RI 8948

Analysis of Sampling Variance From Certain Platinum and Palladium Deposits in Alaska

By James C. Barker, Dana L. Thomas, and Daniel B. Hawkins

UNITED STATES DEPARTMENT OF THE INTERIOR

BUREAU OF MINES 75TH ANNIVERSARY
1910 1985

Analysis of Sampling Variance From Certain Platinum and Palladium Deposits in Alaska

By James C. Barker, Dana L. Thomas, and Daniel B. Hawkins

CONTENTS

ILLUSTRATIONS

TABLES

UNIT OF MEASURE ABBREVIATIONS USED IN THIS REPORT

Abbreviation	Unit of measure	To convert to--	Multiply by--
°F	degree Fahrenheit		
ft	foot	meters	0.30
g	gram	{ ounces	.0353
		troy ounces	.0322
h	hour		
in	inch		
lb	pound	kilograms	.4535
	mile	kilometers	1.6093
mm	millimeter	inches	.03937
ppb	part per billion		
pct	percent		
tr oz/ton	troy ounce per ton[1]	parts per billion	.00002916

[1]Conversions useful in text:
  0.003 tr oz/ton = 100 ppb.
   .010 tr oz/ton = 340 ppb.
   .029 tr oz/ton = 1,000 ppb.
   .10 tr oz/ton = 3,400 ppb.

ANALYSIS OF SAMPLING VARIANCE FROM CERTAIN PLATINUM AND PALLADIUM DEPOSITS IN ALASKA

By James C. Barker,[1] Dana L. Thomas,[2] and Daniel B. Hawkins[3]

ABSTRACT

The analytical variability encountered when sampling for platinum-group metals (PGM) was statistically evaluated during a 1983 study by the Bureau of Mines and the University of Alaska. Sampling procedures were designed to systematically incorporate commonly used reconnaissance exploration techniques under actual field conditions. Analytical variability was evaluated at each procedural step. Analytical data from each sample from a single deposit grouped relatively well around the calculated mean for that deposit. Standard deviations did not exceed 43 pct of the mean for any of the deposits; more commonly, the deviation was about 25 pct. Statistical treatment of the data by analysis of variance indicated the most important source of error is selection of the sample site. Two or more replicate samples appear necessary to adequately quantify the PGM content. Secondly, significant variation occurs during preparation of a pulverized split for fire assay. The optimum sample size was in the 11-lb range; larger samples did not appreciably reduce variability. Smaller samples generally indicated a higher degree of variability. Evaluation of the sample method suggested the common grab sample, if carefully taken, is a useful indicator of the general range of PGM concentration but potentially includes a considerable margin of error.

[1]Supervisory mining engineer, Alaska Field Operations Center, Bureau of Mines, Fairbanks, AK.

[2]Assistant professor of statistics, Math Department, University of Alaska, Fairbanks, AK.

[3]Professor of geology, Department of Geology and Geophysics, University of Alaska, Fairbanks, AK.

2

INTRODUCTION

The Bureau of Mines Alaska Field Operations Center is evaluating "critical and strategic" mineral deposits in Alaska. The platinum-group metals (PGM)[4] are among the minerals being evaluated. The ability to estimate the quantity of a given mineral in a specific deposit depends on a reliable means of sampling and analyzing the quantity of that mineral contained in its natural geologic setting. The sampling of PGM deposits is subject to poorly understood variables that may be introduced at each step of the sampling and analytical procedures. Appraising PGM deposits requires the ability to obtain reproducible analytical results from geologic structures containing PGM at levels as low as 5 ppb.

Concentrations of PGM as low as 50 ppb have economic value as byproducts of many of the world's copper-nickel mines. It has been necessary for the Bureau to review many poorly known mineral occurrences where low PGM values may represent a byproduct source of these critical and strategic minerals. The need to evaluate such extremely low concentrations magnifies the effects of potential errors normal to all sampling procedures. Each of the principal steps where variance may be introduced during sampling was statistically evaluated for this report. The research documented in this report is a cooperative effort between the Bureau of Mines and the University of Alaska, Fairbanks.

ACKNOWLEDGMENTS

SOURCES OF ANALYTICAL VARIABILITY PERTAINING TO SAMPLING OF PGM DEPOSITS

Unquantified sources of error can be introduced during the collection and analysis of samples and may affect the confidence level of the analytical results. Regardless of the commodity sought, these sources of error can be grouped according to the normal procedural steps of sampling, including selection of the sample sites, sampling method, size of the sample, preparation, and analytical procedures.

Selection of the sample site is particularly difficult when evaluating metals of high intrinsic value (gold, silver, and PGM) that are typically present at concentrations so low they are rarely seen in the field. However, unlike PGM deposits, gold and silver commonly occur within visually identifiable chemical alteration zones. These zones are logical units for gold and silver sampling. PGM

distribution, on the other hand, is typically controlled by complex magmatic processes, the results of which are difficult to recognize, particularly when the local geology is poorly understood in the early stages of exploration.

Once a potential source of PGM is recognized, the method of collecting the sample must be chosen. Reconnaissance-level exploration will frequently rely on a grab sample consisting of a few typical pieces of mineralized rock. This method is commonly used to merely indicate the presence or absence of a mineral, although it is assumed to have a high level of inaccuracy. Depending upon the degree of interest and commitment of time and resources, a more representative continuous chip or channel sample or drill core may instead be collected on a line across the width of the mineralized zone.

Qualitatively, the factors affecting the minimum sample size that is adequate to represent a mineralized deposit at a given level of precision are grade,

---

[4]The platinum-group metals include platinum, palladium, rhodium, ruthenium, osmium, and iridium.

heterogeneity, and grain size of the mineral of interest. At very low grades, which are typical of precious metal deposits, the sample size (in pounds) must be proportionally larger to capture sufficient mineral grains to represent the deposit. The number of mineral grains that must be present in a sample in order to achieve a desired level of precision has been researched for gold analysis by Clifton (1).[5] Grain size and heterogeneity of the mineralization are commonly unknown at the time of field sampling. Platinum metals typically form from moderately high temperature magma and solidify by reductive exsolution at submagmatic temperatures. The resulting PGM grains often have an erratic distribution in the host rock. The presence of heterogeneous mineral grains (informally known as the "nugget effect") may render individual sample analyses meaningless if the sample is of insufficient size. Conceptually, the nugget effect is the random dispersement of mineral grains (nuggets), which results in a relatively few samples capturing a grain while most do

not. Consequently, unless greater sample weights are collected, sporadic high assay results are encountered among a preponderance of nil assays.

Sample preparation, including crushing, pulverizing, and preparation of splits for preconcentration, offers repeated opportunities for error. The nugget effect further exerts an influence when a sample is split in the laboratory and only a small portion of the original sample is analyzed. Contamination from other samples, stratification of heavy grains in the pulp, and statistically predictable error introduced by splitting a sample are also sources of error.

It is widely suspected that past PGM analytical procedures have frequently been unable to provide reproducible results. Recent research and development[6] evolving from PGM exploration of the Stillwater Complex in Montana and elsewhere has improved analytical capabilities and provided the rapid and inexpensive procedure (2) that was selected for this investigation.

OBJECTIVES

The objective of this investigation was to examine and statistically quantify the analytical variability that occurred, and the cumulative degree of error that resulted, at each of the following steps of sampling:

a. Selection of the sample site within the deposit.

b. Sampling method; the reliability of two common methods of sampling was statistically compared, i.e., continuous chip sampling on a line across the mineralized structure and grab sampling.

c. Size of sample collected; three size ranges were evaluated.

d. Crushing and splitting the 10-mesh material.

e. Pulverizing and splitting for fire assay preconcentration.

f. Chemical analyses of replicate splits.

It was not the intent of this investigation to develop new methods of evaluating PGM deposits; rather it was intended to provide quantified practical examples of the analytical limitations of present methodology. Furthermore, it was not intended to achieve the best estimate of PGM within the deposits, for which universal kriging (3) is probably appropriate, but rather to evaluate more commonly used methods of sampling.

Statistical data were obtained by systematic field sampling of PGM-bearing deposits. The experiment was designed to simulate typical examples of field examinations that would be encountered during exploration for PGM. The known PGM deposits in Alaska are either placers containing native PGM, or PGM associated with iron-copper-nickel-cobalt sulfide

[5]Underlined numbers in parentheses refer to items in the list of references at the end of this report.

[6]Fire assay-atomic absorption analytical process developed by Bondar-Clegg, Inc., Lakewood, CO.

deposits. Presently there are no known lode deposits of oxide association (e.g., chromite, magnetite) that warrant consideration as even subeconomic resources. For this project, five mineralized deposits where previous Bureau of Mines sample data indicated PGM to be present were chosen for sampling. The five deposits represent a range of deposit sizes and geologic settings; however, all five deposits contain total PGM on the order of 0.003 to 0.10 tr oz/ton (100 to 3,400 ppb). In each case the platinum and palladium are in an undefined association with iron-copper-nickel-cobalt minerals. Although all six platinum-group metals have been detected in some of the deposits, only platinum and palladium were evaluated during this project.

Four of the deposits are in the Rainbow Mountain area of the central Alaska Range (figs. 1 and 2), and the fifth is near Chitina in the Chugach Range (figs. 1 and 3).

Near Rainbow Mountain, the deposits are (1) Rainbow Mountain, a gabbronorite dike, (2) Emerick Prospect, a massive sulfide lens, (3) Glacier Lake, mineralization at a quartz diorite-serpentinite contact, and (4) Ann Creek, sulfide mineralization in an olivine gabbronorite sill. The Spirit Mountain deposit, southeast of Chitina, is a sulfide mineralized ultramafic sill.

FIGURE 1. - Location map.

