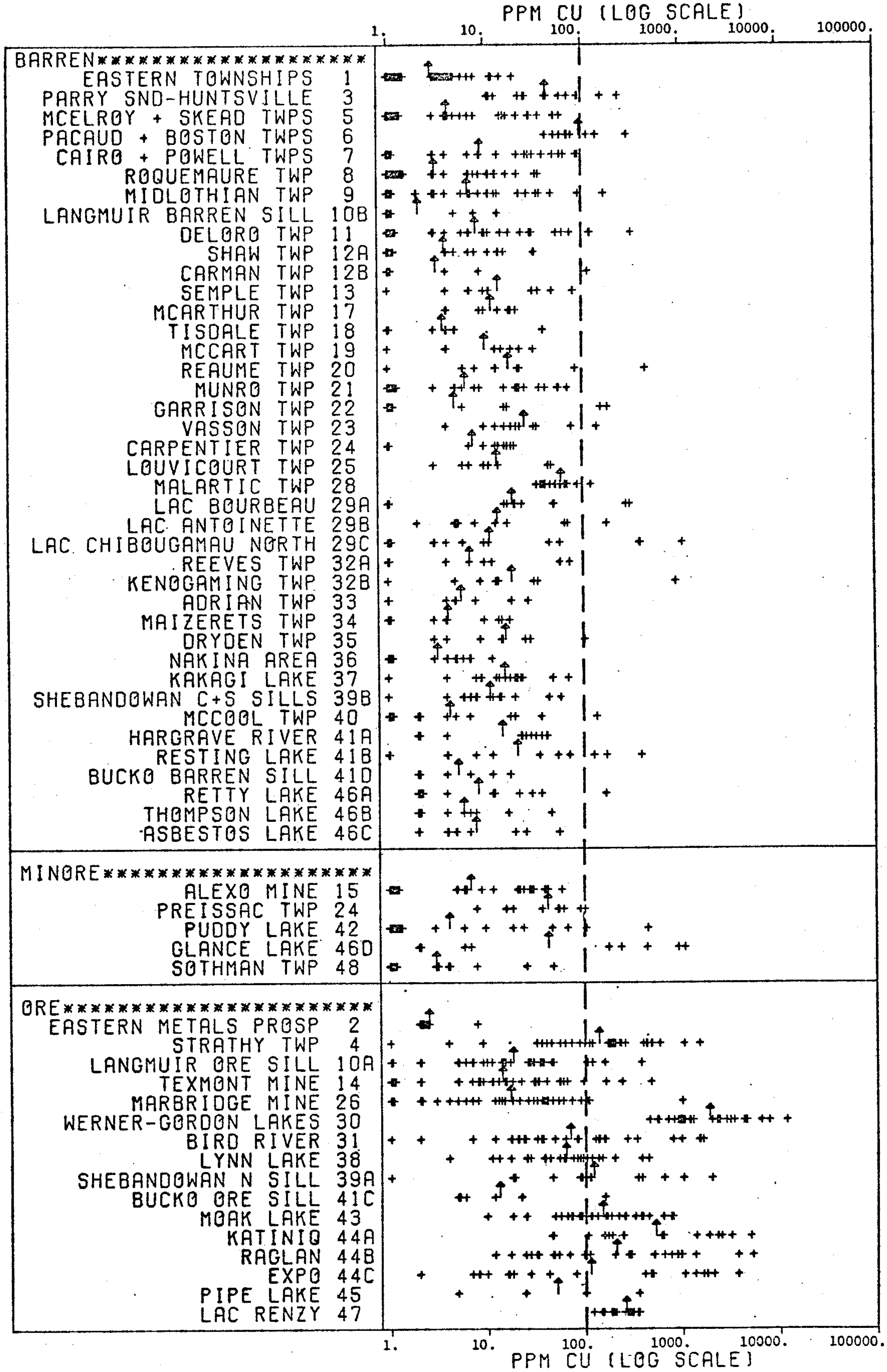


Figure 2. Distribution of copper as ascorbic acid - hydrogen peroxide soluble metal.

