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GEOLOGICAL SURVEY OF CANADA

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PAPER 71-35

THREE GEOCHEMICAL STANDARDS OF SULPHIDE-BEARING ULTRAMAFIC ROCK: U.M. 1, U.M. 2, U.M. 4

(Report and 3 tables)

compiled by E. M. Cameron



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ABSTRACT

Large quantities of three samples of sulphide-bearing ultramafic rock have been collected; two from the Werner Lake-Gordon Lake district of northwestern Ontario and one from the Giant Mascot Mine at Hope, B.C. The latter sample is a peridotite; the other two are serpentinized hornblende metaperidotites. All three samples have been crushed, ground, and thoroughly homogenized before being analyzed for a number of elements.

The primary purpose of the samples is to provide standards for the determination of S and ascorbic acid-hydrogen peroxide soluble Cu, Cu, Ni, and Co in ultramafic rocks. Analysis for these components forms the basis of a geochemical method for evaluation of the ore potential of ultramafic rocks. Repeated determinations of the latter three components using a two-way analysis of variance design shows that each of the samples is homogeneous for these components. The samples may also prove to be useful standard material for other elements, such as chromium and the platinum metals. They have been analyzed for Si, Ti, Al, Fe, Mn, Mg, Ca, Na, K, P, H2O, CO₂, Cr, Zn, and total Cu, Ni, and Co in addition to the components listed above. Bottles of each of the standards U.M. 1, U.M. 2, and U.M. 4 are being distributed by the Canadian Standard Reference Materials Project.

RÉSUMÉ

Trois sortes d'échantillons de roche ultramafique sulfurée ont été recueillis en grande quantité. Deux proviennent du district des lacs Gordon et Werner du nord-ouest de l'Ontario, et le troisième de la mine Giant Mascot à Hope, en Colombie-Britannique. Ce dernier est un échantillon de péridotite; les deux autres sont constitués de métapéridotites de hornblende serpentinisées. Ces trois échantillons ont été concassés, broyés et parfaitement homogénéisés avant d'être analysés relativement à un certain nombre d'éléments.

L'objectif initial de l'étude de ces échantillons est d'obtenir des normes permettant de déterminer leur teneur en S et en Cu, Ni et Co, solubles en acide ascorbique et en peroxyde d'hydrogène. L'analyse de ces composants constitue la base d'une méthode géochimique d'évaluation du potentiel minéral des roches ultramafiques. Des analyses répétées de la présence des trois derniers composants (Cu, Ni et Co), à l'aide d'une méthode en deux étapes (analyse et preuve par l'inverse) indiquent que chaque échantillon a une teneur homogène de ces composants. Ces échantillons peuvent également être utiles en tant qu'éléments de normalisation pour d'autres éléments, tels que le chrome et le platine. On a analysé leur teneur en Si, Ti, Al, Fe, Mn, Ca, Na, K, P, H₂O, CO₂, Cr, Zn, et leur teneur totale en Cu, Ni et Co, outre les composants précités. Des flacons contenant chacun des échantillons-normes, U.M.1, U.M.2 et U.M.4 sont distribués par le Canadian Standard Reference Materials Project.

INTRODUCTION

Recently, a geochemical method for evaluating the ore potential of bodies of ultramafic rock has been described (Cameron et al., 1971). It was shown that samples taken from bodies that are associated with Ni-Cu sulphide ores are richer in S, and in the sulphides of Cu, Ni, and Co, than are samples from barren ultramafic rocks. In order to measure the amount of these metals present as sulphides in the samples, it was necessary to use a selective leach technique (Lynch, 1971) which dissolved the sulphides, but did not remove any of these metals present in the rock as a solid solution component of silicate minerals.

Such selective leaching techniques are much more subject to interlaboratory analytical bias than are analytical methods designed to measure the total element content of rocks. This is because selective leaching rarely makes a perfect separation between the different components of the samples. In the case of the method used in this work, at least some of the silicate component of the metals is dissolved along with the sulphide fraction. Variation in the strength of the leaching solution or in the conditions under which leaching is carried out between laboratories or from day to day within the same laboratory may markedly influence the amount of metal dissolved from a given sample.