LEGEND

A Rainbow Mountain – gabbro-norite dike

B Emerick Prospect – massive sulfide lens

C Glacier Lake – quartz diorite-serpentinite contact

D Ann Creek – gabbro sill

0   1
Scale, mile
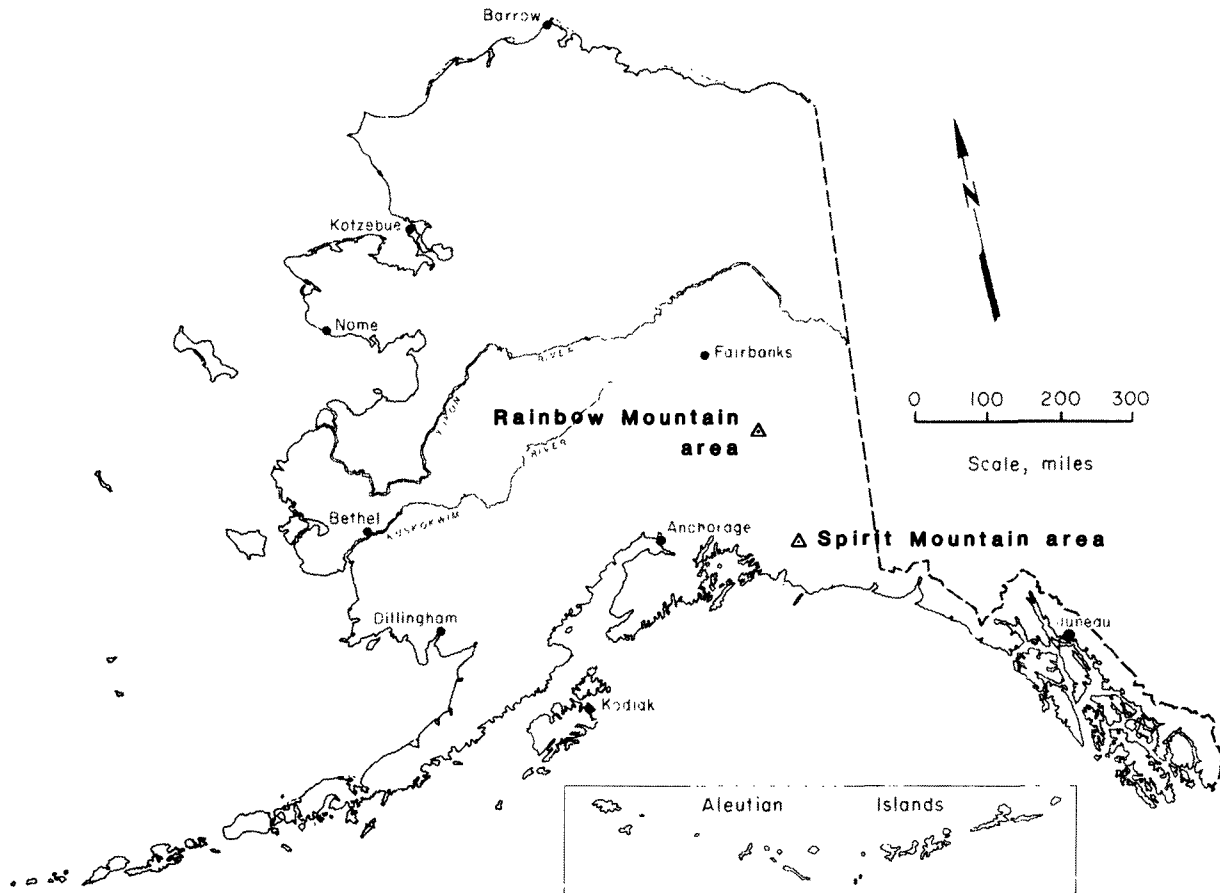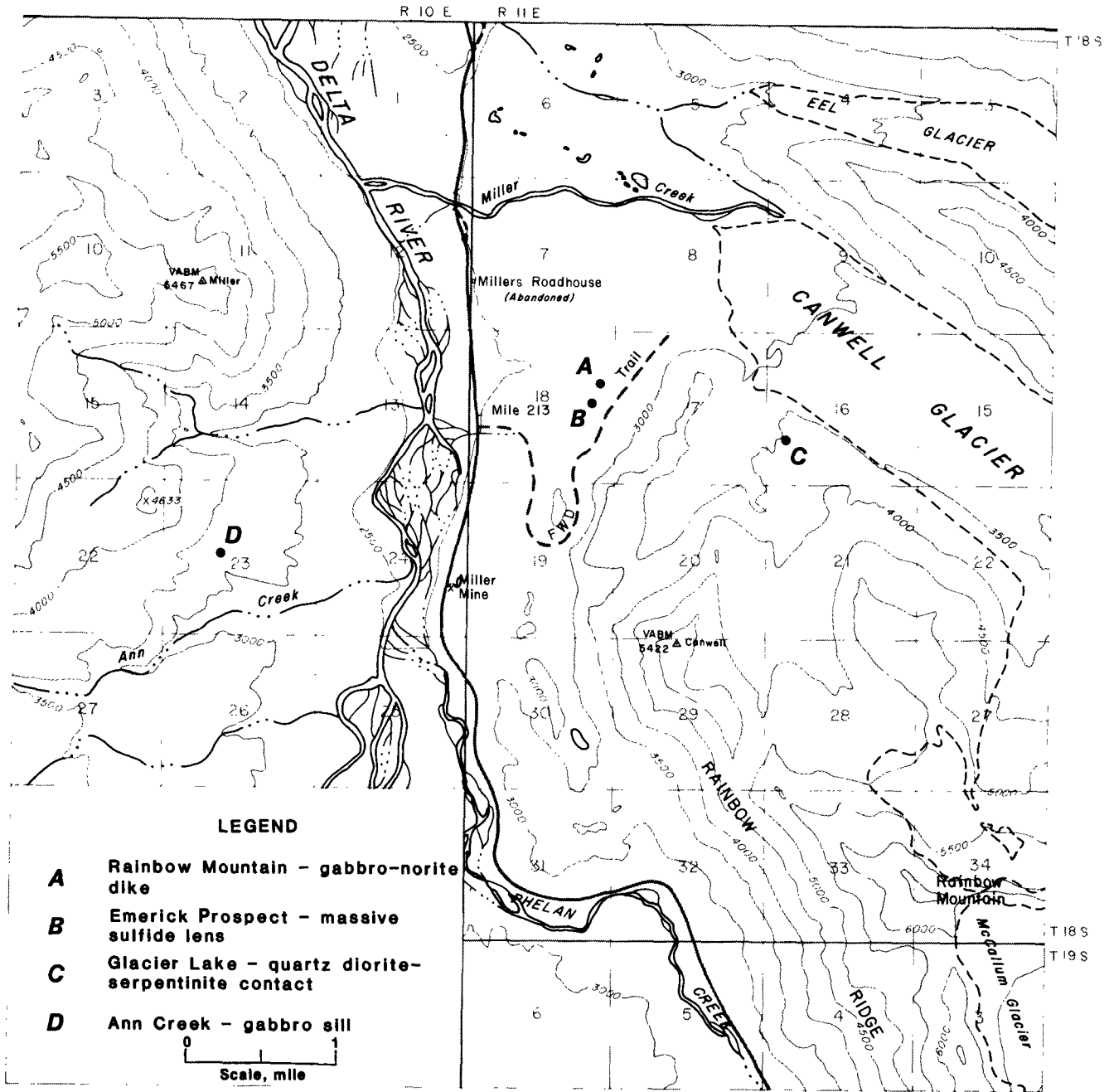
FIGURE 2. - Rainbow Mountain area.
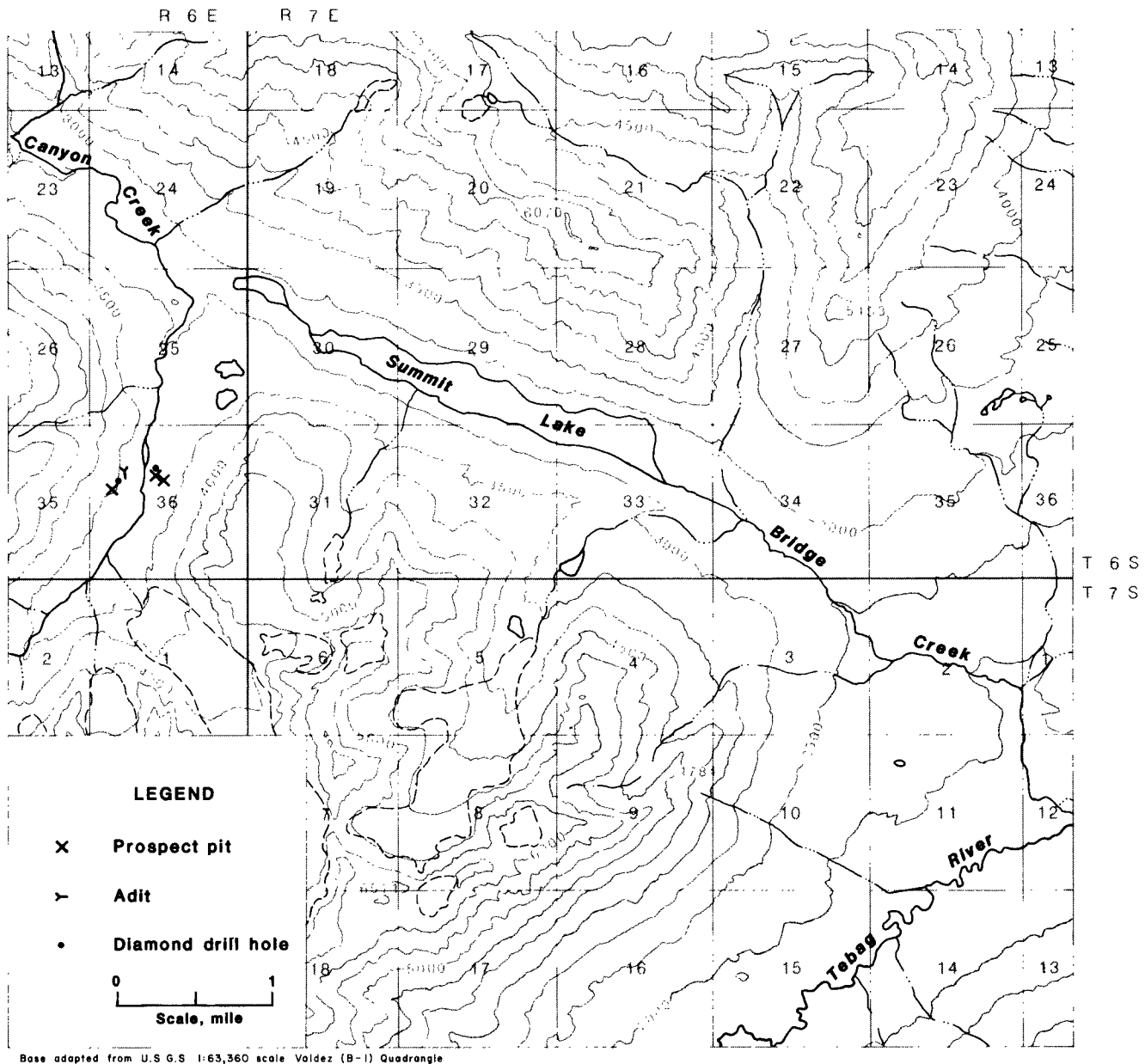
FIGURE 3. - Spirit Mountain area.

