# USGS Rock Standards, III: Manganese-Nodule Reference Samples USGS-Nod-A-1 and USGS-Nod-P-1

GEOLOGICAL SURVEY PROFESSIONAL PAPER 1155



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By F. J. FLANAGAN and DAVID GOTTFRIED

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Analytical data by various techniques and best estimates for some elements in two manganese-nodule reference samples



#### UNITED STATES DEPARTMENT OF THE INTERIOR

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			0

## **CONVERSION FACTORS**

Metric unit	Inch-Po	ound equivalent	Metric unit	Inch-P	ound equivalent	
	Length	L	Specific combinations—Continued			
millimeter (mm) meter (m) kilometer (km)	$\begin{array}{rrrr} = & 0.03937 \\ = & 3.28 \\ = & .62 \end{array}$	inch (in) feet (ft) mile (mi)	liter per second (L/s) cubic meter per second per square kilometer	= .0353 = 91.47	cubic foot per second cubic feet per second per square mile [(ft <sup>3</sup> /s)/mi <sup>2</sup> ]	
	Area		meter per day (m/d)	= 3.28	feet per day (hydraulic conductivity) (ft/d)	
square meter (m <sup>2</sup> ) square kilometer (km <sup>2</sup> )	= 10.76 = .386	square feet (ft <sup>2</sup> ) square mile (mi <sup>2</sup> )	meter per kilometer (m/km)	= 5.28	feet per mile (ft/mi)	
hectare (ha)	<u>= 2.47</u>	acres	kilometer per hour (km/h)	= .9113	foot per second (ft/s)	
	volume	e	meter per second (m/s)	= 3.28	feet per second	
cubic centimeter (cm <sup>3</sup> ) liter (L)	= 0.061 = 61.03	cubic inch (in <sup>3</sup> ) cubic inches	meter squared per day (m <sup>2</sup> /d)	= 10.764	feet squared per day (ft²/d) (transmissivity)	
cubic meter (m <sup>3</sup> ) cubic meter	= 35.31 = .00081	cubic feet (ft <sup>3</sup> ) acre-foot (acre-ft)	cubic meter per second (m <sup>3</sup> /s)	= 22.826	million gallons per day (Mgal/d)	
liter	= 2.113 = 1.06	acre-leet pints (pt) quarts (qt)	cubic meter per minute (m <sup>3</sup> /min)	=264.2	gallons per minute (gal/min)	
liter	= .26	gallon (gal)	liter per second (L/s)	= 15.85	gallons per minute	
cubic meter	= .00026	million gallons (Mgal or 10 <sup>6</sup> gal)	liter per second per meter [(L/s)/m]	= 4.83	gallons per minute per foot [(gal/min)/ft]	
			kilometer per hour (km/h)	= .62	mile per hour (mi/h)	
	weight	· · · · · · · · · · · · · · · · · · ·	meter per second (m/s)	= 2.237	miles per hour	
gram (g) gram	= 0.035 = .0022	ounce, avoirdupois (oz avdp) pound, avoirdupois (lb avdp)	gram per cubic centimeter (g/cm <sup>3</sup> )	= 62.43	pounds per cubic foot (lb/ft <sup>3</sup> )	
metric tons (t) metric tons	= 1.102 = 0.9842	tons, short $(2,000 \text{ lb})$ ton, long $(2,240 \text{ lb})$	gram per square centimeter (g/cm <sup>2</sup> )	= 2.048	pounds per square foot (lb/ft <sup>2</sup> )	
S	pecific comb	inations	gram per square centimeter	= .0142	pound per square inch (lb/in <sup>2</sup> )	
kilogram per square centimeter (kg/cm <sup>2</sup> )	= 0.96	atmosphere (atm)		Temperat	ure	
kilogram per square centimeter	= .98	bar (0.9869 atm)	degree Celsius (°C)	= 1.8	degrees Fahrenheit (°F)	
cubic meter per second (m <sup>3</sup> /s)	= 35.3	cubic feet per second (ft <sup>3</sup> /s)	degrees Celsius (temperature)	$=$ [(1.8 $\times$ °C	)+32] degrees Fahrenheit	

