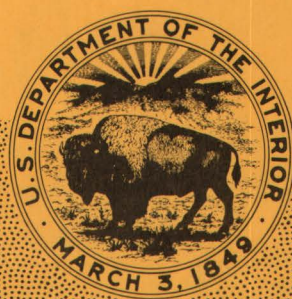


GEOLOGICAL SURVEY CIRCULAR 545



**Marine Sediment Sample
Preparation for Analysis
For Low Concentrations
Of Fine Detrital Gold**

Marine Sediment Sample Preparation for Analysis For Low Concentrations Of Fine Detrital Gold

By H. Edward Clifton, Arthur Hubert,
and R. Lawrence Phillips

GEOLOGICAL SURVEY CIRCULAR 545



United States Department of the Interior
STEWART L. UDALL, *Secretary*



Geological Survey
William T. Pecora, *Director*



First printing 1967
Second printing 1970

Free on application to the U.S. Geological Survey, Washington, D.C. 20242

CONTENTS

	Page
Abstract	1
Introduction	1
Analytical methods	2
Particle sparsity effect	2
Sample preparation	4
Examples of calculation of gold content of samples from analyses of concentrates	8
Conclusions	10
References	10

ILLUSTRATIONS

FIGURE	1. Map showing location of samples used in this study	Page 2
	2. Diagram of suggested procedure for concentrating any given quantity of marine sands	9

CONTENTS
TABLES

	Page
TABLE 1. Gold content of splits of samples of unprocessed sand compared with actual gold content determined on concentrates of these samples	3
2. Gold content of size fractions of offshore, beach, marine-terrace, and alluvial sand	5
3. Gold content of light and heavy fractions separated with methylene iodide (sp gr = 3.3)	6
4. Change in gold content in gravimetric fractions with increasing duration of heavy liquid (methylene iodide) separation of six splits of the fraction smaller than 0.124 mm from sample M660C-56	6
5. Gold content of the <0.124 mm heavy (sp gr>3.3) fraction of samples after magnetic separation	7
6. Reproducibility of analyses of concentrates, recalculated to gold content of original sample	8

MARINE SEDIMENT SAMPLE PREPARATION FOR ANALYSIS FOR LOW CONCENTRATIONS OF FINE DETRITAL GOLD

By H. Edward Clifton, Arthur Hubert, and R. Lawrence Phillips

Abstract

Analyses by atomic absorption for detrital gold in more than 2,000 beach, offshore, marine-terrace, and alluvial sands from southern Oregon have shown that the values determined from raw or unconcentrated sediment containing small amounts of gold are neither reproducible nor representative of the initial sample. This difficulty results from a "particle sparsity effect", whereby the analysis for gold in a given sample depends more upon the occurrence of random flakes of gold in the analyzed portion than upon the actual gold content of the sample.

The particle sparsity effect can largely be eliminated by preparing a gold concentrate prior to analysis. A combination of sieve, gravimetric, and magnetic separation produces a satisfactory concentrate that yields accurate and reproducible analyses. In concentrates of nearly every marine and beach sand studied, the gold occurs in the nonmagnetic fraction smaller than 0.124 mm and with a specific gravity greater than 3.3. The grain size of gold in stream sediments is somewhat more variable.

Analysis of concentrates provides a means of greatly increasing the sensitivity of the analytical technique in relation to the initial sample. Gold rarely exceeds 1 part per million in even the richest black sand analyzed; to establish the distribution of gold (and platinum) in marine sediments and its relationship to source and environmental factors, one commonly needs to know their content to the part per billion range. Analysis of a concentrate and recalculation to the value in the initial sample permits this degree of sensitivity.

INTRODUCTION

The U.S. Geological Survey, as part of its program to identify sources of supply of many minerals that are in shortage categories, initiated a geologic investigation of the origin and resource potential of marine black-sand deposits on the continental shelf of southern

Oregon (fig. 1). The gold and platinum potential of this area has long been recognized (Blake, 1854; Day and Richardson, 1906; Diller, 1914; Hornor, 1918; and Pardee, 1934); black sands are common on modern beaches and in marine terrace deposits of Pleistocene age (Griggs, 1945).

At the time of this study, routine analyses of trace amounts of platinum could not be made; therefore nearly all analytical effort in this investigation has concerned gold. Platinum, however, generally accompanies gold in the black sand, so that auriferous sands may be considered also to contain some platinum. To date, 2,000 analyses have been made for gold in offshore, beach, and marine-terrace sands and in stream sediment. The results of these analyses and their significance will be reported separately; this paper deals only with those data that apply to sample preparation.

Initial work indicated a disconcerting lack of consistency among gold values found by several analyses of a single sample. Subsequent experiments showed that analyses of gold in splits of raw sand samples generally do not accurately measure the amount of gold in the original sample. Furthermore, such analyses did not permit detection of gold in concentration smaller than 0.1 ppm, a value rarely exceeded by most beach and offshore sands. The gold content of such sands, however small, must be established in order to understand the principles of gold distribution in these environments.