SAMPLING AND ANALYTICAL PROCEDURES

Sampling and analytical procedures were designed to permit statistical evaluation of each step of the sampling process that would contribute to analytical variability. The field sampling procedure is illustrated and described for an example deposit in figure 4.

SAMPLE SITE SELECTION

Samples comprise a continuous series of chips that were moiled along a sampling line (A) oriented perpendicular to the strike of the mineralized zone. A replicate sample was then collected from a second, parallel line (B) spaced 2 to 4 ft from the first line. The length of the parallel sample lines ranged from 6 in at the Emerick prospect to 100 ft at the Ann Creek site. Care was taken to avoid high-grading and to assure that a reasonably equal amount of material was collected uniformly across the zone. These replicate lines allow for the

FIGURE 4.- Parallel sampling lines across the Rainbow Mountain gabbronorite dike. The dike strikes into the slope and dips nearly vertical. Sampling lines, indicated by flagging, are perpendicular to the strike. Continuous chip samples of approximately 3- to 4-lb, 10- to 12-lb, and 20- to 22-lb size ranges were collected along each line. A random grab sample of "typical" mineralization was also collected in the vicinity of the lines.
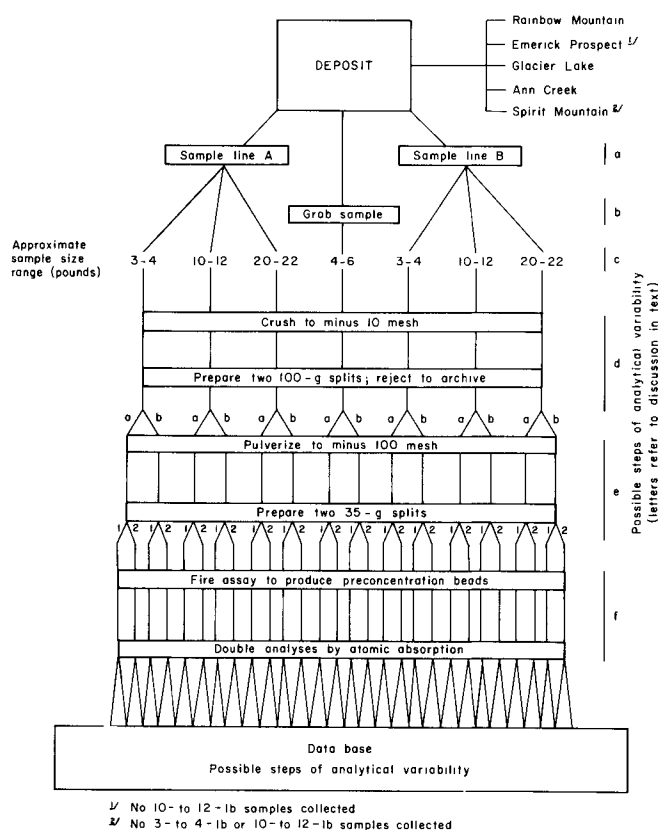
FIGURE 5. - Flowchart showing sample data generation by deposit.

1/ No 10- to 12 - lb samples collected
2/ No 3- to 4 - lb or 10- to 12 -lb samples collected

measurement of the sample variance caused by sample site selection and are analogous to resampling of an outcrop from which an earlier sample was collected.

SAMPLE METHOD

In addition to the two parallel sampling lines designated for each deposit, a single "typical" grab sample was collected. Comparison of analytical results allows determination of the accuracy range of this common form of reconnaissance-level exploration sampling. Grab samples weighing 3 to 6 lb consist of three or four pieces of mineralization that were visually estimated to be typical of the mineralized zone.

SAMPLE SIZE

Three size ranges typical of the sample sizes commonly collected during mineral exploration were collected at each deposit. Variation in analytical results of these samples represents the variability that is dependent upon the size of the sample. Each of these sample size ranges (i.e., 3 to 4 lb, 10 to 12 lb, and 20 to 22 lb) was collected along both sampling lines. Chip size and depth of moiling varied according to the desired sample size range and length of the sampling line.

CRUSHING AND SPLITTING THE 10-MESH MATERIAL

Samples were processed at the Bureau's sample preparation facility in Juneau, AK, according to the flow chart shown in figure 5. Initially, samples were dried in a hot air electric dryer at approximately 180° F for 8 h. After drying, the samples were fed through a jaw crusher which reduced the material to approximately minus 4 mesh. The samples were then further reduced in a gyrocrusher to approximately minus 10 mesh. The minus 10-mesh material then was passed through a splitter to produce about 100 g of homogeneous sample. Producing a split of the original sample incorporates a possible variance. Therefore, to determine the level of this error, a second 100-g split was also produced. Each 100-g split shown on figure 5 is labeled with an a or b and is so listed in table 1.

TABLE 1. – Analytical results

Sample site and method[1]	Sample size, lb	Minus 10-mesh split (100 g)	Split for pre-concentration--dore bead (35 g)	Double analysis by atomic absorption, ppb			
				1st analysis		2d analysis	
				Pt	Pd	Pt	Pd
RAINBOW MOUNTAIN DIKE							
A..........	4	a............	1	1,250	1,190	1,210	1,160
			2	1,090	940	1,120	940
		b............	1	1,290	1,140	1,320	1,240
			2	1,420	1,340	1,400	1,370
	13	a............	1	1,160	1,020	1,160	1,040
			2	1,240	1,140	1,260	1,180
		b............	1	1,180	1,160	1,180	1,150
			2	1,190	1,170	1,190	1,230
	22	a............	1	1,230	1,240	1,230	1,220
			2	1,290	1,200	1,290	1,160
		b............	1	1,210	1,200	1,180	1,220
			2	1,130	1,180	1,150	1,160
B..........	3.75	a............	1	575	720	565	720
			2	720	610	705	625
		b............	1	380	515	390	525
			2	375	410	395	400
	13	a............	1	1,020	975	1,030	965
			2	1,010	955	1,030	965
		b............	1	1,050	925	1,060	915
			2	1,110	935	1,090	945
	22	a............	1	995	895	990	905
			2	895	840	875	840
		b............	1	1,030	1,010	1,050	995
			2	895	945	895	850
Grab.......	4	a............	1	1,080	1,160	1,080	1,180
			2	1,060	1,050	1,060	1,120
		b............	1	1,140	1,010	1,120	1,070
EMERICK PROSPECT							
A..........	3	a............	1	355	775	400	780
			2	355	930	375	945
		b............	1	295	945	365	920
			2	300	755	300	740
	20	a............	1	650	1,170	630	1,190
			2	675	1,230	665	1,190
		b............	1	555	1,110	550	1,100
			2	545	1,200	590	1,200
B..........	3	a............	1	635	1,310	650	1,330
			2	535	825	700	890
		b............	1	355	585	445	555
			2	630	720	660	740
	20	a............	1	350	545	410	555
			2	470	605	440	645
		b............	1	585	740	535	740
			2	490	745	475	760
Grab.......	5	a............	1	130	480	110	460
			2	210	655	235	675
		b............	1	405	780	375	760
			2	495	855	480	850

See footnotes at end of table.

TABLE 1. - Analytical results--Continued

Sample site and method[1]	Sample size, lb	Minus 10-mesh split (100 g)	Split for pre-concentration--dore bead (35 g)	Double analysis by atomic absorption, ppb			
				1st analysis		2d analysis	
				Pt	Pd	Pt	Pd
GLACIER LAKE							
A..........	3	a............	1	360	485	355	500
			2	340	475	345	475
		b............	1	385	515	390	510
			2	385	470	370	490
	12	a............	1	450	510	410	530
			2	325	550	295	565
		b............	1	360	535	375	540
			2	300	505	285	505
	20	a............	1	400	505	375	505
			2	360	510	330	520
		b............	1	265	525	320	535
			2	295	525	345	525
B..........	4	a............	1	255	535	280	505
			2	525	595	515	610
		b............	1	370	495	365	485
			2	380	480	395	475
	12	a............	1	445	410	480	420
			2	390	405	360	400
		b............	1	420	435	380	485
			2	420	535	445	570
	22	a............	1	530	515	520	485
			2	400	475	405	485
		b............	1	440	470	420	470
			2	445	520	450	515
Grab.......	5	a............	1	375	475	395	465
			2	315	485	305	500
		b............	1	350	465	320	480
			2	440	515	405	490
ANN CREEK							
A..........	3.5	a............	1	185	135	210	145
			2	95	145	120	130
		b............	1	125	135	125	140
			2	130	125	140	130
	12.0	a............	1	ND	70	ND	90
			2	ND	125	ND	145
		b............	1	ND	60	ND	40
			2	60	75	ND	55
	22.0	a............	1	ND	135	ND	135
			2	ND	100	ND	110
		b............	1	ND	125	ND	120
			2	ND	80	ND	105

See footnotes at end of table.

TABLE 1. – Analytical results--Continued

Sample site and method[1]	Sample size, lb	Minus 10-mesh split (100 g)	Split for pre-concentration--dore bead (35 g)	Double analysis by atomic absorption, ppb			
				1st analysis		2d analysis	
				Pt	Pd	Pt	Pd
ANN CREEK--Continued							
B..........	3.5	a............	1	130	80	175	95
			2	ND	90	ND	95
		b............	1	ND	95	ND	90
			2	50	85	85	100
	12.0	a............	1	ND	95	95	100
			2	ND	60	ND	65
		b............	1	65	95	65	85
			2	ND	80	90	75
	21.0	a............	1	ND	85	ND	90
			2	80	100	70	125
		b............	1	80	105	70	105
			2	ND	50	ND	50
Grab.......	6.5	a............	1	75	145	80	145
			2	ND	165	ND	185
		b............	1	50	85	55	95
			2	ND	155	ND	140
SPIRIT MOUNTAIN							
A..........	19.5	a............	1	70	225	50	220
			2	110	240	80	265
		b............	1	245	230	275	225
			2	210	225	195	240
B..........	20	a............	1	205	190	220	195
			2	150	190	155	195
		b............	1	150	170	165	175
			2	250	200	235	190
Grab.......	5	a............	1	60	135	70	140
			2	ND	135	ND	135
		b............	1	155	140	150	145
			2	240	150	235	160

ND Not detected, <50 ppb.

[1]A denotes line A--continuous chip sample. B denotes line B--continuous chip sample. Grab denotes grab sample.

PULVERIZING AND SPLITTING FOR FIRE ASSAY PRECONCENTRATION

The splits were pulverized to minus 100 mesh in a metallic-ceramic swing mill. Two 35-g splits were then prepared for fire assay and numbered 1 or 2 as shown in figure 5. After each sample was passed through either a crusher or the pulverizing mill, the equipment was cleaned with barren quartz to remove any residual material and blown clean with compressed air.