IV

### USGS ROCK STANDARDS, III: MANGANESE-NODULE REFERENCE SAMPLES USGS-NOD-A-1 AND USGS-NOD-P-1

By F. J. FLANAGAN and DAVID GOTTFRIED

#### ABSTRACT

Restricted amounts of manganese nodules from the Atlantic and Pacific Oceans have been processed as reference samples USGS-Nod-A-1 and USGS-Nod-P-1, respectively. Analysts used various analytical methods and the experimental design with the bottles of sample as the single variable of classification simultaneously to establish the compositions of the samples and to demonstrate the homogeneity of the powdered material. At the 5-percent probability level, 95.2 percent of F tests for elements in Nod-A-1 and 96.6 percent of tests for those in Nod-P-1 are not significant, and these elements are inferred to be homogeneously distributed. Best estimates were obtained by the sequential procedure of (1)determining by Cochran's test which of k sets of data have a common variance, (2) calculating from this variance the standard deviation of the mean of six determinations, and (3) using this deviation to calculate the Studentized range to decide which means could have been derived from the same population mean. Present best estimates, in percentages, for elements of potential economic interest are:

	USGS-Nod-A-1	USGS-Nod-P-1
Cu	0.110	1.15
Ni	.636	1.337
Co	.311	.224
Mn	18.54	29.14
Pt(ppm)	.45	.12

#### **INTRODUCTION**

Various aspects about manganese nodules on the sea floor have occupied the attention of mineral economists, economic geologists, and analysts for several decades. Mineral economists and economic geologists consider the related questions of whether the nodules are so distributed on the ocean floor that they may be readily mined and whether such elements as manganese, nickel, copper, and cobalt are present in sufficient amounts that the mining of the nodules and the recovery of these elements may be economically feasible. The chemical characteristics of manganese nodules, a source of scientific interest since their discovery during the voyage of the Challenger a century ago, have been the subject of reports in numerous journals, and data published through August 1973 have been compiled by Monget and others (1976). Texts concerned with the several aspects about manganese nodules have been published by Mero (1965) and Glasby (1977).

The analysts using most chemical and physical techniques may be classified roughly into those who study the distribution of elements within individual nodules (studies that lead to theories about nodule formation) and those who are interested in the determination of several elements in a powdered nodule, the interest being either scientific or economic. The two classes of analysts, whose concerns are not mutually exclusive, also share the lack of a sufficient quantity of a nodule sample that may be processed as a natural reference material. The analysts therefore must resort to the use of pure chemicals, either as solids or in solution, or must use portions of the scarce analyzed nodules in their possession.

The availability of a large sample of manganese nodules that may be processed as a reference sample may be no panacea, because the variation in analytical data among analysts and among techniques may be as severe as those in data for rock reference samples by spectroscopic techniques (Flanagan, 1979). Such possible wide variation in data may have prompted the Kennecott Copper Corp. to prepare a nodule sample to which the number GRLD-126 was assigned (Raab, oral commun., 1974). This sample, intended to test interlaboratory determinations of several elements, was analyzed by six or seven laboratories, but a description of the sample and the data have not been published. Several laboratories that participated in the analyses use the sample as a provisional reference material.

Despite the suggestion that reference samples of manganese nodules be prepared (Flanagan, 1974, p. 1741), no such samples were being prepared by late summer 1975, to our knowledge. We therefore obtained about 100 pounds of nodules from each of two oceans (Atlantic and Pacific) from Deepsea Ventures, Inc., to whom we are indebted, and prepared them as reference samples. The sample from the Atlantic Ocean, processed as USGS-Nod-A-1, was taken from the Blake Plateau, and the other sample, now numbered USGS-Nod-P-1, was taken from the region of the Pacific Ocean of current interest in nodules. More precise locations for the samples are:

Location	USGS-Nod-A-1	USGS-Nod-P-1
Latitude	31°02′ N.	14°50' N.
Longitude	78°22′ W.	124°28' W.
Depth meters	788	4,340

#### PREPARATION OF SAMPLES

The samples were prepared by the procedure normally used for rock samples (Flanagan, 1967), with the following exception: The material of either nodule sample, after being processed through a roller crusher, was dried overnight in an oven at about  $150^{\circ}$  F ( $65^{\circ}$  C) before being placed in a ball mill. Soluble sea salts were not removed by washing the large crushed nodule samples with water. Hence, some portion of the sodium and chlorine data reported for either sample may be due to the sea salts.