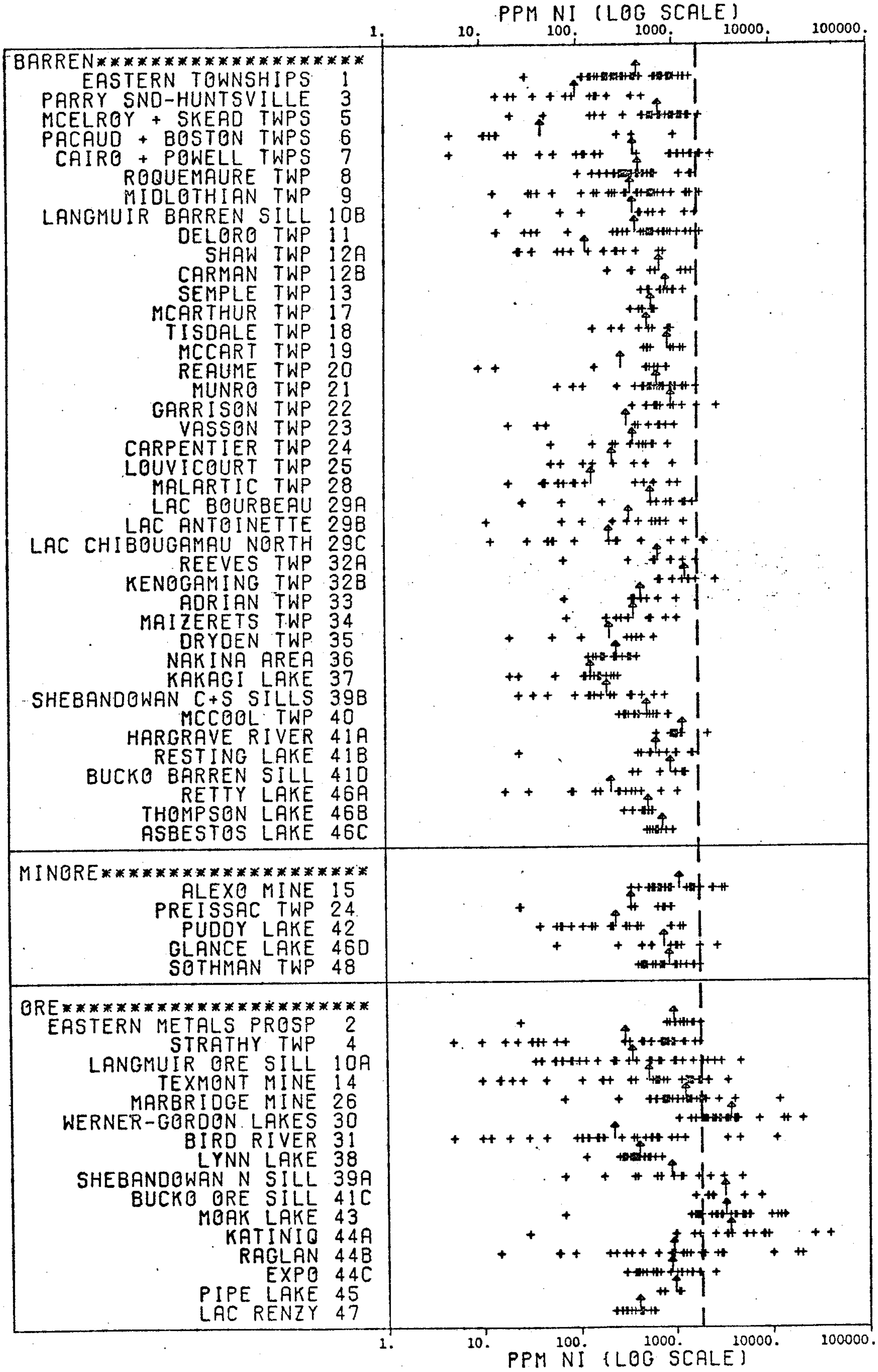


Figure 3. Distribution of nickel as ascorbic acid - hydrogen peroxide soluble metal.