CHEMICAL ANALYSIS

The values for platinum and palladium were determined by Bondar-Clegg, Inc., using fire assay preconcentration followed by atomic absorption analysis (2). A 35-g sample was fused and cupelled using conventional fire assay procedure. Due to high sulfide metal contents of some samples, it was impossible to fuse the entire sample in one crucible. As many as four separate preconcentrations of 8.75 g each were required to composite

the equivalent of 35 g. The dore bead was dissolved, and the resulting solution was buffered with an aliquot of copper-cadmium buffer solution to reduce interference from other noble metals. The buffered solution was analyzed by flame atomic absorption spectroscopy. The analyses were referenced to appropriate standards in a similar matrix. This procedure provides a detection limit of 50 ppb for platinum and 5 ppb for palladium. To appraise the variability of the chemical analyses stage, each dore bead digestion was analyzed twice by atomic absorption and recorded in table 1.

DEPOSIT DESCRIPTIONS

RAINBOW MOUNTAIN

The Rainbow Mountain deposit (location A, fig. 2) consists of a mineralized gabbronorite dike that crops out on an east-facing terrace slope approximately 1 mile east of Milepost 214 on the Richardson Highway (fig. 2). Access to the site is possible by automobile via an unimproved trail, locally known as the Red Rock Canyon trail, which departs the Richardson Highway near Milepost 213. The dike occurs on inactive mining claims known as the Emerick Prospect after the original locator in the late 1950's who explored nearby massive copper-nickel sulfide lenses. Although the dike is visibly mineralized, it appears to have been largely ignored during the original prospecting. Local terrain is glaciated and has vertical relief of about 2,000 ft. Lower slopes are mantled by glacial till.

The dike has intruded an ultramafic body (fig. 4) which consists of highly serpentinized fine-grained pyroxenite and peridotite. The ultramafic is exposed for about 0.5 mile along the face of a glacial escarpment. Petrographic examination of samples from the ultramafic rocks revealed antigorite with chlorite, actinolite, and accessory clinopyroxenes, magnetite, chromite, asbestos, and calcite. Amphibolite segregations are common, as are crosscutting serpentinite-magnetite veinlets. Local geology of the Rainbow Mountain area has been described further by Hansen (4) and Rose (5).

The dike is 13.2 ft wide and strikes west-northwest with a steep northerly dip (fig. 4). The presence of slickenside and mylonite on the wall rock indicates the dike has been emplaced along a fault zone. Observations are tenuous since outcrop is limited to less than 20 ft in either the vertical or horizontal dimension of the dike.

Sulfide copper-nickel-cobalt mineralization with PGM and gold values is disseminated throughout the gabbronorite dike. Thin-section examination showed hypersthene with lesser clinopyroxenes, olivine, and biotite (partially altered to chlorite) to be present in the dike. Feldspars are altered to sericite and clay. From examination of hand samples and polished sections, it was estimated that the dike contained 10 to 15 pct sulfide. In order of abundance the sulfide minerals are pyrrhotite, pyrite, chalcopyrite, pentlandite, and trace bornite. Trace amounts of magnetite are also present.

A 200-lb bulk channel sample for metallurgical testing by the Bureau was also collected across the dike in 1981. Head analyses for precious metals were 0.01 tr oz/ton Pd, 0.031 tr oz/ton Pt, 0.002 tr oz/ton Ir, 0.009 tr oz/ton Rh, 0.148 tr oz/ton Ag, and 0.007 tr oz/ton Au. Additionally, this sample contained 0.25 pct Co, 0.8 pct Cu, and 1.17 pct Ni. Another nearby channel sample contained 0.92 pct Cu and 1.44 pct Ni with 0.032 tr oz/ton Pt, 0.03 tr oz/ton Pd, and 0.01 tr oz/ton Au.

EMERICK PROSPECT

The serpentinized intrusive body, which contains the gabbronorite dike previously discussed, is also host to nearby segregations of massive iron-nickel-copper sulfide (location B, fig. 2). During prospecting and trenching in the 1960's, at least nine lenses and numerous smaller

wisps and segregations of massive sulfide were exposed along the base of the glacial escarpment. At the time of this investigation in 1983 only one lens remained exposed.

The sulfide lenses[7] are associated with northwest-trending shear zones that generally have a steep northeast dip. Lenses range in thickness from several inches to 2.5 ft and typically have a 10:1 length-to-width ratio. Several limonitic, gossan zones, up to 6 ft across, suggest wider zones may have existed prior to surficial oxidation. The lens sampled for this study was about 6 in wide at its midpoint.

Petrographic examination of polished sections from the lenses identified pyrrhotite, pentlandite, chalcopyrite, and minor magnetite and pyrite. The pentlandite occurs as compact, rounded to rectangular, subhedral grains and fine lamellae embedded in other sulfide (primarily pyrrhotite) and silicate minerals. Pyrrhotite and chalcopyrite are anhedral. Sulfides compose approximately 50 pct or more of the rock and contain interstitial phenocrysts of fine- to medium-grained clinopyroxene. Gypsum, limonite, goethite, malachite, and nickel bloom are common coatings on weathering surfaces.

Head analyses of a 50-lb metallurgical sample collected in 1981 from a nearby 2.5-ft-wide lens were 6.87 pct Ni, 0.72 pct Cu, and 0.2 pct Co. Precious metal content of this sample was 0.018 tr oz/ton Pt, 0.01 tr oz/ton Pd, 0.09 tr oz/ton Ir, 0.02 tr oz/ton Os, 0.018 tr oz/ton Rh, 0.066 tr oz/ton Ru, 0.001 tr oz/ton Au, and <0.1 tr oz/ton Ag. The average value of similar analyses of grab samples of three other sulfide lenses were 5.23 pct Ni and 1.41 pct Cu, with 0.018 tr oz/ton Pt, 0.040 tr oz/ton Pd, and 0.003 tr oz/ton Au. None of these lenses were exposed in 1983.

---

[7]Description of the lenses is based on previous examination by the author in 1971; unpublished notes by B. Thomas, Alaska Field Operations Center, Bureau of Mines, Fairbanks, AK, in 1961-1963; and an unpublished report by R. Saunders, Alaska Division of Mines and Minerals, 1961.

GLACIER LAKE

The Glacier Lake deposit (location C, fig. 2) was discovered by R. Forbes in 1962 and briefly described by Hansen (4) in 1963. Copper-nickel sulfide mineralization is located at the 3,700-ft elevation on a northwest-facing slope, approximately 2 miles east of Milepost 214 of the Richardson Highway. The Red Rock Canyon trail terminates within 0.5 mile of the deposit, which can then be reached on foot. Limited prospecting and trenching occurred following the 1962 discovery; however, exploration ceased soon thereafter. Local geology has been described by Hansen (4) and Rose (5).

Sheared, highly serpentinized peridotite appears to be intruded by at least three granodiorite to quartz diorite dikes. The dikes, which form prominent knobs, are approximately parallel to the contact of a quartz diorite pluton that crops out 75 ft upslope of the innermost dike. Mineralization is localized along with the inner contact of the closest dike to the pluton (fig. 6). The other dikes, which occur within 300 ft and in the downslope direction of the innermost dike, contain only iron staining.

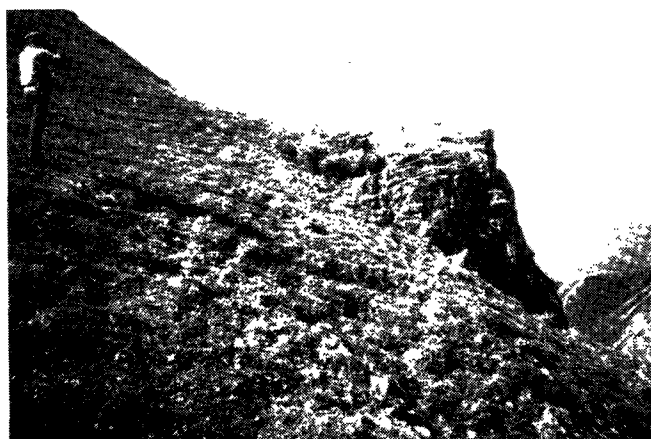The deposit consists of a 5- to 6-ft-wide zone containing disseminated to

FIGURE 6. - Glacier Lake deposit. A steeply dipping mineralized zone approximately 5 to 6 ft wide occurs between the quartz diorite knob and the underlying serpentinized peridotite to the left of the knob. Flagging shows location of sample lines. The steep slope in the upper left of the photograph is the margin of a quartz diorite pluton.

massive sulfide mineralization. The more massive mineralization occurs along the serpentinized peridotite contact of the zone, whereas disseminated mineralization occurs in the quartz diorite dike. The mineralization dips steeply and is exposed for 40 ft along the N 50° E strike. Similar mineralization is also exposed in a prospect pit 75 ft farther along strike. If this mineralization is a continuation of the zone in the first outcrop, a total inferred mineralized length of about 145 ft or more exists. Further extension under talus cover to the southwest is indicated by scattered mineralized float rock. A fault has possibly displaced the eastern portion of the zone 10 to 20 ft to the northwest, although this could not be verified.

The mineralized zone is cut by a stockwork of barren quartz veinlets. Feldspars in the dike are altered to epidote. Gypsum occurs commonly on weathering surfaces. Polished section examination of the sulfide minerals identified pyrrhotite, pentlandite, cubanite, and chalcopyrite. Rounded grains of magnetite occur with oxidative exsolution lamellae of ilmenite and blebs of chalcopyrite. The sulfides occur as abundant irregular masses that locally exhibit remobilization into veinlike fillings. Pyrrhotite, pentlandite, and cubanite appear to be internally associated.

Samples were collected from the prominent outcrop on the northeast end of the sulfide zone shown in figure 6. Because of the outcrop configuration the samples were taken diagonal to the true thickness; consequently, the sample lines were 11.7 ft long.

Assays reported in 1963 by Hansen (4) indicated 1.9 to 6 pct Cu, 1.1 to 1.5 pct Ni, and trace to 0.4 tr oz/ton Au in samples of disseminated mineralization, whereas a sample of the more massive sulfide mineralization contained 6.6 pct Ni, 1.1 pct Cu, and 0.04 tr oz/ton Au. No analyses were done at the time for PGM. A 1981 Bureau channel sample for metallurgical testing across the zone contained 2.75 pct Ni, 1.39 pct Cu, 0.07 pct Co, 0.008 tr oz/ton Pt, 0.012 tr oz/ton Pd, 0.005 tr oz/ton Au, and 0.088 tr oz/ton Ag. Other Bureau samples

contain traces of iridium, rhodium, and ruthenium.