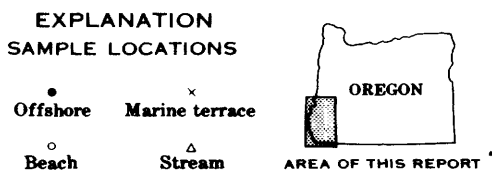
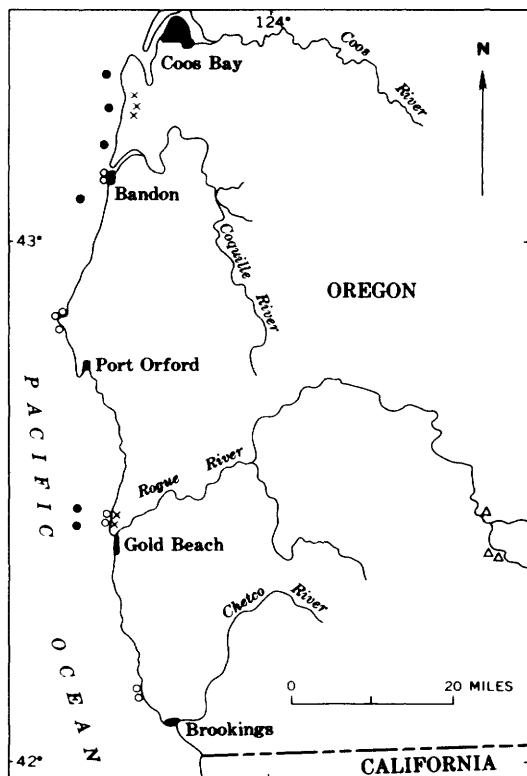


Figure 1.—Location from which samples used in this study were obtained. Each location shown represents one or more samples.

Analytical Methods

Samples were prepared for analysis either in a mobile sedimentology laboratory in the field or in the U.S. Geological Survey laboratories in Menlo Park, Calif. Nearly all the gold analyses were made by a wet chemical method (Lakin and Nakagawa, 1965) in conjunction with atomic absorption spectrophotometry, although a few were made spectrographically. Most of the atomic absorption analyses were conducted in the field in a mobile laboratory.

We gratefully acknowledge the assistance of F. J. Swanson, David D'Armond, and R. G. Winkler with the preparation of the sediment samples and the help of Alan Chleborad and Francis Michaels with the atomic absorption analysis. E. M. Baldwin and Sam Boggs, Jr., of the University of Oregon, contributed stream sediment samples for the study. Off-

shore samples were taken with a grab-type bottom sampler from the U.S. Geological Survey research vessel *Polaris* under the direction of G. A. Rusnak.

Particle Sparsity Effect

The major difficulty in obtaining a gold analysis representative of a given sample results from the sparsity of gold particles in marine sediments. A single flake of gold, 0.010–0.020 mm thick and 0.060–0.090 mm in diameter (a size and shape commonly found in beach and terrace sands) in a gram of sand constitutes about 1 ppm (part per million). This is very fine gold from a miner's standpoint; this hypothetical flake, at about 31 million particles per Troy ounce, is one-third the size described as typical flour gold (10 million particles per ounce) by Hite (1933a, p. 260) and less than one-thirty fifth as large as the fine flour (885,000 particles per ounce) of Ingersoll (1932, p. 8), but even with such fine gold only a limited number of particles are required to produce the grades (mostly less than 1 ppm) found to date in the Oregon black sands.

The analytical data can readily be misinterpreted where samples contain a limited number of gold particles. A 500-gram sample of sand containing 0.5 ppm gold in flakes weighing 5×10^{-6} g (approximately 0.03 mm thick and 0.1 mm in diameter) would contain 50 flakes. If the sample were split into 250 2-g portions, none of which contained more than a single flake of gold, 50 portions would contain one flake and the remaining 200 would contain no gold. In other words, in analyzing this sample, the chances are 80 percent that no gold will be detected; if a gold-bearing split were analyzed, its gold content would be a misleading 2.5 ppm.

This circumstance we have termed the particle sparsity effect, wherein the detrital gold content of a given sample, as determined by analysis of a split, depends not so much on the actual gold content of the sample as on whether or not a random flake occurs in the analyzed portion. Obviously, natural samples are more complicated than the hypothetical sample discussed above, as they contain several sizes of gold flakes and any one portion could readily contain more than one flake. The principle, however, of the particle sparsity effect

does apply to natural samples as noted by Pardee (1934, p. 34) and as may be seen from table 1. The gold content of splits from a single sample of unprocessed sand differed as much as 29 ppm (table 1, sample M660C-56); the analyses are not reproducible, nor is any of them representative of the initial sample.

The particle sparsity effect is most significant in samples containing relatively little gold. As the content increases, the effect will decline if particle size remains constant. It is unquestionably important in the range of concentrations that we have found in beach and offshore sand.