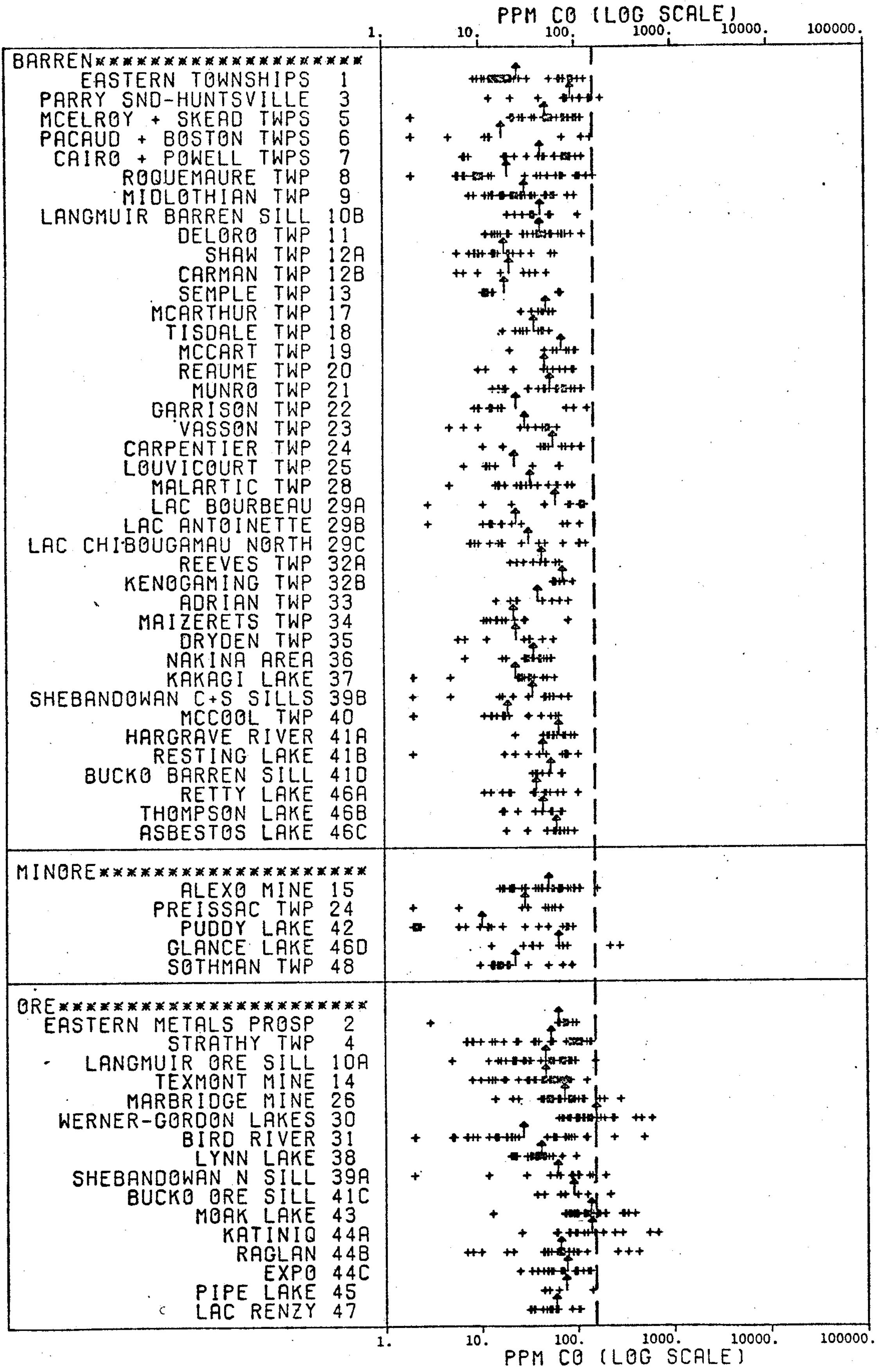


Figure 4. Distribution of cobalt as ascorbic acid - hydrogen peroxide soluble metal.