ANN CREEK

The Ann Creek deposit (location D, fig. 2; fig. 7) consists of a low-grade sulfide-bearing olivine gabbronorite sill located 1.5 miles west of Mile 213 on the Richardson Highway. Access requires crossing the Delta River, which is easily done with a small boat. The deposit was originally located and prospected by Emerick in the 1950's; however, exploration activity has long since lapsed. Geology and a brief geologic description are given by Rose (5-6), Stout (7), and Saunders (8).

The mineralized sill is part of a zone of east-trending mafic and ultramafic dikes and sill-like bodies intruded into Pennsylvanian-Permian volcanic rocks. These dikes and sills are found along a probable fault zone(s) that separates siliceous sedimentary rocks, dacitic agglomerates, and tuff to the south from andesitic and basaltic volcanic rocks, graywacke, and felsic to intermediate tuff to the north. In the immediate vicinity of the Ann Creek deposit the sill is hosted by olive-green andesitic volcanic and pyroclastic rocks. Peridotite

FIGURE 7. - Ann Creek deposit. A mineralized olivine gabbronorite sill approximately 120 ft thick dips steeply into the hill. The hanging wall is approximately coincident with the top of the slope. The footwall is to the right of the photograph. Sampling was done in shallow trenches parallel to the downslope direction. Photograph is oriented looking north.

dikes, subparallel to the gabbro sill, occur approximately 300 ft to the north and 200 ft to the south.

The sill is exposed for 250 ft along a strike of N 60° E where relatively recent downcutting by Ann Creek has exposed several hundred vertical feet of bedrock. The width of the sill, which dips steeply to the north, is about 120 ft. Elsewhere, bedrock is mantled by glacial till. A magnetometer survey indicated the structure has an additional unexposed strike length to the east of at least 350 ft and a thickness of 100 to 200 ft. The sill may extend farther yet to the east; however, magnetic response diminished. The magnetic data indicate a northerly dip of about 70°. The western end of the sill is terminated by a presumed fault. Another fault has apparently displaced the eastern half of the sill approximately 100 ft to the north.

The olivine gabbronorite sill is composed of both orthopyroxene and clinopyroxene, olivine, plagioclase, and sulfides. Magnetite is a common accessory. Sulfide minerals include pyrrhotite, pentlandite, pyrite, and chalcopyrite that occur as interstitial grains, generally less than 2 mm across. Crude layering is indicated by variable concentrations of sulfides, which average about 3 to 5 pct by volume, but which are also nearly absent in some layers. Massive lenses of similar sulfide mineralogy, up to 18 in thick, occur along the serpentinized footwall of the sill.

This deposit was chosen for sampling to represent analytical variability encountered when sampling deposits with very low-grade platinum and palladium values. To collect samples from two parallel sampling lines, it was necessary to trench 1 to 2 ft deep through the scree to reach the fractured bedrock. The incline of the south-facing slope was approximately 90° to the northerly dip of the sill. Therefore, the sample lines were nearly perpendicular to the dip. The lines transect only the upper 100 ft of the 120-ft-thick sill as measured down the slope from the hanging wall, thereby excluding the high-grade sulfide lenses which occur near the footwall. Owing to the length of the sample lines, inclusion of the footwall zone could unintentionally result in high-graded samples and statistically alter the results.

Previously reported assays from the sill are 0.2 to 0.46 pct Ni and 0.17 to 0.32 pct Cu, with traces of gold and silver (5). Values for cobalt and PGM were not determined at the time. Copper-nickel grades in excess of 5 pct combined occur within the massive sulfide lenses near the base of the sill. Three grab samples collected during a recent Bureau investigation have indicated an average of 0.003 tr oz/ton Pt and 0.004 tr oz/ton Pd for the main body of the sill. Two samples from the sulfide lenses on the footwall averaged 0.01 tr oz/ton Pt and 0.016 tr oz/ton Pd. Trace levels of iridium and rhodium were also detected in the footwall zone. A continuous chip sample across the sill collected for metallurgical testing contained 0.08 pct Cu, 0.21 pct Ni, and 0.02 pct Co. Precious metals assayed <0.001 tr oz/ton Pt, 0.003 tr oz/ton Pd, <0.001 tr oz/ton Au, and <0.02 tr oz/ton Ag.

SPIRIT MOUNTAIN

The Spirit Mountain copper-nickel deposit (figs. 1 and 3) is located 15 air miles south-southeast of Chitina, AK, and 8 miles east of the Copper River, in glaciated mountainous terrain. Access to the deposit is on foot from a float plane landing site at the western end of Summit Lake situated about 1.5 miles to the northeast. Old workings consisting of a 50-ft adit, prospect pits, drill sites, abandoned equipment, and camps are found at this location on both sides of Canyon Creek, a tributary to the Copper River. Although the Copper River is only 400 ft above sea level, local peaks near the deposit rise abruptly to altitudes of 6,000 to 7,000 ft.

Copper mineralization, later determined to contain nickel, was first discovered along Canyon Creek about 1907. Claims were located on the discovery, and in 1915 a short adit was driven in an unsuccessful attempt to cross-cut the mineralization. The project was abandoned in 1917 and it was not until World War II that further investigations occurred

(9-10), sponsored by the Government because of the wartime shortage of nickel. In 1954 the claims were relocated, and during the 1960's a private exploration company drilled the property; no results of this work are available. Following the drilling program, the property was again allowed to lapse. The most recent detailed description of the geology and mineralization was compiled in 1970 by Herreid (11), who, like earlier investigators, concluded that the small size of the deposit, its remote location, and the lack of other discoveries in the area make development most unlikely.

Mineralization is associated with a peridotite sill that is one of a series of west-northwest trending sills and dikes that range in composition from quartz diorite to hornblendite and peridotite. These bodies intrude a sequence of Permian metavolcanic rocks, chert, and marble known as the Strelna Formation.

The mineralized, irregularly shaped, sill-like intrusion crops out in cliffs about 500 ft above the west side of Canyon Creek. The intrusive body is 200 ft long and is known to extend through a vertical distance of 150 ft. The best exposure is in a trench (fig. 8) that transects the sill across the width of 22 ft. Elsewhere, the sill pinches abruptly to less than a few inches. Disseminated mineralization occurs across the entire sill, whereas massive sulfide mineralization occurs near the footwall.

Copper-nickel sulfides occur sparsely in a 1-ft-wide hornblendite dike about 250 ft to the east of the sill and in a 3-ft-wide sulfide lens on the east side of Canyon Creek.

Herreid (11) described the peridotite as unbanded, medium- to coarse-grained rock containing irregular blebs of sulfides in antigorite, which has replaced

FIGURE 8. - Trench exposure of a mineralized peridotite sill near Spirit Mountain. Flagging indicates the lower sample line B; line A occurs 3 ft farther to the left. The hanging wall is visible in the upper right; the footwall can be seen along the lower edge of the photograph.

olivine, and minor amounts of fosterite, tremolite, and diopside. Sulfide minerals occur as interstitial, disseminated grains and as massive lenses and comprise pyrrhotite, bravoite, chalcopyrite, sphalerite, pentlandite, and pyrite (9). Magnetite and limonite are also present. Kingstron (9) reported the bravoite is partially interstitial to the anhedral pyrrhotite grains but elsewhere replaces the pyrrhotite. Sphalerite is limited to a single massive sulfide lens, where it replaces earlier sulfide minerals along fractures and cleavage planes.

Reported grade of the deposit averages 0.22 to 1.44 pct Ni and 0.12 to 1.40 pct Cu. Massive sulfide lenses contain 0.18 pct Co and up to 7.6 pct Ni (9). In 1982, Miller (12) published geochemical results for platinum and palladium with values up to 0.05 ppm and 7.0 ppm, respectively, for undescribed samples of ultramafic rock from the Spirit Mountain deposit.

STATISTICAL ANALYSIS

SUMMARY STATISTICS

The mean and standard deviation of all platinum and palladium assays for each deposit are given in table 2. Figure 9 illustrates the relationship between average assay of each deposit and the standard deviation for platinum, and figure 10 shows this relationship for palladium. A linear trend showing an increase in the standard deviation with increasing assay values is evident for both metals, but the relationship is slightly more distinct for platinum. Table 3 presents descriptive statistics of platinum and palladium assays for each

TABLE 2. – Descriptive statistics for each deposit, parts per billion

Deposit	Platinum		Palladium	
	Mean	Standard deviation[1]	Mean	Standard deviation[1]
Rainbow Mountain................	1,038.93	251.42(56)	998.13	222.52(56)
Emerick Prospect...............	460.25	153.49(40)	849.63	239.69(40)
Glacier Lake...................	381.43	64.43(56)	499.29	41.02(56)
Ann Creek......................	100.20	43.38(25)	104.732	31.96(56)
Spirit Mountain................	167.05	69.70(22)	188.13	39.61(24)

[1]Numbers in parentheses are the number of assays used in the calculations.

NOTE.--Calculations are based on all the assays from each deposit.

TABLE 3. – Descriptive statistics for platinum and palladium, parts per billion

Sample site and method[1]	Sample size, lb	Platinum		Palladium	
		Mean	Standard deviation[2]	Mean	Standard[2] deviation
RAINBOW MOUNTAIN					
A....................................	4	1,262.50	119.97(8)	1,165.00	160.71(8)
	13	1,195.00	36.25(8)	1,136.00	71.10(8)
	22	1,213.75	59.03(8)	1,197.50	29.16(8)
B....................................	3.75	513.13	147.38(8)	565.63	124.80(8)
	13	1,050.00	35.05(8)	947.50	21.21(8)
	22	953.13	70.35(8)	910.00	67.72(8)
Grab.................................	4	1,085.00	52.37(8)	1,065.00	83.28(8)
EMERICK					
A....................................	3	343.13	39.73(8)	848.75	93.34(8)
	20	607.50	54.05(8)	1,173.75	45.65(8)
B....................................	3	576.25	120.71(8)	869.38	299.40(8)
	20	469.38	72.48(8)	666.88	90.35(8)
Grab.................................	5	305.00	153.157(8)	689.38	153.19(8)
GLACIER LAKE					
A....................................	3	366.25	19.23(8)	490.00	16.90(8)
	12	350.00	59.40(8)	530.00	22.04(8)
	20	336.25	43.57(8)	518.75	10.94(8)
B....................................	4	385.63	96.49(8)	522.50	52.92(8)
	12	417.50	39.46(8)	457.50	65.08(8)
	22	451.25	49.04(8)	491.88	21.37(8)
Grab.................................	5	363.13	48.62(8)	484.38	17.20(8)
ANN CREEK					
A....................................	3.5	141.25	37.58(8)	135.63	7.29(8)
	12	[3]60.00	[3]0. (1)	82.50	35.96(8)
	22	([3])	([3])	113.75	18.85(8)
B....................................	3.5	110.00	[3]54.31(4)	91.25	6.41(8)
	12	78.75	[3]16.01(4)	81.88	14.62(8)
	21	75.00	[3]5.77(4)	88.75	26.69(8)
Grab.................................	6.5	65.00	[3]14.72(4)	139.38	33.75(8)
SPIRIT MOUNTAIN					
A....................................	19.5	154.38	87.03(8)	233.75	14.58(8)
B....................................	20	191.25	41.04(8)	188.13	10.33(8)
Grab.................................	5	151.67	[3]77.24(6)	142.50	8.86(8)

[1]A denotes line A--continuous chip sample. B denotes line B--continuous chip sample. Grab denotes grab sample.