TABLE 1.—Gold content of splits of samples of unprocessed sand compared with actual gold content determined on concentrates of these samples

[Gold reported in parts per million. Analyses by atomic absorption unless otherwise noted]

Sample	Actual gold content	Scoop a/		Portion selection sample splitter b/		Powder c/	
		Number of analyses with the same gold content	Gold found	Number of analyses with the same gold content	Gold found	Number of analyses with the same gold content	Gold found
M660C-56-----	0.52	7	<0.1	6	<0.1	8	<0.1
		1	3.0	1	.25	1	3.0
		1	1.8	1	.89	1	.35
		1	29.	1	4.7	1	<.1
		-----	-----	1	2.9	1	1.4
55-----	.31	9	<.1	9	<.1	7	<.1
		1	1.2	1	.36	1	.55
		-----	-----	-----	-----	1	1.0
		-----	-----	-----	-----	1	2.3
51-----	.023	4	<.1	5	<.1	4	<.1
		1	.4	1	2.9	1	.4
		1	1.8	-----	-----	1	1.8
		-----	-----	-----	-----	2	<.1
67-----	.07	4	<.1	6	<.1	2	<.1
		1	1.4	-----	-----	-----	-----
		1	1.1	-----	-----	-----	-----
71-----	.075	5	<.1	6	<.1	2	<.1
		1	1.4	-----	-----	-----	-----
M660H71-24---	d/<.1	1	13.	-----	-----	1	3.0
		4	<.1	-----	-----	1	.25
		-----	-----	-----	-----	32	<.1
T-22-----	e/	1	7.0	-----	-----	-----	-----
		1	<.1	-----	-----	-----	-----
		2	f/<.2	-----	-----	-----	-----
M660C-12-----	.22	1	9.5	-----	-----	-----	-----
		1	.3	-----	-----	-----	-----
M660P-16-----	1.1	2	3.0	-----	-----	-----	-----

a/ Approximately 2-g portion taken randomly from a larger split of the initial sample using a scoop with capacity of approximately 2 g of sample.

b/ Two-g portion, weighed to 0.01 g, after being split from initial sample through a riffle-type sample splitter.

c/ Approximately 2-g portion taken randomly with a scoop from powder produced in a small ball mill from 8- to 40-g splits from the initial sample.

d/ No gold detected in concentrate. Available sample too small (28.6 g) to provide representative concentrate.

e/ No analysis of concentrate.

f/ Spectrographic analysis.

Sample Preparation

The foregoing discussion demonstrates the futility of analysis for detrital gold in small splits of unprocessed sand. Analysis of a weighed concentrate which contained all the gold in a sample would obviate this difficulty. Such a concentrate can be obtained using sample-preparation techniques such as those described by Wager and Brown (1960).

The simplest and most rapid concentrating mechanism is probably the gold pan. Panning, however, although doubtlessly an effective field expedient, presents some major difficulties. The potential for operator error is inherently high. The panning technique must be rigorously uniform from sample to sample, particularly as the smallest flecks of gold can easily be lost during panning. Ewing (1931, p. 136) compared heavy mineral concentrates taken gravimetrically from panned and unpanned samples and found discrepancies that he attributed to panning losses. Furthermore, unless the weights of the original sample and the panned residue are carefully compared, which may be difficult in the field, the gold content of the initial sample remains unknown. It seems, therefore, that panning, unless done rigorously and expertly, is a generally impractical method of concentrating gold for analysis, particularly if flour gold is abundant.

Some of the difficulties inherent in panning may be overcome by use of mechanically controlled riffles, such as a wilfley table or a micropanner (Muller, 1959). Our limited experience, however, with a wilfley table indicated that, although it effectively concentrates heavy minerals from the sand, too many heavy grains are lost to permit its use for precise quantitative separation.

A different concentrating technique is based on the physical characteristics of detrital gold in contrast to the sand in which it occurs. Because of its extremely high specific gravity ($sp\ gr = 15-19$), grains of gold are much smaller than hydraulically equivalent grains of quartz. To establish the size range of detrital gold, samples were initially dry sieved into three size fractions: 0.991–0.246 mm, 0.246–0.124 mm, and smaller than 0.124 mm. To further concentrate the gold, each fraction was separated into a heavy and a light fraction using the heavy liquid methylene iodide ($sp\ gr = 3.3$). Analysis of these fractions indicated

that in nearly every beach and offshore sand sample studied, gold occurs mostly in the smaller than 0.124 mm fraction (table 2). It should be noted, however, that in one sample (M660C-13) an appreciable amount of gold (6.2 ppm) is in the 0.246–0.124 mm fraction, whereas the finest fraction contained no gold. Another sample (M660P-1) contained 0.02 ppm gold in the 0.991–0.246 mm fraction; however, the gold content of the 2-g analyzed portion (1.5 ppm) is less than would result from the occurrence in this split of a single gold particle in this size class. Such a value may result either from adherence of tiny gold flakes to coarser sand grains, from incomplete disaggregation, or from gold inclusions within quartz grains. Where significant amounts of coarser gold are suspected, the sieve size should be increased (for example, from 0.124 to 0.177 mm) to accommodate all the gold.