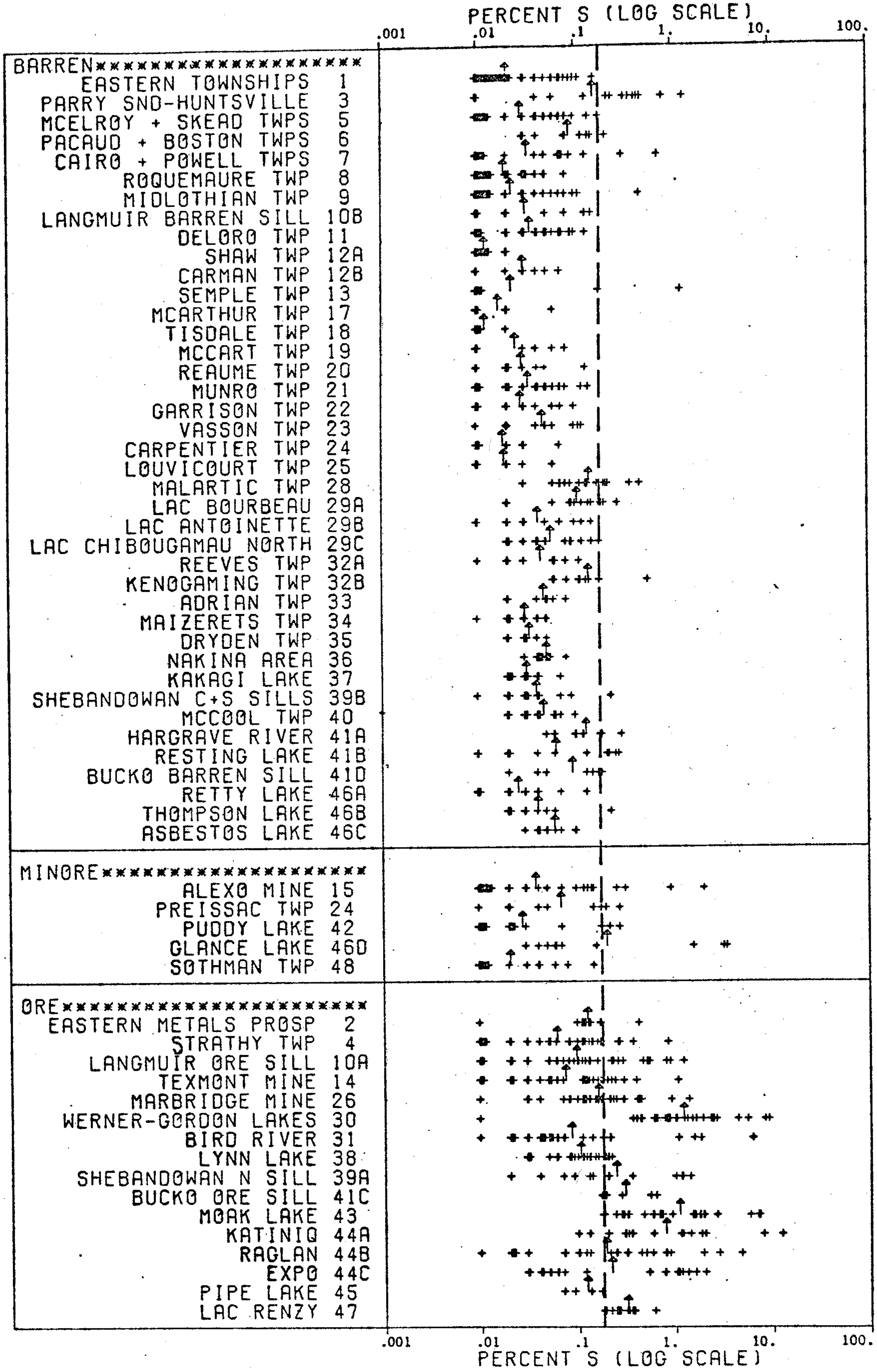


Figure 5. Distribution of sulphur.