In the practical application of these techniques to exploration, if valid comparisons are to be made between the data obtained from different laboratories, it is important that the various sets of analyses are not analytically biased with respect to each other. To help achieve this we have prepared and have analyzed large quantities of a number of mineralized ultramafic rocks which will serve as standards to calibrate the results of different laboratories. They will initially serve as a reference to the data on S, Cu, Ni, and Co published by Cameron <u>et al.</u> (1971). They may eventually prove to be useful standards for other components, such as chromium and the platinum group of metals.

Four 150-pound samples of ultramafic rock were collected, two from the Giant Mascot Mine at Hope, British Columbia, and two from outcrops in the Werner Lake-Gordon Lake district of northwestern Ontario. These were crushed, ground, sieved, and homogenized in the laboratories of the Mines Branch of the Department of Energy, Mines and Resources. The material was stored in 130-gram bottles. Eight of these bottles, chosen at random for each of the four samples, were repeatedly analyzed for ascorbic acid-hydrogen peroxide leachable Cu, Ni, and Co. The resulting data have been examined by analysis of variance techniques to estimate the homogeneity of the material between bottles and to measure the day-to-day and within-day analytical variaance. Sulphur; total Cu, Ni, and Co; and the total content of certain other major and trace components have also been determined, but less exhaustively. Two of the samples (from the Giant Mascot Mine) resembled each other quite closely in Cu and Ni contents. One of these samples has, therefore, been discarded. The following description will be for the three samples that were retained.

Original manuscript submitted, December 1,1971 Approved for publication, December 17, 1971 - 2 -

A number of persons have taken part in the collection, preparation, and analysis of these samples. Their contribution is listed below. The assistance of the managements of Giant Mascot Mines Ltd., and of Consolidated Canadian Faraday Ltd. in obtaining the sample material is gratefully acknowledged.

Sample collection: M. Larose.

- Sample preparation: R.P. Bailey, D.E. Pickett, L.E. Shaheen, (Mines Branch); C.C. Durham.
- Ascorbic acid-hydrogen peroxide leachable metals: J.J. Lynch, Mrs. A. MacLaurin.
- Atomic absorption analysis for total Cu, Ni, Co, Zn (A.A.S.1, in Table 2): J.J. Lynch, Mrs. A. MacLaurin.

Various determinations (A.A.S.2, Table 2; Codes 2-5, Table 3): S. Abbey, J.L. Bouvier.

- Direct-reading emission spectrometry: R. Horton.
- Description of thin sections: T.N. Irvine.
- Description of polished sections: C.R. McLeod,

Analysis of variance: E. M. Cameron, D. Hobbs.

Bottles containing approximately 130 grams of each of the three standards may be obtained at a cost of C. \$45.00 per set, including cost of delivery by air, from:

> Dr. A. H. Gillieson, Canadian Standard Reference Materials Project, Mineral Science Division, Mines Branch, Department of Energy, Mines and Resources, 555 Booth Street, Ottawa K1A 0E8, Ontario.

SAMPLE COLLECTION

Sample U. M. 1 (lat 49° 28'; long. 121° 30') This sample was taken from an outcrop of peridotite, with disseminated sulphides, immediately north of the main shaft of the Giant Mascot Mine at Hope, British Columbia. The outcrop is part of a large peridotite stock, about 1.5 square miles in area.

Sample U. M. 2 and U. M. 4 These samples were taken from small lenticular masses of peridotite that occur along a major east-west fault zone in the Werner Lake district of northwestern Ontario. Sample U. M. 2(lat. 50° 27'; long. 94° 52') was taken from a small mineralized intrusion beside the mouth of a creek that flows from the north into the eastern end of Werner Lake. Sample U. M. 4 (lat. 50° 28'; long. 95° 01') was taken from a surface outcrop in the immediate vicinity of the shaft on the northwest corner of an island on Almo Lake, to the west of Werner Lake.

SAMPLE PREPARATION

The samples were kindly prepared by the Mines Branch of the Department of Energy, Mines and Resources. Large, unwieldy blocks of sample were first broken to fist-size pieces with a sledge hammer. The sample was then processed to 1/4-inch chips by a "Chipmunk" crusher with steel jaws and then to a coarse powder by a vertical steel cone grinder. The sample was then split into 25-pound charges and each was rolled in a steel ball mill for five hours. The powder from the ball mill was sieved to -200 mesh through a continuous feed vibrating steel screen, and the oversize returned for ball milling. Eventually, all of the sample was reduced to -200 mesh. The entire sample, of approximately 150 pounds, was then loaded into a rotating steel cone drum blender with mixing blades. It was rolled for eight hours to completely homogenize the sample. The powder was then sampled and bottled immediately from the drum. All of the ultramafic samples were treated identically and quite separately.