While one sample was being processed, the cover used to contain the nodule material in the mill was not secured in place until processing was resumed on the next day. The partly powdered material apparently absorbed sufficient atmospheric moisture overnight to form a cementlike mixture, which was deposited as layers on the vertical end pieces of the mill when it was operated. The layers, about 4 cm thick, were removed with a geologist's pick, broken, redried, and processed again in the mill. In cross section, broken pieces of the layers showed the variegated colors from black through purple to dark brown that are characteristic of manganese oxide materials.

One analyst, M. Saunders (table 24), later noted that both dried nodule samples rapidly absorbed moisture during weighing. When weighed portions of either sample were exposed to the atmosphere overnight, the portions absorbed about 10 percent moisture.

## EXPERIMENTAL DESIGN FOR THE DETERMINATIONS

The analysts were expected to use the experimental design, with a single variable of classification, that had been used almost exclusively in U.S. Geological Survey Professional Paper 840 (Flanagan, 1976). The variable of classification would be three bottles of one nodule sample, and the analysts were to make determinations of an element on two portions from each bottle. The determinations among the bottles were to be made in random order.

Three bottles of each nodule sample were packaged as a set, and 22 such sets were sent to laboratories, both within and outside the Geological Survey, that had volunteered to contribute data. Data from those who reported are shown in the several tables, and their contributions are gratefully acknowledged.

Analysts were requested to remove the caps from the bottles of nodule samples and to place the bottles in an oven at  $110^{\circ}$  C for 24 hours before weighing portions for analysis. Those who were to use chemical procedures were requested to make their determinations on portions for which the entire sample was put into solution, and not just on the acid-soluble portion, so that chemical data would be directly comparable with data obtained by physical methods, such as optical-emission spectroscopy, X-ray-fluorescence spectroscopy, or instrumental neutron-activation analysis.

Not all analysts made the determinations as requested, because they lacked time or misunderstood the purpose of the experimental design; as a result, a few tables are not directly comparable with those of the majority. The tables are therefore presented in four categories, and tables within a category are randomized where possible. The categories are listed as follows:

- 1. Semiquantitative spectrographic analysis (see discussion below).
- 2. Data by analysts or organizations who made the determinations in the manner requested.
- 3. Data by those who may have misinterpreted the intent of the experimental design and who therefore introduced other variance components into the data.
- 4. Data by those who, principally because of a lack of time, were unable to complete the set of data requested.

#### SEMIQUANTITATIVE SPECTROCHEMICAL ANALYSIS AND HOMOGENEITY

The data in table 1—estimates derived from semiquantitative spectrochemical determinations of several elements in the samples—are discussed briefly. The USGS rocks first released in 1964 and for which 

 TABLE 1.—Estimates from the analysis of variance of computerized semiquantitative spectrochemical determinations of trace-element data for USGS-Nod-A-1 and USGS-Nod-P-1

[Analyst: N. Rait, U.S. Geological Survey. Determinations, in parts per million. d.f., degrees of freedom; S, significant; NS, not significant; Neg., negative bottle variance obtained; F ratios tested at F<sub>0.95</sub> or the fractile indicated in parentheses]

			Nod-	- <b>A</b> -1			No	d-P-1	
	-		Standard	deviation			Standar	d deviation	
	Element	Mean	Bottle (d.f.=2)	Error (d.f.=3)	F ratio	Mean	Bottle (d.f.=2)	Error (d.f.=3)	F ratio
в.		94	Neg.	19	0.24 NS	120	2.9	5.8	1.5 NS
Ba		978	Neg.	150	.08 NS	1.038	136	53	14.1 NS(0.975)
Be		8.5	.16	1.37	1.03 NS	1.5	.67	.15	41.9 S(0.99)
Ce		930	Neg.	104	.26 NS	278	49	43	3.57  NS
Co		1,093	$3\overline{4}$	70	1.47 NS	1,100			
$\mathbf{Cr}$		14	Neg.	1.9	.62 NS	11.8	.81	.91	2.60 NS
Cu		807	21	102	1.08 NS	>3,200			
Eu		4.1	Neg.	.99	.29 NS	<sup>1</sup> 3.3			
La		152	Neg.	14.7	.54 NS	107	8	10	2.21  NS
Mn		177,000	Neg.	23,100	.50 NS	252,000	20,200	21,200	2.81 NS
Mo		226	13	<b>´</b> 19	1.95 NS	718	74	41	7.55 NS
Nd		101	1.4	21	1.01 NS	<sup>1</sup> 82.6			
Ni		3,150	264	122	10.3 NS(0.975)	14,170	1,000	1,470	1.92 NS
Pb		788	Neg.	115	.36 NS	470	Neg.	35	28.38 NS(0.99)
Sc		13	Neg.	1.7	.39 NS	7.6	1.9	.78	12.35 NS (0.975)
$\mathbf{Sr}$		1,062	Neg.	110	.07 NS	332	<b>76</b>	<b>44</b>	6.93 NS
Tl		148	Neg.	27	.82 NS	265	23	12	8.33 NS
ν.		261	Neg.	27	.56 NS	265	17	13	4.36 US
Υ.		138	Neg.	18	.21 NS	55	16	5.0	20.86 NS(0.99)
Yb		16	Neg.	2.6	.22 NS	8.6	3.0	.79	29.01 NS(0.99)
Zn		460	33	15	10.53 NS (0.975)	1,530	119	58	9.50 NS
Zr		498	2.9	64	1.00 NS	260	100	24	36.79 S(0.99)