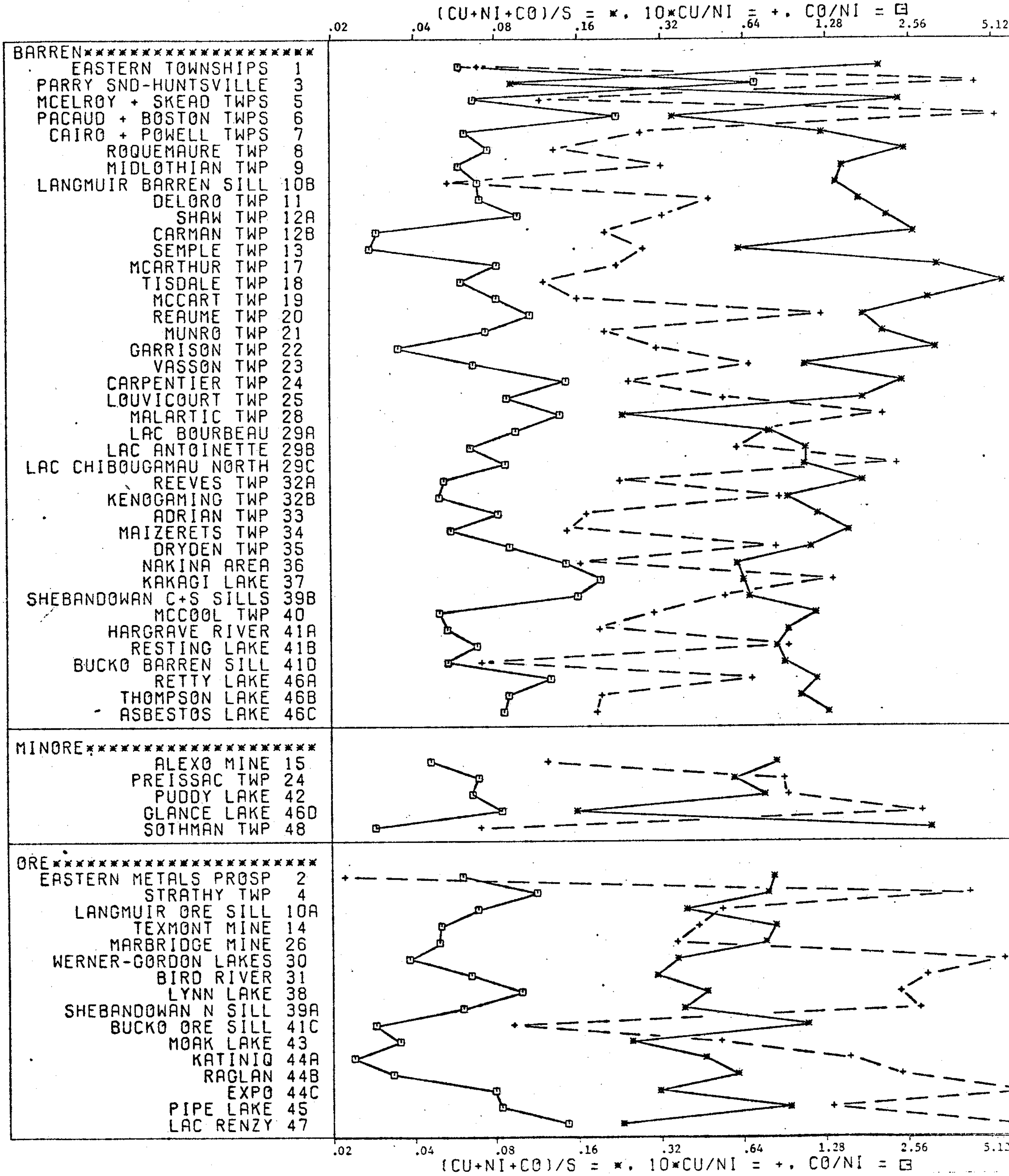


Figure 6. Distribution of element ratios [* = (Cu + Ni + Co)/S; + = Cu/Ni x 10; □ = Co/Ni].