The fraction smaller than 0.124 mm of a few samples known to contain gold was further split through 0.088-, 0.062-, and 0.038-mm sieves. Table 2 shows that the bulk of the gold in these samples is distributed through these size classes; the amount of gold passing through a 0.038-mm sieve was insignificant.

Consideration was given, before analysis of gravimetric concentrates, to the possibility that the fine size and flakiness of the gold might cause it to adhere to coarser sand grains and to be concentrated in the lighter fraction. Analysis of both light and heavy fractions (table 3) shows that some gold does occur in the light fraction. Nearly all samples, however, with gold in the light fraction contained much more gold in their heavy fraction. The occurrence of gold in the light fraction is undoubtedly due to incomplete separation; table 4 shows how gold shifts into the heavy fraction as the time of separation is increased. Analysis of both light and heavy fractions in several size classes indicates that, as might be expected, the finer gold is more difficult to separate gravimetrically (table 3, samples M660C-56, M660C-55). Complete separation of the gold might be facilitated by attaching a vibratory motor to the heavy-liquid separatory funnel system to provide continuous slight agitation.

Some samples, even after sieve and gravimetric concentration, contain enough magnetite and ilmenite or, locally, chromite

TABLE 2.—Gold content of size fractions of offshore, beach, marine-terrace, and alluvial sand
 [Results in parts per million. Less-than values are calculated from analysis of only the heavy fraction of the size class]

Sample	Gold content of indicated size fractions (mm)					
	0.991-0.246	0.246-0.124	0.124-0.088	0.088-0.062	0.062-0.038	<0.038
Offshore-marine sand:						
P-66-13-----	<0.02	<0.004	a/0.03	-----	-----	-----
19-----	<.02	<.005	a/ .03	-----	-----	-----
22-----	<.02	<.007	a/ .01	-----	-----	-----
27-----	<.02	<.005	a/ .02	-----	-----	-----
53-----	<.06	<.006	b/ <.02	-----	c/0.03	-----
55-----	<.08	<.004	b/ <.02	-----	c/ .08	-----
Beach sand:						
M660P-1-----	.02	<.06	a/22	-----	-----	-----
3-----	<.0009	<.0003	a/5.0	-----	-----	-----
4-----	<.008	<.05	a/17.5	-----	-----	-----
5-----	<.003	<.02	a/4.0	-----	-----	-----
6-----	<.03	<.08	a/11.0	-----	-----	-----
7-----	<.008	<.09	a/20	-----	-----	-----
8-----	<.008	<.03	a/1.8	-----	-----	-----
9-----	<.04	<.07	a/22	-----	-----	-----
M660C-13-----	<.02	6.2	a/ <.1	-----	-----	-----
14-----	<.07	<.1	a/2.4	-----	-----	-----
15-----	<.02	<.08	a/1.2	-----	-----	-----
51-----	<.07	<.06	a/ .5	-----	-----	-----
52-----	<.04	<.05	a/ .6	-----	-----	-----
66Ahp35A-----	<.13	<.05	a/ .1	-----	-----	-----
M660P-77-----	-----	-----	4.7	18	8.6	<0.1
Marine-terrace sand:						
M660C-12a-----	-----	<.09	a/7.1	-----	-----	-----
12b-----	<.02	<.08	a/1.3	-----	-----	-----
21-----	<.06	<.07	a/6.5	-----	-----	-----
23-----	<.03	<.07	a/ .4	-----	-----	-----
55-----	<.03	<.03	.8	<.0003	6.9	-----
56-----	<.03	.2	7.2	150	140	.3
Alluvial sand:						
M660EB-126-----	<.05	.03	a/ <.01	-----	-----	-----
134-----	.3	8.1	a/ <.07	-----	-----	-----
139-----	<.008	.02	a/ .1	-----	-----	-----

a/ <0.124 mm.

b/ 0.124-.062 mm.

c/ <0.062 mm.

TABLE 3.—Gold content of light and heavy fractions separated with methylene iodide (sp gr=3.3)

[Results in parts per million]

Sample	Size fraction (mm)	Light fraction (sp gr =>3.3)	Heavy fraction (sp gr =>3.3)	
M660C-46a-----	<0.124	1.0	11	
	a/ <.124	<.1	15	
b-----	<.124	<.1	8.3	
	a/ <.124	<.1	15	
c-----	<.124	<.1	16	
	a/ <.124	<.1	7.2	
d-----	<.124	4.5	8.9	
	a/ <.124	<.1	5.8	
M660C-47a-----	<.124	3.3	230	
	b-----	<.1	230	
	c-----	<.124	3.3	240
	d-----	<.124	<.1	240
M660C-12a-----	<.124	<.1	1.3	
	b-----	<.1	7.5	
	c-----	<.124	3.3	24
	d-----	<.124	<.1	6.5
	e-----	<.124	<.1	15
M660C-56-----	0.124-.088	<.1	8.0	
	.088-.062	<.1	220	
	<.062	.9	930	
M660C-56----- (recheck)	.124-.088	<.1	3.4	
	.088-.062	1.5	230	
	.062-.038	93	680	
	<.038	.35	<.1	
M660C-55-----	<.124	.27	5.2	
	.124-.088	<.1	<.1	
	.088-.062	<.1	<.1	
	<.062	60	26	

a/ Duplicate.