During sample preparation some iron and alloy element contamination was introduced, particularly from the steel grinding balls. Minor zinc contamination may have been introduced when the coarse sample chips passed through a galvanized metal sample collector.

DESCRIPTION OF MINERALS EXAMINED IN THIN SECTION

Sample U. M. 1 This rock is a peridotite consisting of 80-85% olivine, 3-5% orthopyroxene (bronzite), 5% augite, 5% pale yellow hornblende, 5-10% sulphides, and 1% chromite. The rock has a cumulate-type texture, with closely packed, subhedral grains of olivine surrounded by interstitial to slightly poikilitic pyroxenes, hornblende, and sulphides. Chromite occurs as small, euhedral grains, some as inclusions in the olivine. There appears to be little alteration of the rock.

Sample U. M. 2 This distinctly banded rock originally consisted of about 30% olivine, 10% hypersthene, 45% hornblende, 5% semi-opaque olive-green spinel, and 5% opaques, mainly sulphides. The olivine is now mostly altered to greenish yellow serpentine, with veinlets of secondary magnetite. The pyroxene and the hornblende grains tend to be elongated parallel to the banding and, together with the olivine, show mutual interference textures. The overall fabric and mineral assemblage of the sample indicate relatively high grade (amphibolite facies) metamorphism of a primary ultramafic rock, perhaps a picrite. The sample is best classified as a serpentinized hornblende metaperidotite.

Sample U. M. 4 The rock was originally composed of about 30% olivine, 55% pale hornblende, 10% hypersthene, 5% dark olive-green spinel, and minor sulphides. The olivine is now completely altered to pale serpentine; secondary magnetite is present, but is comparatively rare; and a small amount of talc is present. The olivine was apparently granular; the pyroxene is somewhat poikilitic; the spinel grains are commonly almost spherical; and the hornblende occurs in fine grains with mutual interference boundaries. There is no marked preferred orientation of the minerals, or conspicuous banding. Like the previous sample this rock has formed by high grade metamorphism of a

primary ultramafic rock. It may similarly be classified as a <u>serpentinized</u> hornblende metaperidotite.

DESCRIPTION OF OPAQUE MINERALS EXAMINED IN POLISHED SECTION

Sample U. M. 1 Sulphides commonly occur as elongated, multiphase, grains up to 1 mm in length that are usually irregular in shape and are interstitial to the silicate minerals. Pyrrhotite is the most common constituent of these grains, usually with associated pentlandite that occurs as subhedral grains, or as flame-type inclusions, or as intergrowths that range in size from a few microns to nearly 0.5 mm. Many of the grains of pyrrhotite and pentlandite also contain some chalcopyrite. There are some grains composed of chalcopyrite-pentlandite, with no pyrrhotite, but these are rare. Grains of monominerallic sulphide are, most usually, chalcopyrite. This mineral also occurs as a fracture filling within silicates or interstitially between them. Sulphide grains 2-3 microns in diameter are present, but are rare. They lie within the silicates or within chromite. Their composition could not be determined by optical methods. However, composite grains of pentlanditechalcopyrite, less than 10 microns in diameter, were observed.

Chromite is the most common oxide; it occurs associated with the sulphides, or interstitial to the silicates, and as inclusions up to a few microns in diameter within silicates. The maximum grain size is about 0.1 mm. Magnetite is present, associated with the chromite, but it is rare.

Sample U. M. 2 The sulphides are disseminated through the sample, commonly interstitial to the silicates. The maximum grain size of the sulphides is about 0.5 mm. These grains consist of intimately associated violarite, pyrrhotite, chalcopyrite, and pyrite. Both the pyrite and pyrrhotite show evidence of alteration to marcasite and goethite, and violarite presumably has replaced pentlandite. Many of the sulphide grains have associated magnetite, commonly as crosscutting veinlets or as fracture fillings. Sulphide grains a few microns in diameter, and too small to be identified optically, occur disseminated within the silicates, but this occurrence is relatively rare.