[2]Numbers in parentheses are the number of assays used in the calculations.

[3]Samples with Pt content below the detection limit (<50 ppb) not included in calculations.
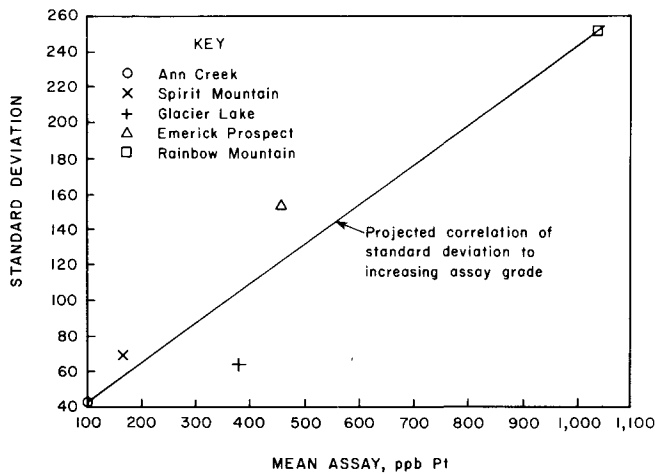
FIGURE 9. - Plot of mean platinum values and standard deviations for each deposit.
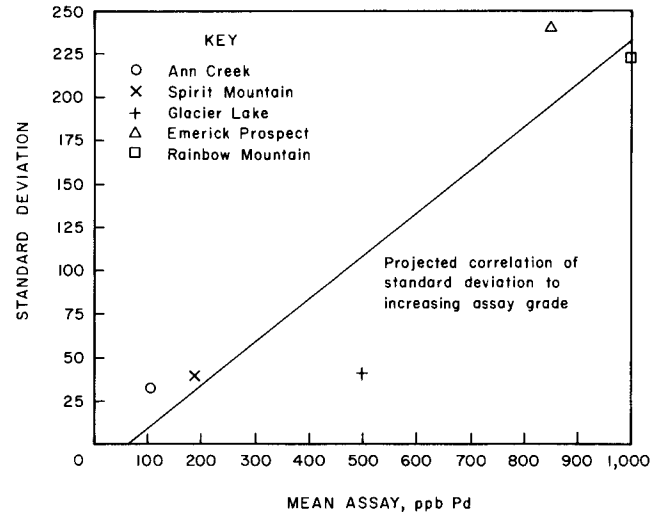

FIGURE 10. - Plot of mean palladium values and standard deviations for each deposit.

deposit-sample site-sample size combination. Examination of this table indicates that although a broad range of average metal content and variability is present, some general trends are evident. First, as expected, the variability of the assays, as measured by the standard deviation, usually decreases for larger sample sizes. Second, relatively little difference in variability is evident when comparing 3- to 4-lb line samples with 4- to 6-lb grab samples. Third, there is often a clear reduction in variablity when comparing a 10- to 12-lb or 20- to 22-lb line sample to a 3- to 4-lb line sample, but little difference between 10- to 12-lb and 20- to 22-lb line samples.

There is good correlation between platinum and palladium values in the samples analyzed. The platinum values of some samples from the Ann Creek and Spirit Mountain deposits were below detection limits, but a first estimate of platinum content can be determined by the platinum and palladium correlation. Figure 11 shows a plot of platinum versus palladium for the 28 samples. The relationship was summarized by means of linear regression of platinum and palladium, for which the

R^2 (coefficient of determination)[8] value is 0.81. This indicates that 81 pct of the variance of the platinum is associated with the regression on palladium. The equation for the regression line is

$$Pt = 0.91 \ Pd - 41.8.$$

This equation can be used as a first approximation[9] to estimate platinum values from the analytical results for palladium; however, its use is restricted to the deposits studied and to the range of palladium values used.

---

[8]An excellent summary of regression statistics can be found in Applied Linear Statistical Models, by J. Neter and W. Wasserman, R. D. Irwin, IC Publication, Homewood, IL, 1974. Ch. 1-6.

[9]Note that the variability in platinum content increases as palladium content increases. For the purpose of estimation the fitted line is appropriate; however, for hypothesis testing a transformation (probably logarithmic) is necessary. As a further generalization, the expected platinum values should be about 90 pct of the observed palladium values.
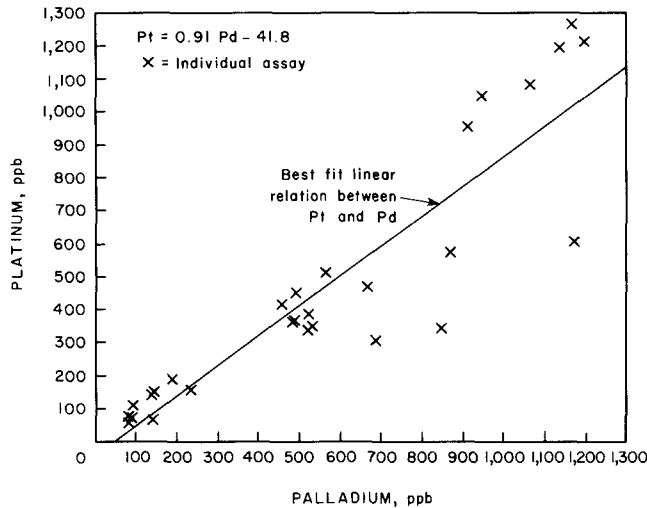
18

FIGURE 11. - Linear regression of platinum versus palladium values.

ANALYSIS OF VARIANCE

Data presented in table 2 were used to determine the variability in analytical results introduced at different steps in the sampling and analytical procedure. The steps (a through f, p. 3) at which analytical variability occurs are cited in the "Objectives" section, shown on figure 5, and discussed in the section on "Sources of Analytical Variability."

Analysis of variance (ANOVA) for nested classifications (13, pp. 571-583) was used to partition potential sources of variability in the sampling procedure. Tables 4 and 5 give the results of ANOVA calculations for each deposit-sample size-sample method (line versus grab) combination for platinum and palladium, respectively. The results for all combinations were not pooled into a single ANOVA table representing a composite of the deposits for hypothesis testing because of large differences in assay variability. As an example of such differences, table 4 gives the mean square for analyses within 35-g splits of a 3- to 4-lb line sample from the Emerick prospect as 2,735.94; the same mean square from Glacier Lake is 79.69. These two mean squares, supposedly measuring the

same source of variability, differ by a factor of 34. A transformation (probably logarithmic) might solve the unequal variance hypothesis testing problem, but it would complicate interpretation. Treating the deposit-sample size-sample method combinations separately for the purpose of hypothesis testing seems more reasonable for the objectives of this study.

F-tests (variance ratio test, 13, pp. 171-173) were calculated for each step in the sampling procedure representing a source of error and listed in the ANOVA classifications (tables 4-5). These tests were used to determine if the source of variability associated with any given step in an ANOVA table contributes significantly to the overall variability in the assays. Exact significance levels (p-values) are given for each F-test. The p-values that were greater than 10 pct were considered nonsignificant and are labeled NS in the tables.

Within-deposit variability may be measured by the variability between sample sites (between sample lines A and B) on the same deposit. The significance of the sample site as a source of variability was not consistent from deposit to deposit nor from platinum to palladium. Variability here is a function of the homogeneity of a deposit with respect to the PGM distribution. The results coincide with intuition on the part of the field geologist by indicating that some of the deposits are relatively uniform whereas others are not.

The variance components for each source of variability in each deposit-sample size-sample method combination are also given in tables 4 and 5. These were calculated for future users attempting to determine the number of assays required to estimate the average element content of a given deposit within a specified error for a given confidence level. Variance components are also useful in cost evaluations of various sampling strategies where the size of the sample to be collected and processed represents a significant cost factor.

TABLE 4. - Nested analyses of variance of platinum for each deposit-sample
size-sample method combination

Sample size, lb	Source of variance	d.f.	Mean square	Significance	Variance component
	RAINBOW MOUNTAIN				
4.0.......	Sample site.................	1	2,246,251.56	p = 0.042	268,060.94
	Crushing and splitting at 10 mesh.	2	101,764.06	p = 0.035	22,506.25
	Pulverizing and splitting..	4	11,739.06	p < 0.001	5,725.00
	Chemical analyses..........	8	289.06	NAp	289.06
12.0......	Sample site.................	1	84,100.00	p = 0.038	10,084.38
	Crushing and splitting at 10 mesh.	2	3,425.00	NS	215.63
	Pulverizing and splitting..	4	2,562.50	p < 0.001	1,237.50
	Chemical analyses..........	8	87.50	NAp	87.50
19.0......	Sample site.................	1	271,701.56	p = 0.033	[1]32,754.95
	Crushing and splitting at 10 mesh.	2	9,382.81	NS	Negative
	Pulverizing and splitting..	4	9,801.56	p < 0.001	4,834.38
	Chemical analyses..........	8	132.81	NAp	132.81
4 (grab)..	Crushing and splitting at 10 mesh.	1	800.00	NS	Negative
	Pulverizing and splitting..	2	6,500.00	p = 0.086	2,575.00
	Chemical analyses..........	4	1,350.00	NAp	1,350.00
	EMERICK				
3.0.......	Sample site.................	1	217,389.06	p = 0.062	[1]25,274.74
	Crushing and splitting at 10 mesh.	2	14,720.31	NS	Negative
	Pulverizing and splitting..	4	15,426.56	p = 0.019	6,345.31
	Chemical analyses..........	8	2,735.94	NAp	2,735.94
20.0......	Sample site.................	1	76,314.00	NS	7,065.62
	Crushing and splitting at 10 mesh.	2	19,789.06	p = 0.059	4,150.00
	Pulverizing and splitting..	4	3,189.06	p = 0.023	1,289.06
	Chemical analyses..........	8	610.94	NAp	610.94
5 (grab)..	Crushing and splitting at 10 mesh.	1	143,112.50	p = 0.063	33,276.56
	Pulverizing and splitting..	2	10,006.25	p = 0.003	4,868.75
	Chemical analyses..........	4	268.75	NAp	268.75
	GLACIER LAKE				
3.0.......	Sample site.................	1	1,501.56	NS	[1]Negative
	Crushing and splitting at 10 mesh.	2	1,320.31	NS	Negative
	Pulverizing and splitting..	4	16,120.31	p < 0.001	8,020.31
	Chemical analyses..........	8	79.69	NAp	79.69

See explanatory notes at end of table.