(Griggs, 1945) to dilute the gold below the detection level. For such samples, further concentration can be made magnetically. A hand magnet readily removes the magnetite, and a Frantz magnetic separator (Gaudin and Speden, 1943) removes ilmenite and chromite when set at 0.6 amps, with a forward slope of 25° and a side slope of 15°. Some of the gold in the southern Oregon black sands reportedly is coated with iron oxide (Pardee, 1934, p. 38). This coating, however, seems not to increase significantly the magnetic susceptibility of the gold, which occurs almost entirely in the less magnetic fraction (table 5). The occurrence of gold in the magnetic fractions of sample M660C-56a is thought to result from incomplete magnetic separation, a conclusion supported by the results of analysis of M660C-

56b, a carefully processed split of the same sample.

A few concentrates—even after sieve, gravimetric, and magnetic separation—are too large for a single analysis. Such concentrates can be split into analyzable portions with a riffle-type sample splitter. Analysis of all the portions establishes the total gold content; analysis of one provides a value presumably representative of the total concentrate. The particle sparsity effect, however, may still apply if the sample contains very little gold. Splits of such concentrates show less consistency in their values than do concentrates of large splits of a single sample (table 6). Therefore, analysis of the total concentrate provides a better gold determination than does analysis of a presumably representative split from the concentrate. It is most important that the analyst utilize the entire portion of concentrate submitted for the analysis, lest the particle sparsity effect reappear even after the most careful concentration of the gold.

A concentrating technique for marine sands is suggested in figure 2. This method requires modification depending on the size of the gold and the sorting of the sediment. Beach, offshore, and marine-terrace sand² of southern Oregon contain very little gold coarser than 0.124 mm. These sands lie tens of miles from the inland original source of the gold; therefore, their detrital gold may be expected to be very fine. In beach sand in Alaska, however, gold less than 2 miles from the source (D. M. Hopkins, oral commun., 1966) is of similar grain size. Additional data may show that the grain size of gold in beach and shelf sands is everywhere smaller than 0.124 mm.

Gold in stream sediments shows less consistency in size (table 2). The concentration

TABLE 4.—Change in gold content in gravimetric fractions with increasing duration of heavy liquid (methylene iodide) separation of six splits of the fraction smaller than 0.124 mm from sample M660C-56

	[Results in parts per million. Sample stirred after 15 and 30 minutes]					
	Change in gold content with indicated length of separation time (minutes)					
	15	30	45	60	90	120
Light fraction (sp gr <3.3)--	2.9	1.7	<0.1	<0.1	<0.1	<0.1
Heavy fraction (sp gr >3.3)--	60	35	26	36	25	34

technique, obviously, must take the size factor into account. In stream sediments, for example, the material greater than 2 mm may be eliminated by sieving, clay-size particles may be removed by washing and decanting, and the light fraction may be separated gravimetrically, thus concentrating the gold in the heavy intermediate grain-size fraction. In the Snake River, Idaho, the finest detrital gold flake observed by Hite (1933b, p. 698) was approximately 0.01 mm across and 0.002 mm thick. Such a particle would weigh about 0.0033 millionths of a gram, and 300 of these

particles in a gram of sample (or 600 in 2 g) would constitute 1 ppm, enough to eliminate the particle sparsity effect and the need for concentration. Our studies, however, have not yet established the presence of any appreciable amount of such extremely fine gold.

Similarly, the sorting of a sediment may determine the concentration technique. The well-sorted beach sands of the Oregon coasts contain very little light material in the smaller than 0.124 mm fraction. Because the gold is nearly always restricted to this size fraction,

TABLE 5.—Gold content of the < 0.124 mm heavy (sp gr>3.3) fraction of samples after magnetic separation
[Results in parts per million]

Sample	Gold content (hand-magnet separation)		Gold content of <0.124-mm heavy fraction (separation with Frantz isodynamic magnetic separator after hand- magnet separation—setting in amperes indicated)				
	Magnetic portion	Nonmagnetic portion	<0.4	0.4-0.6	0.6-1.2	1.2-1.5	>1.5
M660C-46a-----	<0.1	11	-----	-----	-----	-----	-----
	<.1	15	-----	-----	-----	-----	-----
b-----	<.1	8.2	-----	-----	-----	-----	-----
	<.1	15	-----	-----	-----	-----	-----
c-----	<.1	16	-----	-----	-----	-----	-----
	<.1	7.2	-----	-----	-----	-----	-----
d-----	<.1	8.9	-----	-----	-----	-----	-----
	<.1	5.7	-----	-----	-----	-----	-----
47a-----	<.1	230	-----	-----	-----	-----	-----
b-----	<.1	230	-----	-----	-----	-----	-----
51-----	<.1	3.5	<.1	a/1.3	-----	-----	-----
52-----	<.1	3.5	<.1	a/1.5	-----	-----	-----
55-----	<.1	1.6	<.1	a/120	-----	-----	-----
56a-----	8.5	34	5.5	a/540	-----	-----	-----
b-----	<.1	-----	<.1	<.1	3.5	470	<0.1
71-----	<.1	1.7	-----	.83	<.1	<.1	42
67a-----	<.1	6.2	<.1	<.1	<.1	5.8	<.1
b-----	<.1	-----	-----	<.1	<.1	<.1	11
c-----	<.1	-----	-----	.3	<.1	<.1	-----
d-----	<.1	-----	-----	.4	<.1	<.1	-----

a/ >0.4 amp.