<sup>1</sup> Average of 5 determinations.

there are two compilations of data (Flanagan, 1969, 1976) are now used to supplement the original spectrographic standards made to approximate the composition of the "average" silicate (Myers and others, 1961). Neither set of standards even remotely approximates the composition of manganese nodules, and because of the effects of the different matrices, spectrographic estimates for the nodules could be expected to deviate from any best values subsequently calculated.

Nevertheless, the data in table 1 serve a useful purpose. Because the size of the sample taken for the spectrographic technique is small, these data may serve as an initial indication of the homogeneity of the material. If the samples are homogeneous for this techniuge, then as a first approximation they should be homogeneous for those methods for which a larger portion of sample is taken for analysis. The homogeneity of the material does not depend on the closeness of the data to any "true" value but on the variation between and within bottles of a sample. Thus, if for a given element the F ratiothe variation attributable to bottle means divided by the variation within these bottles—is not larger than the value listed in the table for the 95-percent fractile of the F distribution with the appropriate degrees of freedom, one may infer that the element is homogeneously distributed among the bottles.

The conclusions resulting from the analysis of variance of the semiquantitative spectrographic determinations that have the form of quantitative data are of some interest. Of the 22 elements for which complete sets of data are available for USGS-Nod-A-1, the calculated F ratios for 20 sets of data are not significantly larger than the allowable value of  $F_{0.95}(d.f. 2,3) = 9.55$ , and the two remaining F ratios are not significant when tested against  $F_{0.975}(d.f. 2,3) = 16.0$ . We may conclude on the basis of these spectrographic data that the elements determined are homogeneously distributed among the bottles.

Complete sets of data for 19 elements are available for USGS-Nod-P-1. As the six determinations for cobalt are identical, no F test could be made, and cobalt is distributed homogeneously. The calculated F ratio is not significant at  $F_{0.95}$  for 11 elements and at  $F_{0.975}$  for 2 others, and these elements may be inferred to be homogeneously distributed.

Of the remaining five elements for which sets of data are complete, the F ratios for three elements do not equal or exceed the allowable value for  $F_{0.99}$  (d.f. 2,3), and ratios for two elements exceed this value. Because the allowable ratio, 30.8, is rather

large, the five elements appear to be heterogeneously distributed.

Perhaps because of the semiquantitative nature of the technique, we should not be unduly worried by the five conclusions of heterogeneity, but some brief discussion is justified. Samples to be analyzed routinely by the technique are never dried, and the powdered nodule samples were also not dried. Furthermore, the nodules from the Pacific were partly covered with a thin coating of gray pelagic clay. This clay was not removed from the nodules, and the small amount of clay was incorporated in the ground sample. The tendency of both the nodule material and the clay to absorb atmosphere moisture and the tendency of clays to absorb cations may be partly responsible for some of the demonstrated heterogeneity.

#### **TESTS FOR HOMOGENEITY**

The elements silicon through manganese were reported as percentage of either the oxide or the element, probably because no method of reporting had been specified. The elements reported as the oxides were reduced by the appropriate gravimetric factor to percentages of the elements before the analysis of variance. Such transformations from oxide to element also reduce the variation among the determinations of an element. Cobalt, copper, and nickel are considered as minor elements in the nodules, and data are listed therefore as percentages because their contents in the samples equal or exceed 0.1 percent. Elements usually considered as trace elements in rocks but reported as percentages were converted to parts per million by multiplying by 10<sup>4</sup> and adding nonsignificant zeros where necessary; these data were used in the calculations for the analysis of variance and for the best values.