TABLE 4. - Nested analyses of variance of platinum for each deposit-sample
size-sample method combination--Continued

Sample size, lb	Source of variance	d.f.	Mean square	Significance	Variance component
	GLACIER LAKE--Continued				
12.0......	Sample site.................	1	18,225.00	p = 0.078	[1]1,612.50
	Crushing and splitting.....	2	1,606.25	NS	Negative
	Pulverizing and splitting..	4	7,184.38	p = 0.001	3,364.06
	Chemical analyses..........	8	456.25	NAp	456.25
20.0......	Sample site.................	1	52,900.00	p = 0.071	[1]6,064.06
	Crushing and splitting at 10 mesh.	2	4,225.00	NS	Negative
	Pulverizing and splitting..	4	4,468.75	p = 0.004	1,996.88
	Chemical analyses..........	8	475.00	NAp	475.00
5 (grab)..	Crushing and splitting at 10 mesh.	1	1,953.13	NS	Negative
	Pulverizing and splitting..	2	6,640.63	p = 0.008	3,156.25
	Chemical analyses..........	4	328.13	NAp	328.13
	ANN CREEK				
[2]3.5......	Sample site.................	1	2,604.17	NS	Negative
	Crushing and splitting at 10 mesh.	2	4,118.75	NS	81.41
	Pulverizing and splitting..	2	4,100.00	p = 0.001	1,704.89
	Chemical analyses..........	6	383.33	NAp	383.33
12.0......	NAp-AD.....................	-	NAp-AD	NAp-AD	NAp-AD
22.0......	NAp-AD.....................	-	NAp-AD	NAp-AD	NAp-AD
6.5 (grab)	NAp-AD.....................	-	NAp-AD	NAp-AD	NAp-AD
	SPIRIT MOUNTAIN				
20.0......	Sample site.................	1	5,439.06	NS	Negative
	Crushing and splitting at 10 mesh.	2	23,945.31	p = 0.059	5,026.56
	Pulverizing and splitting..	4	3,839.06	p < 0.001	1,821.88
	Chemical analyses..........	8	195.31	NAp	195.31
[2]5 (grab).	Crushing and splitting at 10 mesh.	1	22,533.33	NS	3,827.08
	Pulverizing and splitting..	1	7,225.00	p < 0.001	3,600.00
	Chemical analyses..........	3	25.00	NAp	25.00

d.f.   Degrees of freedom.

NAp   Not applicable.

NAp-AD Not applicable due to assay determinations below detection limits for platinum.

NS   Not significant, p ≥ 0.10.

p   Probability.

[1]Variance component calculated by replacing the negative value immediately below with zero.

[2]Samples with platinum content below the detection limit (<50 ppb) not included in calculations.

TABLE 5. – Nested analysis of variance of palladium for each deposit-sample size-sample method combination

Sample size, lb	Source of variance	d.f.	Mean square	Significance	Variance component
	RAINBOW MOUNTAIN				
3.0........	Sample site.................	1	1,437,001.56	p = 0.057	168,529.69
	Crushing and splitting at 10 mesh.	2	88,764.06	NS	15,554.69
	Pulverizing and splitting..	4	26,545.31	p < 0.001	12,890.63
	Chemical analyses..........	8	764.06	NAp	764.06
12.0......	Sample site.................	1	142,506.25	p = 0.052	16,809.38
	Crushing and splitting at 10 mesh.	2	8,031.25	NS	793.75
	Pulverizing and splitting..	4	4,856.25	p = 0.002	2,237.50
	Chemical analyses..........	8	381.25	NAp	381.25
19.0......	Sample site.................	1	330,625.00	p = 0.019	40,500.00
	Crushing and splitting at 10 mesh.	2	6,625.00	NS	485.94
	Pulverizing and splitting..	4	4,681.25	p = 0.014	1,960.94
	Chemical analyses..........	8	759.38	NAp	759.38
5 (Grab)..	Crushing and splitting at 10 mesh.	1	31,250.00	NS	6,206.25
	Pulverizing and splitting..	2	6,425.00	p = 0.066	2,656.25
	Chemical analyses..........	4	1,112.50	NAp	1,112.50
	EMERICK				
3.0.......	Sample site.................	1	1,701.56	NS	Negative
	Crushing and splitting at 10 mesh.	2	192,807.81	NS	29,493.75
	Pulverizing and splitting..	4	74,832.81	p < 0.001	37,196.88
	Chemical analyses..........	8	439.06	NAp	439.06
19.0......	Sample site.................	1	1,027,689.06	p = 0.025	125,085.16
	Crushing and splitting at 10 mesh.	2	27,007.81	p = 0.051	5,770.31
	Pulverizing and splitting..	4	3,926.56	p = 0.001	1,827.50
	Chemical analyses..........	8	251.56	NAp	251.56
5 (Grab)..	Crushing and splitting at 10 mesh.	1	118,828.13	NS	24,103.13
	Pulverizing and splitting..	2	22,415.63	p < 0.001	11,131.25
	Chemical analyses..........	4	153.13	NAp	153.13
	GLACIER LAKE				
3.0.......	Sample site.................	1	4,225.00	NS	Negative
	Crushing and splitting at 10 mesh.	2	6,162.50	NS	1,020.31
	Pulverizing and splitting..	4	2,081.25	p = 0.001	981.25
	Chemical analyses..........	8	118.75	NAp	118.75
12.0......	Sample site.................	1	21,025.00	NS	1,401.56
	Crushing and splitting at 10 mesh.	2	9,812.50	NS	1,754.69
	Pulverizing and splitting..	4	2,793.75	p = 0.003	1,256.25
	Chemical analysis..........	8	281.25	NAp	281.25

See explanatory notes at end of table.

TABLE 5. - Nested analysis of variance of palladium for each deposit-sample
   size-sample method combination--Continued

Sample size, lb	Source of variance	d.f.	Mean square	Significance	Variance component
GLACIER LAKE					
20.0......	Sample site.................	1	2,889.06	p = 0.095	[1]289.86
	Crushing and splitting.....	2	320.31	NS	Negative
	Pulverizing and splitting..	4	695.31	p = 0.005	309.38
	Chemical analysis..........	8	76.56	NAp	76.56
5 (Grab)..	Crushing and splitting at 10 mesh.	1	78.13	NS	Negative
	Pulverizing and splitting..	2	703.13	p = 0.087	278.13
	Chemical analysis..........	4	146.88	NAp	146.88
ANN CREEK					
3.5.......	Sample site.................	1	7,876.56	p = 0.006	[1]978.68
	Crushing and splitting at 10 mesh.	2	45.31	NS	Negative
	Pulverizing and splitting..	4	32.81	NS	Negative
	Chemical analysis..........	8	54.69	NAp	54.69
12.0......	Sample site.................	1	1.56	NS	Negative
	Crushing and splitting at 10 mesh.	2	2,514.06	NS	339.06
	Pulverizing and splitting..	4	1,157.81	p = 0.003	523.44
	Chemical analysis..........	8	110.94	NAp	110.94
19.0......	Sample site.................	1	2,500.00	NS	[1]171.35
	Crushing and splitting at 10 mesh.	2	662.50	NS	Negative
	Pulverizing and splitting..	4	1,362.50	p = 0.001	637.50
	Chemical analysis..........	8	87.50	NAp	87.50
Grab......	Crushing and splitting at 10 mesh.	1	3,403.13	NS	325.00
	Pulverizing and splitting..	2	2,103.13	p = 0.006	1,006.25
	Chemical analysis..........	4	90.63	NAp	90.63
SPIRIT MOUNTAIN					
19.0......	Sample site.................	1	8,326.56	p = 0.016	[1]1,005.47
	Crushing and splitting at 10 mesh.	2	132.81	NS	Negative
	Pulverizing and splitting..	4	357.81	p = 0.022	145.31
	Chemical analysis..........	8	67.19	NAp	67.19
5 (Grab)..	Crushing and splitting at 10 mesh.	1	312.50	NS	57.81
	Pulverizing and splitting..	2	81.25	p = 0.100	31.25
	Chemical analysis..........	4	18.75	NAp	18.75

d.f. Degrees of freedom.   NS Not significant, $p > 0.10$.
NAp   Not applicable.   p   Probability.
[1]Variance component calculated by replacing the negative value immediately below with zero.