TABLE 6.—*Reproducibility of analyses of concentrates, recalculated to gold content of original sample*

Sample	Gold (ppm)	Splits of concentrate	Gold (ppm)
M660C-46a-----	a/0.22	1	0.18
	-----	2	.27
b-----	a/ .27	1	.19
	-----	2	.35
c-----	a/ .26	1	.35
	-----	2	.16
d-----	a/ .18	1	.23
	-----	2	.15
47a-----	.96	-----	-----
b-----	1.1	-----	-----
c-----	1.2	-----	-----
d-----	1.2	-----	-----
55a-----	.11	-----	-----
b-----	.22	-----	-----
56a-----	a/0.15-.16	1	.20
	-----	2	.21
	-----	3	.05
	-----	4	<.03
	-----	5	.46
	-----	6	<.03
b-----	a/ .11	1	.17
	-----	2	.05
c-----	a/ .49	1	.66
	-----	2	.32
d-----	.27	-----	-----
e-----	.35	-----	-----
f-----	.25	-----	-----
g-----	.25	-----	-----
h-----	.18	-----	-----
i-----	.20	-----	-----
j-----	.27	-----	-----
57-----	a/ .44	1	.49
	-----	2	.39
58-----	a/ .25	1	.35
	-----	2	.14
M660P-77-----	a/ .049-.056	1	<.005
	-----	2	<.01
	-----	3	.24
	-----	4	<.01
	-----	5	<.01
	-----	6	<.005

a/ Based on calculation from the values of the splits of concentrates. Where less-than values exist for splits of concentrates, the averaged value given indicates maximum and minimum gold content.

sieving commonly is the only concentration required.

Sieve, gravimetric, and magnetic separations constitute an effective concentration method for gold. Reproducibility is good, as indicated by analysis of concentrates of several splits of the same original sample (table

6). The gold content of the initial sample as calculated from the analysis will be accurate, providing the concentrate contains all the gold in the sample. Examples of this calculation are shown under "Examples of calculation of gold content of samples from analyses of concentrates."

The concentration method described herein has several disadvantages. Very large samples are difficult to handle with standard laboratory facilities. The method is rather slow, although with proper facilities two technicians can readily process 30 1,000-g samples a day. Currently, we use a stack of duplicate sieves separated by insert pans in order to process several samples simultaneously. Heavy liquid separation is done in a battery of large funnels. Magnetic separation can doubtlessly be improved by using an electromagnetic separator (Holmes, 1930, p. 89), as separation by hand magnet is particularly time consuming. The method has provided a byproduct of useful information about the size of gold particles and the size distribution and heavy-mineral content of auriferous sand.

Examples of Calculation of Gold Content of Samples from Analyses of Concentrates

Example 1.—M660C-41, a sample of 368 g, yields a concentrate of 0.44 g, that, as analyzed, contains 6.6 ppm gold. What is the gold content of this sample, assuming that the concentrate contains all the gold in the sample?

Gold content of the sample = weight of the portion analyzed times its gold content (ppm) divided by the weight of the initial sample, that is, $(0.44 \times 6.6) / 368 = 0.008$ ppm.

Example 2.—M660C-39, a sample of 155 g, yields a concentrate of 4.13 g. A 2.02-g split of the concentrate, as analyzed, contains 0.7 ppm gold. What is the gold content of this sample, assuming that the concentrate contains all the gold in the sample and that the gold content of the split is representative of that of the concentrate?

Gold content of the sample = weight of the whole concentrate times the gold content of the analyzed portion divided by the weight of the initial sample, that is, $(4.13 \times 0.7) / 155 = 0.019$ ppm.

Example 3.—M660P-77, a sample of 2,530 g, yields heavy (sp gr >3.3) fractions that were analyzed for gold with the following results:

0.124–0.088 mm size class, 128 g from which six portions were split for analysis:

- a. 2.09 g, <0.1 ppm
- b. 1.22 g, <0.2 ppm
- c. 1.13 g, <0.2 ppm
- d. 1.23 g, <0.2 ppm

e. 1.27 g, 4.7 ppm

f. 1.95 g, <0.1 ppm

0.088–0.062 mm size class, 1.95 g, 18 ppm

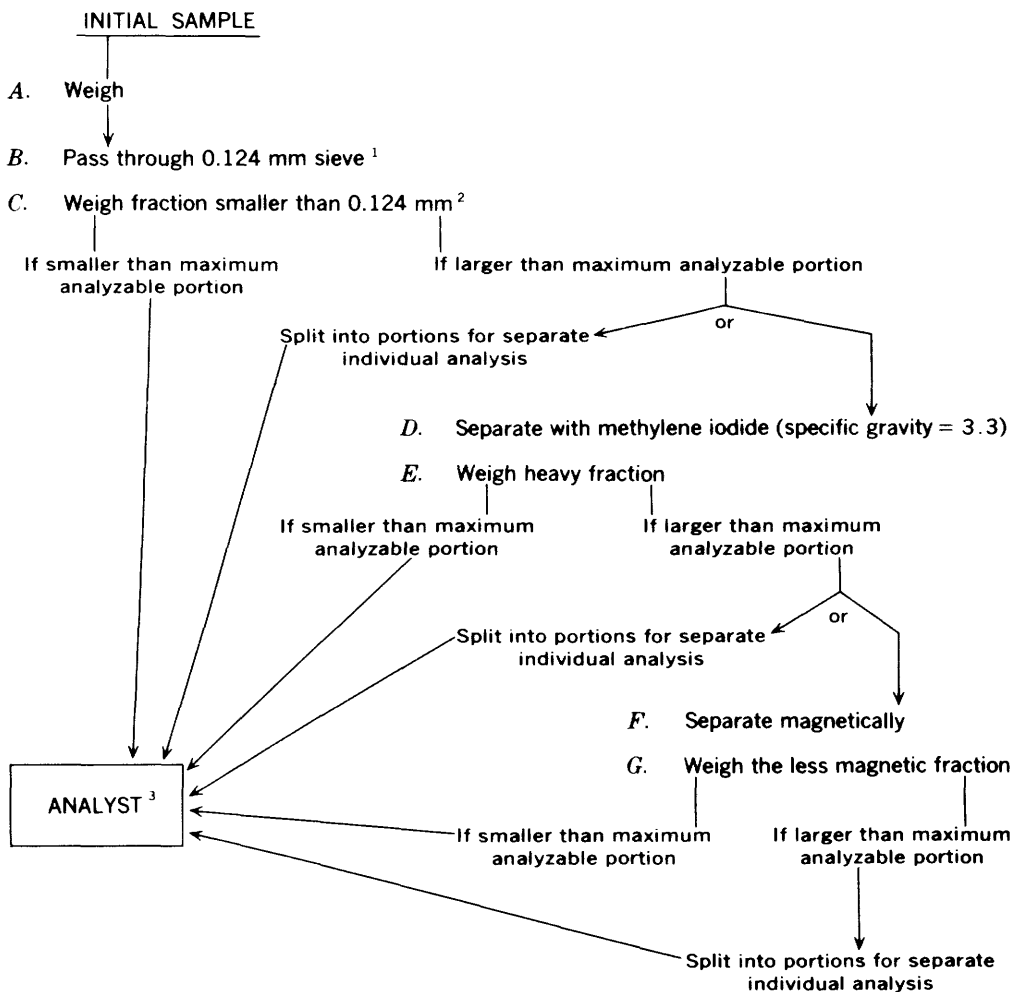
0.062–0.038 mm size class, 0.44 g, 8.6 ppm

<0.038 mm size class, 0.50 g, <0.4 ppm

What is the gold content of this sample?

Calculation of the contribution of gold from the 0.124–0.088 mm heavy fraction:

Maximum gold content of the portions analyzed = sum of the products of the weights of the portions



¹ The sieve size used in this separation should be large enough to allow passage of all the gold particles in the sample.

² If sieving yields only a small fraction, resieving through a slightly coarser sieve may be advisable before submittal for analysis or before further processing.

³ It must be recognized that, where different concentrating techniques are used, the analytical results may differ accordingly.

Figure 2.—Suggested procedure for concentrating any given quantity of marine (beach, offshore, and marine-terrace) sands. Quantity of sample used for analysis may be modified to conform with analytical technique.

analyzed times their maximum gold content divided by the sum of the weights of the portions analyzed, that is, $[(2.09 \times 0.1) + (1.22 \times 0.2) + (1.13 \times 0.2) + (1.23 \times 0.2) + (1.27 \times 4.7) + (1.95 \times 0.1)] / [2.09 + 1.22 + 1.13 + 1.23 + 1.27 + 1.95] = 7.09 / 8.89 = 0.80$ ppm.

Minimum gold content of the portions analyzed = sum of the products of the weights of the portions analyzed times their minimum gold content divided by the sum of the weights of the portions analyzed, that is, $(1.27 \times 4.7) / (2.09 + 1.22 + 1.13 + 1.23 + 1.27 + 1.95) = 5.97 / 8.89 = 0.68$ ppm.