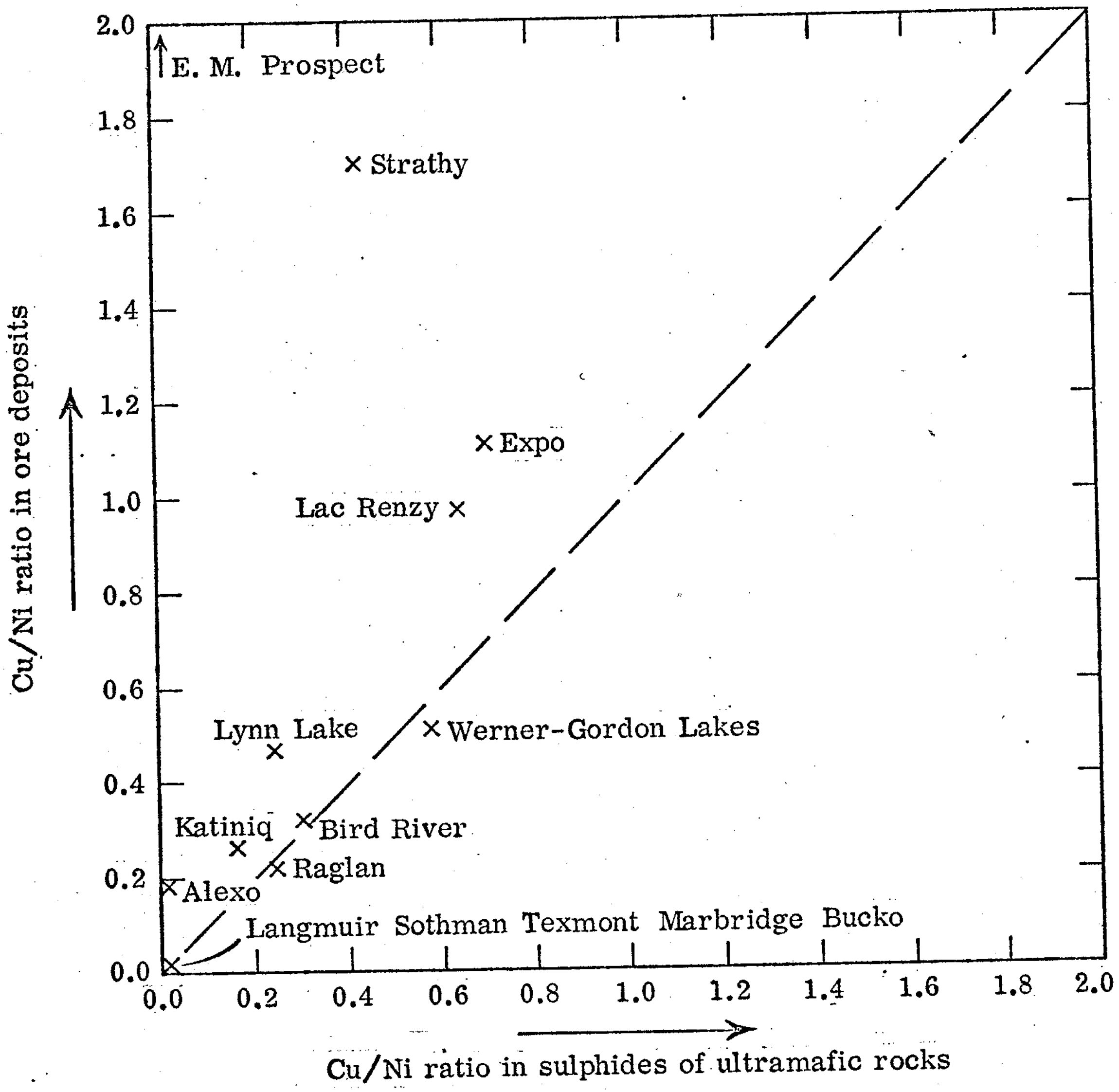


Figure 7. Comparison of Cu/Ni ratio of ore deposits with the Cu/Ni ratio of sulphides in associated ultramafic rocks.