Magnetite is commonly found as inclusions within chromite grains and as veinlets and fracture fillings in chromite and the sulphides. It also occurs as thin wispy films between the silicates and along fracture planes within them. Individual chromite grains are up to 1 mm in diameter, and as well as containing the abundant inclusions of magnetite noted above also contain some multiphase sulphide inclusions. Most of the chromite shows evidence of fracturing, with thin sulphide inclusions along, or at the junction of fractures; hence they should be readily released by crushing and grinding.

Sample U. M. 4 Sulphide grains, that range up to 1 mm in length, are generally interstitial to the silicates, and are concentrated primarily in a band 3-5 mm wide that crosses the section. Magnetite shows a close association with the sulphides, apparently the result of replacing the latter. The sulphides present are violarite, chalcopyrite, pyrite, pyrrhotite, pentlandite, and marcasite, with the violarite having replaced pentlandite, and the marcasite having replaced the pyrite and pyrrhotite. Tiny specks of sulphide, up to a few microns in diameter, lie within silicate minerals, commonly along grain boundaries of the silicates. The section examined has one cluster of about 20 chromite grains, plus a few other isolated grains. They range up to 1.3 mm in size and a few contain sulphide inclusions, some of which are only a few microns in diameter.

ANALYSIS OF ASCORBIC ACID - HYDROGEN PEROXIDE SOLUBLE COPPER, NICKEL AND COBALT

For this analytical method it is desirable to estimate the short term, or within batch, error variance; and the long term, or between batch, variation. For a material to be used as a chemical standard it is essential to estimate its homogeneity. These three conditions have been measured for each element in all the samples by repeatedly analyzing this material. This has been done in a fashion that allows the interpretation of the data by a two-crossed classification, with replication, analysis of variance design (Bennett and Franklin, 1954, pp. 368-379):

$$x_{ija} = \mu + \xi_i + \eta_j + \lambda_{ij} + \epsilon_{ija}$$

For each element in each standard there are determinations, x_{ija} , where i = 1, 8 different bottles of each standard; j = first and second batches; and a = first and second replicates in each batch/bottle cell. In the above equation ξ_i is the variation due to sample inhomogeneity between bottles; N_j is the analytical variation or bias between batches; λ_{ij} is the interaction effect measuring the variation due to particular combinations of bottles and batches; and ε_{ija} measures the variation between replicates. This latter quantity can be considered to be the analytical variance for identical sample material analyzed in the same batch.

Eight bottles of each standard were chosen at random from all bottles of each standard. Two 100-mg aliquots of sample were taken from each of these bottles and analyses for ascorbic acid-hydrogen peroxide soluble Cu, Ni, and Co following the method described by Lynch (1971). All samples were analyzed on the same day using the same bottles of chemical reagent, constant instrument settings, and the same working curves. The entire procedure was repeated the next day to produce a second batch of results with a different set of sample aliquots from the same bottles, new reagent solutions, and different instrument settings and working curves. The two day's work thus produced 32 determinations per element per sample.

The results of the analysis of variance are given in Table 1. The analytical variance of replicates within batch/bottle cells (MS_E) is satisfactorily low. If these variances are expressed as a coefficient of variation C_{xy} for element x in sample y:

$$C_{xy} = \frac{100.MS_{E, xy}^{1/2}}{\bar{x}_{xy}}$$

they vary between 1.6 - 5.1%. The low ratios of mean squares between bottles, MS_B , to error mean squares, MS_E , expressed as F_B with 7 and 16 degrees of freedom, shows there is no sample inhomogeneity between bottles for the three components measured. F_B values less than unity for 7 of the 9 columns may indicate some deviation from randomness in analyzing the samples. The ratios F_D with 1 and 16 degrees of freedom indicate that there is a significant difference for more than half of the sample/element combinations between analyses made on different batches. The ratios F_{BD} between the interaction mean square and error mean square are low, indicating that there is no analytical variation due to interaction between bottles and batches. Because F_{BD} is so low it may be desirable to pool the interaction sums of squares and the error sums of squares to form the denominator in the ratios giving F_B and F_D (Dixon and Massey, 1957, p. 168). When this was done, the new F ratios did not affect any of the conclusions given above.