Assuming the gold content of the portions analyzed to be representative of the whole 0.124–0.088 mm heavy fraction, this fraction contains between 0.80 and 0.68 ppm gold. The contribution of this fraction to the gold in the total sample may be calculated as follows:

Contribution of gold from a fraction to the total gold content of the sample = weight of the fraction times its gold content divided by the weight of the total sample, that is, $(128 \times 0.80) / 2,530 = 0.041$ ppm (maximum), and $(128 \times 0.68) / 2,530 = 0.34$ ppm (minimum)

Similarly the contributions of the other fractions are as follows:

0.088–0.062 mm heavy fraction = $(1.95 \times 18) / 2,530 = 0.014$ ppm

0.062–0.038 mm heavy fraction = $(0.44 \times 8.6) / 2,530 = 0.001$ ppm

<0.038 mm heavy fraction maximum contribution is insignificant (0.0001 ppm)

The gold content of the initial sample is the sum of the contributions from the different fractions:

Maximum = $0.04 + 0.014 + 0.001$ ppm = 0.055 ppm

Minimum = $0.034 + 0.014 + 0.001$ ppm = 0.049 ppm

The gold content of this sample is between 0.055 and 0.049 ppm.

Conclusions

Analyses of splits of raw samples containing low concentrations of detrital gold are not

reproducible, nor in general is any single analysis representative of the gold content of the sample. Reliable analysis, however, may be made on concentrates. The method described, which combines sieve, gravimetric, and magnetic separations, has generally been adequate for beach and offshore sands. With modification, it may prove effective for analysis of stream sediments.

At the time the concentrating method was being developed, the size of the analyzed portion was restricted to 2 g. Presently the analytical technique is being modified to accommodate 10-g samples (H. W. Lakin, oral commun., 1967). This increase greatly facilitates the concentrating procedure; for example, it will be possible to quickly concentrate a sample to 50 g which can be split into five separately analyzable portions.

The described concentrating method works well in the field within a mobile sedimentology laboratory and requires no highly specialized equipment. Other methods will almost certainly be required to concentrate detrital gold from sedimentary rocks. Techniques such as flotation or electrostatic separation may prove to be highly practical. Industrial-scale facilities may provide the quickest and most convenient means for concentration, particularly for large samples.

Our data are taken only from analyses for detrital gold. The principle of the particle sparsity effect, however, is certain to apply to other minerals of high specific gravity, such as platinum, which will likewise require concentration of the initial sample.

This paper deals only with the problem of obtaining an analysis that is representative of the gold content of a given sample. The problem of taking meaningful samples, of course, remains with the geologist.

References

- Blake, W. P., 1854, On gold and platinum of Cape Blanco [California]: *Am. Jour. Sci.*, 2d ser., v. 18, p. 156.
- Day, D. T., and Richardson, R. F., 1906, [Useful minerals in the] Black sands of the Pacific slope: *U.S. Geol. Survey Mineral Resources*, 1905, p. 1175–1246.
- Diller, J. S., 1914, Mineral resources of southwestern Oregon: *U.S. Geol. Survey Bull.* 546, 147 p.
- Ewing, C. J. C., 1931, A comparison of the methods of heavy mineral separation: *Geol. Mag. [Great Britain]*, v. 68, p. 136–140.
- Gaudin, A. M., and Spedden, H. R., 1943, *Magnetic*

- separation of sulphide minerals: Am. Inst. Mining Metall. Engineers Tech. Pub. 1549, 13 p.
- Griggs, A. B., 1945, Chromite-bearing sands of the southern part of the coast of Oregon: U.S. Geol. Survey Bull. 945-E, p. 113-150.
- Hite, T. H., 1933a, Fine gold and platinum of Snake River, Idaho: Econ. Geology, v. 28, no. 3, p. 256-265.
- 1933b, Special features of fine gold from Snake River, Idaho: Econ. Geology, v. 28, no. 7, p. 686-691.
- Holmes, Arthur, 1930, Petrographic methods and calculations; with some examples of results achieved, rev. ed.: New York, D. Van Nostrand Co., Inc., 516 p.
- Hornor, R. R., 1918, Notes on the black sand deposits of southern Oregon and northern California: U.S. Bur. Mines Tech. Paper 196, 42 p.
- Ingersoll, G. E., 1932, Small scale methods of placer mining and placer mining districts of Washington and Oregon: Washington State Coll. Monthly Bull. (Eng. Expt. Sta., Eng. Bull. 43, 1933), v. 15, no. 6, 59 p.
- Lakin, H. W., and Nakagawa, H. M., 1965, A spectrophotometric method for the determination of traces of gold in geologic materials, in Geological Survey research 1965: U.S. Geol. Survey Prof. Paper 525-C, p. C168-C171.
- Miesch, A. T., 1967, Chart showing number of particles of metal expected per 5-pound sample for various levels of grade and average particle size (regardless of the spatial distribution of particles): U.S. Geol. Survey open-file report.
- Muller, L. D., 1959, The micropanner—an apparatus for the gravity concentration of small quantities of materials: Inst. Mining and Metallurgy [London] Trans., v. 68, pt. 1, p. 1-7.
- Pardee, J. T., 1934, Beach placers of the Oregon coast: U.S. Geol. Survey Circ. 8, 41 p.
- Wager, L. R., and Brown, G. M., 1960, Collection and preparation of material for analysis, Chap. 2, in Smales, A. A., and Wager, L. R., eds., Methods in geochemistry: New York, Interscience Publishers, Inc., p. 4-32.