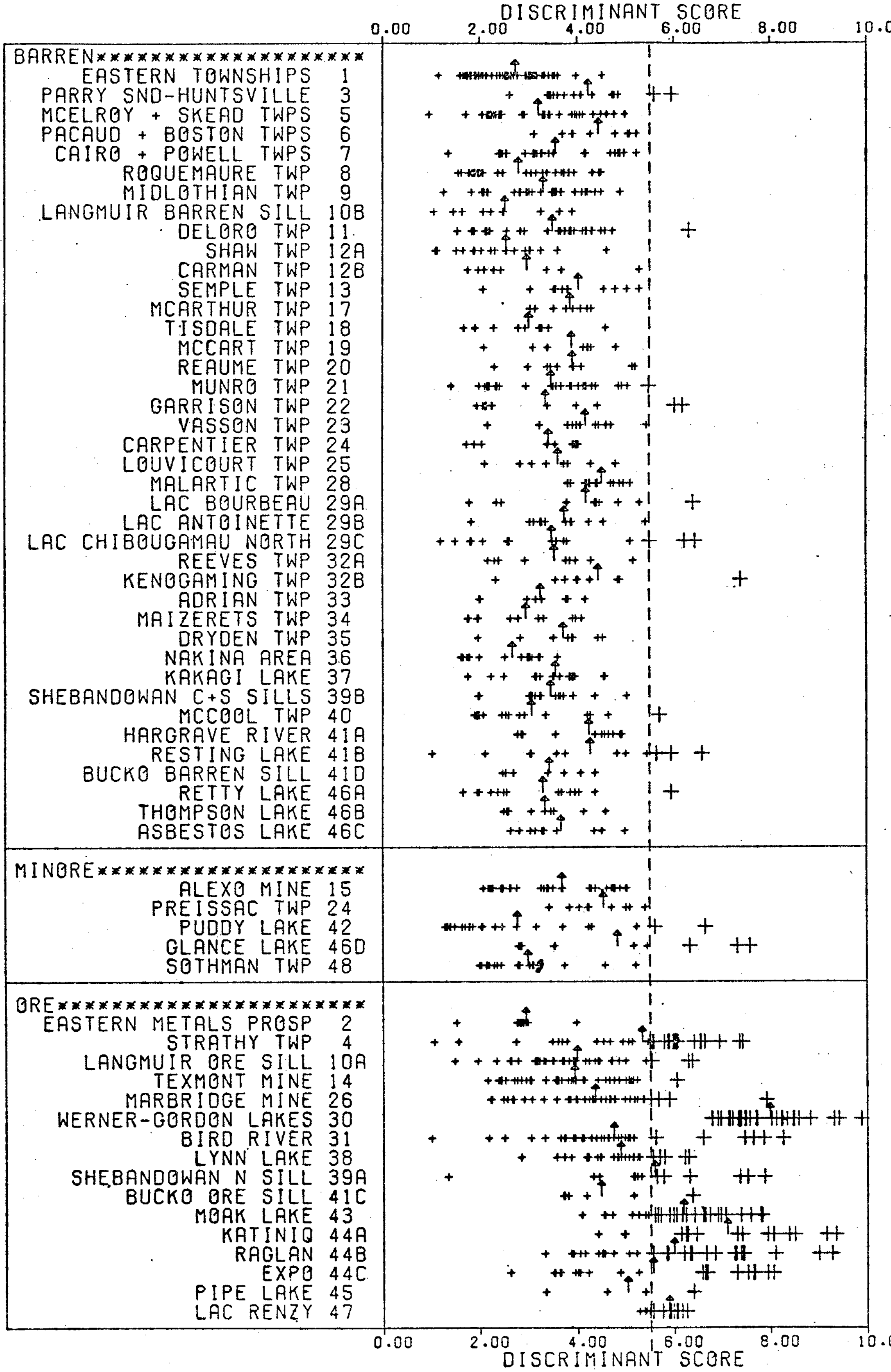


Figure 8. Distribution of discriminant scores computed from copper, nickel, and cobalt.

TABLES

Table 1. Sampling data and geological characteristics for ultramafic rocks.

Table 2. Size of nickel or nickel/copper deposits within or adjoining sampled ultramafic rocks. Upper five localities with less than 5000 tons Ni + Cu classified as MINORE, all others classified as ORE.

Table 3. Ascorbic acid - hydrogen peroxide leaching of silicate minerals.

Table 4. Chemical data for localities and groups of ultramafic rocks. Data for copper, nickel and cobalt is as ascorbic acid - hydrogen peroxide soluble metal.