In summary, the results of this experiment show that the standard materials are homogeneous for the components measured, but that there is significant analytical bias between some determinations carried out on different batches. The degrees of bias may be gauged by comparing the mean element values for each day listed in Table 1. The maximum difference between batches is 7.4% of the average amount present for Co in U.M. 4.

ANALYSIS FOR OTHER COMPONENTS

In Table 2 are listed the total metal analyses for Cu, Ni, Co, and Zn. The former two metals have been independently determined by three different analytical methods. Method A. A. S. 1 involves decomposing the samples with a mixture of HF, HNO3 and HClO4, followed by dissolution and measurement on an atomic absorption spectrometer. The values given in Table 2 are the average of five such analyses. For method A. A. S. 2 the sample was decomposed with a lithium metaborate flux (Abbey, 1970), followed again by measurement on an atomic absorption spectrometer. The results for this method are based on two separate analyses. The third method employed was X-ray analysis, for which the samples were first fused with lithium tetraborate and lanthanum oxide. C. A. A. S. sulphide ore was used as a primary standard for all three methods using the following values: Ni 14,000 ppm, Cu 8200 ppm, Co 510 ppm. For Ni and Cu the three sets of data are in good agreement.

Table 3 contains data for a variety of major and minor elements. These data are included for general information; insufficient work has been carried out on these components for the determinations to be considered as standard values. Si, Ti, Al, Fe, Mn, Mg, and Ca have been determined by two different laboratories. In the first the samples were fused with a lithium tetraborate flux containing stabilizers and internal standards and sparked on a direct-reading spectrometer using a Danielsson tape machine. Three analyses were averaged to provide the data in column (a) of Table 3. In the second laboratory a variety of gravimetric, colorimetric, and atomic absorption procedures were employed. The results are based on single analyses. The chemical measurements for total iron and for MgO are consistently lower than the data from the emission spectrometer. Otherwise, agreement is good. Excepting for the data taken from Table 2, the remainder of the values in Table 3 are based on single analyses by a variety of chemical procedures. The oxidation states of iron in the samples were not measured because of the interfering effects of the sulphides.

Two-way analysis of variance table for the determination of ascorbic acid-hydrogen peroxide soluble Cu, Ni and Co in three ultramafic rocks

Table 1

29.0** 68.4 0.3 65.9 63.5 0.4 പ്പ 14.4 6.6 2.6 3.9 18.4 ۲.٦ 90. 90. 05. 328. SAMPLE U.M. 4 1.2 0.1 1.6 947. 1,915 1,920. , 909. 7,722. 903. 12,822. 15,150. 36,597. 1,103. 903. 1,832. Ni 0.2 3.3 0.7 1.2 3 537. 50. 386. 322. 538. 539. 1,538. 50. 2,704. 5,152. 9,444. 220. 13.5** 0.2 0.0 6.9 0.8 5.] 5.9 37. ട 496. 496. 120. 116. 124. 48. 588. ,138. 2 25.7** 0.2 0.2 3.6 SAMPLE U.M. 243,253. 2,814. 2,300. 1,925. 9,478. 16,097. 13,472. 51,650. 424,472. 2,988. 243,253 N 2,901 2.9* 0.5 0.7 3.2 914. 937. 2,665. 14,622. 25,297. 2,665. 659. 3,399. 4,612. 486. 955. 946. 3 13.4** 1.8 1.0 3.8 ,568. 121. 281. 117. 295. 1,439. 1,568 846. 1,871. 5,724 206. റ്റ 288. 0.5 0.1 0.5 3.2 SAMPLE U.M. 1 6,050. 38,936. 73,031. 39,113. 8,323. 8,337 8,351 273,788. 6,050 1,168,500. 272,550 ,720,888 ž 13.9** 0.3 0.5 2.5 5,567. 11,153. 4,147. 4,217. 21,622. 38,972. 178,450. 394,447. 3,089. 65,403. 4,078 155,403 3 F_B (7, 16) F_D (1, 16) F_{BD} (7,16) X₁ (ppm.) X₂ (ppm.) (bpm.) ss_{BD} ss_E ss_T MS_{BD} MS_E MS B R ss_b ىلى ×

 \overline{X} = overall mean; \overline{X}_1 = mean, day 1; \overline{X}_2 = mean, day 2; SS = sums of squares; MS = mean square; C = Coefficient of variation.