EVALUATION OF THE GRAB SAMPLING METHOD

A common method of sampling during reconnaissance exploration is the grab sample. For this study grab samples consist of three or four pieces of mineralized rock deemed by the sampler to be representative of the overall deposit. How well a grab sample estimates the PGM content in an outcrop is uncertain and at least partially a function of the sampler's experience. A rigorous evaluation of the variance of PGM values for grab samples is not possible without additional replicate sampling. However, an approximate comparison of grab sampling to the composited mean for line samples from each deposit is shown in figures 12 and 13. The individual assays from each split produced from a single grab sample and the mean and standard deviation of these assays as a group are each compared. The within-deposit grab sample assays indicate the variability introduced by the splitting and subsequent analyses. The agreement between the mean of the line samples and the mean of individual analyses for single grab samples is surprisingly good. For a reconnaissance-level examination of PGM values in the deposits studied here, a grab sample provides a good estimate of the mean PGM content of the outcrop. However, because the major source of variability is between sample sites on the deposit, multiple line samples from across the outcrop are absolutely necessary for a reliable estimate of the metal content. Note the significant variability of the grab sample data and mean for the Emerick prospect. A single noncomposite grab sample from a single site on the deposit is potentially misleading.
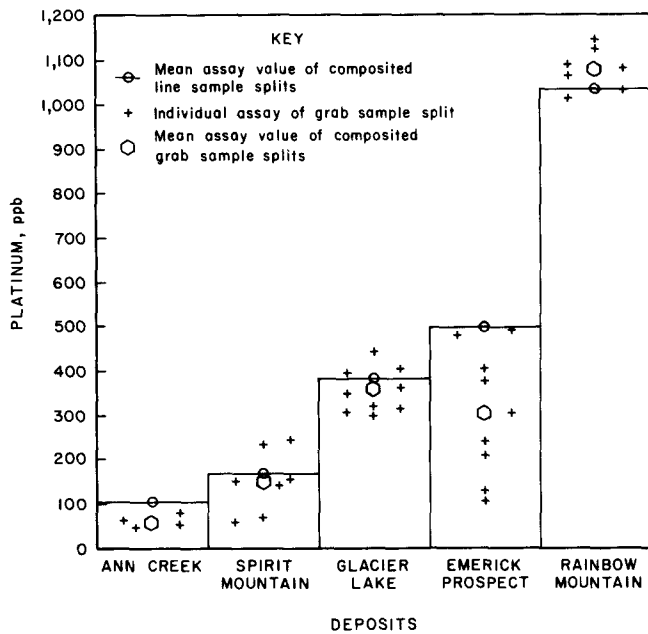

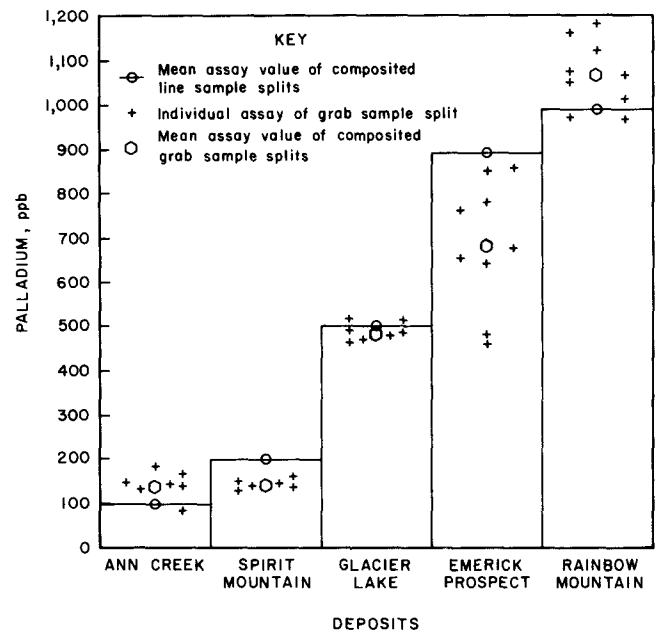FIGURE 12. - Comparison of grab sample versus line sample statistics for platinum.

FIGURE 13. - Comparison of grab sample versus line sample statistics for palladium.

DISCUSSION OF SOURCES OF VARIABILITY

The relative magnitudes of the various components of variability arising from the sampling procedure utilized may be summarized by pooling the sources of variability over all deposits. This provides a more general evaluation of variability than examining ANOVA results from individual deposits. This was not done when testing for significant sources of variability. The pooled variance components for platinum and palladium are given in table 6 using the 20- to 22-lb sample data. The 10- to 12-lb data, although an adequate sample size, were not used for these calculations because complete data for 10- to 12-lb samples were limited to only two deposits.

As previously stated, the 3- to 4-lb line samples and the grab samples indicated a higher degree of variability owing to their smaller size. The single most important source of variability indicated in table 6 is between lines within a deposit. This implies that more than one line should be taken to adequately measure PGM content. The variability between 100-g splits (crushing and splitting step) is less than that between 35-g splits (pulverizing and splitting step) for both platinum and palladium. This is an indication that the sample preparation procedure needs further scrutiny. The variability between chemical analyses is insignificant when compared to the other sources of variability present in the sampling procedure.

Assuming the sample method (line samples) and the sample size (20- to 22-lb) are constants, the estimated variance of the sample mean for platinum (ppb) per analysis is given by--

$$s_{\bar{x}}^2 = \frac{353.51 + n_A(2,485.55) + n_B n_A(2,252.74) + n_C n_B n_A(10,906.64)}{n_A n_B n_C n_D}$$

and for palladium--

$$s_{\bar{x}}^2 = \frac{248.44 + n_A(2,494.48) + n_B n_A(1,854.53) + n_C n_B n_A(11,105.75)}{n_A n_B n_C n_D}$$

where the numerical values come from tables 4 and 5

and n_A = number of analyses per 35-g split,

n_B = number of 35-g splits per 100-g split,

n_C = number of 100-g splits per line,

and n_D = number of lines.

For example, for one sampling line, reduced to one 100-g split, from which one 35-g split is taken, and one analysis is performed, the estimated variability in platinum content (ppb) is $s_{\bar{x}}^2 = 15,998.45$ (ppb)^2. That is, we would expect the value to fall within ±252.97 ppb ($\pm 2s_{\bar{x}}$) of the true average platinum content with 95-pct confidence. This calculation utilizes the pooled variance for all five deposits sampled and is therefore applicable only to the range of PGM values from these deposits. Using data presented in tables 4 and 5, the confidence intervals for PGM content can be calculated for individual deposits.

TABLE 6. - Variance components pooled over all deposits
for 20- to 22-lb samples

Source of variance	Variance component	
	Platinum	Palladium
Sample site--sampling lines A and B................	10,906.64	11,105.75
Crushing and splitting--100-g split................	2,252.74	1,854.53
Pulverizing and splitting--35-g split..............	2,485.55	2,494.48
Chemical analysis..................................	353.52	248.44

NOTE.--Platinum values based on 4 deposits (Ann Creek   excluded because of nondetected values).

CONCLUSIONS AND RECOMMENDATIONS

The purpose of this study was to evaluate the sources of variability in the existing methods of sampling and analysis for PGM, not to determine a new method for sampling an outcrop. As shown in table 6, two sources of variability stand out: the variance within a prospect based on selection of the sample site, and the pulverizing and splitting of the 35-g split of a given sample. The first is a field sampling problem, the second a laboratory sampling problem. Considering the laboratory sample preparation problem first, it is not clear what causes this variability. Sample heterogeneity introduced at this point may arise from a failure to mix the samples thoroughly or from the need to fuse four 8.75-g subsamples, instead of a single 35-g sample. This sampling preparation problem, though of less importance than the field sampling problem, needs to be addressed. In actual practice, if subsequent analyses are made of the sample pulp at a later time, it is at this step that a major source of variabilty is introduced, which leads to poor reproducibility of analyses.

Sample site variability within an outcrop is the major source of variance. This is in accord with the common observation that there is usually a very irregular distribution of PGM within an outcrop. This condition reflects the basic mode of occurrence of the PGM and is not an artifact of the sampling procedure. To minimize this variability, relatively large samples are needed.

Samples should be no smaller than 10 to 12 lb. There is, however, no significant advantage in using 20- to 22-lb samples. Replication of sample lines across the prospect is the most important method whereby the overall variance can be reduced. Small grab samples and small single-line samples offer a good estimate of the general range of PGM content but do not adequately measure the variability of the PGM in a deposit. These considerations are summarized in the following recommendations:

Take four ±3-lb continuous chip or channel samples (12 lb) or two ±12-lb samples. Replication in lines is important.

There is no reason to take more than one 100-g split from a sample, nor to analyze one 35-g split more than once.

The variability at the 35-g split stage should be reduced. This can be done by taking two 35-g splits from each 100-g split or perhaps by addressing the fusion sample size problem.

It should be noted that the variability between chemical analyses is insignificant when compared to these other sources of variability present in the sampling procedure.

To assess the PGM content of a prospect, new sampling schemes should be tried and compared with the traditional method used here. For instance, a systematic grid might work well, especially when used with the universal kriging method.

REFERENCES

1. Clifton, H. E., R. E. Hunter, F. J. Swanson, and R. L. Phillips. Sample Size and Meaningful Gold Analysis. U.S. Geol. Surv. Prof. Paper 625-C, 1969, 17 pp.

2. Cusick, M. J. (Bondar-Clegg, Inc.). Written communication, May 1984; available upon request from J. C. Barker, BuMines, Fairbanks, AK.

3. Clark, I. Practical Geostatistics. Applied Science Publishers, London, 1979, 129 pp.

4. Hanson, L. G. Bedrock Geology of the Rainbow Mountain Area, Alaska Range, Alaska. AK Div. Mines and Miner. (now AK Div. Geol. and Geophys. Surv.), Geol. Rep. 2, Nov. 1963, 82 pp.

5. Rose, A. W. Geology and Mineral Deposits of the Rainy Creek Area, Mt. Hayes Quadrangle, Alaska. AK Div. Mines and Miner. (now AK Div. Geol. and Geophys. Surv.), Geol. Rep. 14, May 1965, 49 pp.

6. _____. Geologic and Geochemical Investigations in the Eureka Creek and Rainy Creek Areas, Mt. Hayes Quadrangle, Alaska. AK Div. Mines and Miner. (now AK Div. Geol. and Geophys. Surv.), Geol. Rep. 20, June 1966, 36 pp.

7. Stout, J. H. Geology of the Eureka Creek Area, East-Central Alaska Range. AK Div. Geol. and Geophys. Surv., Geol. Rep. 46, 1976, 32 pp., 1 plate.

8. Saunders, R. H. Report on the Emerick West Delta Nickel Prospect, Mt. Hayes Quadrangle. Rep. in file of AK Div. of Mines and Miner. (now AK Div. Geol. and Geophys. Surv.), 1962, 9 pp.

9. Kingstron, J., and D. J. Miller. Nickel-Copper Prospect Near Spirit Mountain, Copper River Region, Alaska. U.S. Geol. Surv. Bull. 943-C, 1945, pp. 49-56.

10. Pierce, H. C. Exploration of Spirit Mountain Nickel Prospect, Canyon Creek, Lower Copper River Region, Alaska. BuMines RI 3913, 1946, 8 pp.

11. Herreid, G. Geology of the Spirit Mountain Nickel-Copper Prospect and Surrounding Area. AK Div. Mines and Geol. (now AK Div. Geol. and Geophys. Surv.), Geol. Rep. 40, Aug. 1970, 19 pp.

12. Miller, R. J., G. R. Winkler, R. M. O'Leary, and E. F. Cooley. Analyses of Rock, Stream Sediment, and Heavy-Mineral Concentrate Samples From the Valdez Quadrangle, Alaska. U.S. Geol. Surv. OFR 82-451, 1982, 225 pp.

13. Box, G. E. P., W. G. Hunter, and J. S. Hunter. Statistics for Experimenters: An Introduction to Design, Data Analysis, and Model Building. Wiley-Interscience, 1978.