Table 5. Comparison of Cu/Ni ratio of ore deposits with the Cu/Ni ratio of sulphides in associated ultramafic rocks.

Table 6. Variance - covariance matrices for BARREN and ORE groups of ultramafic rocks.

MINERALS	Complete Decomposition (HF)			Ascorbic Acid-H_2O_2 Leach		
	Cu ppm	Ni ppm	Co ppm	Cu ppm	Ni ppm	Co ppm
Olivine, Duke Island, Alaska	3	695	173	2	26	7
Olivine, Aiken Lake, B.C.	9	2,000	125	5	66	5
Clinopyroxene, Muskox, N.W.T.	7	246	38	3	12	1
Orthopyroxene, Muskox, N.W.T.	33	493	83	30	12	0

Table 3. Ascorbic acid - hydrogen peroxide leaching of silicate minerals.

MAP CODE	LOCALITY	THOUSANDS OF TONS RESERVES AND/OR PRODUCTION (1)			MEAN CONTENT H_2O_2 SOLUBLE ELEMENT IN U.M. ROCKS OF LOCALITY (2)		
		Cu	Ni	Cu/Ni	Cu	Ni	Cu/Ni
15	Alexo Mine	0.4	2.2	0.18	15.2	1208.	0.01
48	Sothman Twp.	-	2.7	Low	6.3	884.	<0.01
2	E. M. Prospect	14.5	4.9	2.96	2.5	1125.	<0.01
4	Strathy Twp.	4.8	2.8	1.71	251.	587.	0.43
10A	Langmuir Ore Sill	-	6.9	Low	40.	742.	0.05
14	Texmont Mine	-	37.	Low	43.5	988.	0.04
26	Marbridge Mine	-	14.5	Low	57.5	1570.	0.04
30	Werner-Gordon Lks.	7.6	14.9	0.51	2500.	4380.	0.57
31	Bird River	4.6	14.3	0.32	263.	882.	0.30
38	Lynn Lake	135.	287.	0.47	94.7	396.	0.24
39A	Shebandowan N.	?	?	?	380.	1350.	0.28
41C	Bucko Ore Sill	-	320.	Low	31.	3330.	<0.01
43	Moak Lake	?	?	?	221.	4180.	0.05
44A	Katiniq	27.	104.	0.26	1180.	7570.	0.16
44B	Raglan	26.	116.	0.22	651.	2690.	0.24
44C	Expo	42.	38.	1.11	669.	960.	0.70
45	Pipe Lake	?	?	?	122.	895.	0.14
47	Lac Renzy	7.1	7.3	0.97	247.	386.	0.64

(1) From Table 2. (2) From Table 4.

Table 5. Comparison of Cu/Ni ratio of ore deposits with the Cu/Ni ratio of sulphides in associated ultramafic rocks.

TABLE 6.

	BARREN GROUP				ORE GROUP			
	Cu	Ni	Co	S	Cu	Ni	Co	S
Cu	.46	-.07	.05	.11	.85	.23	.16	.39
Ni	-.07	.27	.11	.05	.23	.46	.24	.31
Co	.05	.11	.15	.07	.16	.24	.16	.18
S	.11	.05	.07	.20	.39	.31	.18	.46

TABLE 6. Variance - covariance matrices for BARREN and ORE groups of ultramafic rocks.

e following items have been placed on Open File for inspection at the library of the ological Survey of Canada, 601 Booth Street, Ottawa 4.

F 34 Geology of Kekeko Hills and southern portion of S.W. quarter of Beauchastel Township, Temiscaminque County, Quebec, by W.G.Q. Johnston, 1957. 71 pages, 2 figures and 1 map, scale 1" to 1000'. Part of N.T.S. 32 D/3.

F 35 Distribution of ore elements in rocks for evaluating ore potential: Nu, Cu, S in ultramafic rocks of the Canadian Shield, by E.M. Cameron, G. Siddeley and C.C. Durham with an appendix on the determination of Cu, Nu, Co in rocks by atomic absorption spectrometry using a cold leach, by John J. Lynch (Presented at 3rd Int. Exploration Geochemical Symposium 1970). 32 pages, 8 figures, 7 tables.

OK