Subscripts: B = between bottles; D = between days, BD = interaction; E = error;

F significant at: * (~89%), and ** (>99%) levels.

Table 2. Total Ni, Cu, Co, and Zn in Three Ultramafic Rock Samples.

A.A.S. 2: average of two atomic absorption analyses by J.L. Bouvier. X-ray results based on one analysis. A.A.S. 1: average of five atomic absorption analyses by J.J. Lynch and Mrs. A. MacLaurin.

CONSTITUENT	U.M. 1	U.M. 2	U.M. 4	METHOD
Total Ni, %	1.03	0.385	0.251	A.A.S. 1
Total Ni, %	0.877	0.385	0.260	A.A.S. 2
Total Ni, %	0.965	0.396	0.250	X-RAY
Mean	0.96	0.39	0.25	
Total Cu, %	0.420	0.092	0.055	A.A.S. 1
Total Cu, %	0.435	0.104	0.064	A.A.S. 2
Total Cu, %	0.374	0.090	0.054	X-RAY
Mean	0.41	0.095	0.058	
Total Co. p.p.m.	362.	178.	108	A.A.S. 1
Total Zn, p.p.m.	97.	32.	63.	A.A.S. 1

Table 3. Major and Minor Element Content of Three Ultramafic Rock Samples.

Total element contents determined on a moisture-free basis. Analysis totals approximate because oxidation state of iron has not been determined.

Analytical code: I direct reading emission spectrometry, R. Horton, analyst.

Zgravimetric, J.L. Bouvier.

³colorimetric, J.L. Bouvier.

⁴atomic absorption spectrometry, J.L. Bouvier.

⁵titrimetric, J.L. Bouvier.

⁶oxide equivalents of mean of data reported in Table 2.

Mmean of columns (a) and (b).

		ΣM	2 ^M	×	Σ	يع	Σ	M ₂	54	84	23	6 ²	62	45	94	2 ₆	76	146	086			2	6
	5)	39.3	0.0	. 8.9	, 12.8	.0.1	22.5	. 6.2	0.4	.0.1	0.0	. 4.8	0.2	. 0.4	2.5	. 0.3	0.0	0.0	0.0	-	9.66	0.2	66
U.M. 4	(p)	, 39.3 ²	0.34 ³	, 8.86 ⁴	12.2 ⁴	0.154	21.9 ²	6.24															
	(a)	39.4 ¹	0.35	9.10 ¹	13.4	0.15	23.1	6.3 ¹															
	(c)	, 39.2 ^M	0.24 ^M	1.23 ^M	, 12.95 ^M	1 0.08 ^M	, 25.45 ^M	1.68 ^M	0.32 ⁴	0.114	10.02^{3}	, 6.27 ²	, 0.10 ²	• 0.94 ⁵	, 1.51 ⁴	, 0.49 ⁶	0.12 ⁶	• 0.023 ⁶	0.0046	-	99.74	0.47	99.27
U.M. 2	(q)	39.6 ²	0.243	7.424	12.44	0.074	24.3 ²	4.72 ⁴						_		_		_		-			•
	(a)	38.8 ¹	0.25	7.05 ¹	13.5 ¹	0.08	26.6	4.65 ¹															
	(c)	37.6 ^M	, 0.10 ^M	1.00 ^M	17.2 ^M	0.16 ^M	36.05 ^M	, 2.34 ^M	0.084	0.034	0.00 ³	0.42 ²	0.26 ²	1 3.53 ⁵	0.45 ⁴	1.22 ⁶	0.51 ⁶	0.046 ⁶	0.012 ⁶	-	101.01	1.76	99.25
U.M. 1	(p)	37.5 ²	0.13 ³	0.87 ⁴	16.8 ⁴	0.164	35.5 ²	2.304					_										
	(a)	37.7 ¹	0.08 ¹	1.13	17.6 ¹	0.15	36.6 ¹	2.38															
CONSTITUENT		Si0,	Tio	A1,03	Total Fe as Fe0	MnO	MgO	Ca0	Na.,0	K,0	P,0 _E	H_0	c0,	S ^c	Cr.,03	NiO	Cu0	CoO	ZnO			Less 0 for S	Totals

Table 3 (cont'd)

- 10 -

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