The data in tables 7 through 21 presented no special problems, and the calculations of the analysis of variance were made on a laboratory calculator for which a program was available. The data in tables 22 and 23 also presented no special problems, as one analyst reported averages of two determinations and the other made determinations on a single portion from each bottle of sample; these data cannot be used in the calculations. The data in tables 24 and 25 are rather complete comprehensive analyses of each sample, and these single determinations also cannot be used. However, the data in tables 26 to 29 were presented in a form that required a change in the analysis of variance.

The analyst who contributed the data shown in tables 26 and 27 apparently planned to make a single determination on a portion from each bottle of both samples on a single day and then repeat the procedure on two other days. However, the solution for the single determinations for bottle 2 of USGS-Nod-P-1 was contaminated on the first day, and data were therefore not reported. An extra set of determinations on a portion from bottle 2 was reported for the third day. The analyst, by his decision to make determinations on three days, added another variable of classification (days) to the original design with a single variable of classification (the bottles). As the extra determinations for bottle 2 were made on day 3, these determinations could not be legitimately substituted for the missing determinations for day 1. We therefore decided to omit the two determinations on day 1 and the extra determination on day 3 and to treat the remaining data as a two-way analysis of variance, with the bottles and the days as the two variables of classification. The results of the analysis of variance reflect the significance of these variables of classification. Although the determinations for USGS-Nod-A-1 were completed according to the plan of the analyst, we decided to treat both samples similarly, and the determinations for Nod-A-1 on days 2 and 3 were also used in the two-way analysis of variance and in the calculations for best estimates.

The data in tables 28 and 29 presented a similar problem. The analysts for these determinations had intended to make the determinations in two "runs," but some determinations were delayed for about two months. To simplify matters, we ignored the delay and treated the data as a two-way design with bottles of sample and "runs" as the two variables of classification.

In other tables of data, one or both determinations for a bottle of sample were missing in a few places. Such missing data reduced the number of bottles to two, and the changes in degrees of freedom for both bottles and for error are given in footnotes. Where data are missing, the laboratory means and variances were not used in the calculations of best values. Data reported as less than some lower limit were treated the same as missing data. Data in the form < x belong to the ordinal scale of measurement (Stevens, 1946) and cannot be treated by the same mathematical or statistical procedures as the ordinary quantitative data that are classified as the ratio scale of measurement.

The conclusions resulting from the F tests in the analysis of variance for the quantitative data con-

firm the conclusions from similar tests with the semiquantitative spectrochemical data. Table 2 shows the frequencies, by element and by the fractile of the F distribution, for the significance or non-significance of the F ratios for those quantitative data for which the analysis of variance could be made. The proportion of F ratios not significant at  $F_{0.95}$  (118/124=0.952 for USGS-Nod-A-1, and 115/119=0.966 for USGS-Nod-P-1) furnish convincing evidence for both samples that most elements are indeed homogeneously distributed among the bottles.

#### **ESTIMATION OF BEST VALUES**

"Best" values for elements or oxides in rock reference samples have been derived by many (Abbey, 1977; Christie and Alfsen, 1977; Ellis and others, 1977; Flanagan, 1969, 1976; Govindaraju and de la Roche, 1977; and Sutarno and Faye, 1975), but objections can be made to some methods. Deriving best values from large compilations of data is tedious; calculating them where the data are reported in equivalent form is much easier. We decided to derive best values for the data for the nodules by a procedure involving three steps:

- 1. Determine which of the k sets of data have a common variance.
- 2. From this common variance, calculate the standard deviation of the means of the n determinations.
- 3. Use this standard deviation to obtain the Studentized range to determine which of the

Element	U	USGS-Nod-P-1						
	<f<sub>0.95</f<sub>	<f0.975< th=""><th>&lt;<b>F</b>0.99</th><th>&gt;F0.99</th><th><f0.05< th=""><th><f0.975< th=""><th><f 0.99<="" th=""><th>&gt;F0.99</th></f></th></f0.975<></th></f0.05<></th></f0.975<>	< <b>F</b> 0.99	>F0.99	<f0.05< th=""><th><f0.975< th=""><th><f 0.99<="" th=""><th>&gt;F0.99</th></f></th></f0.975<></th></f0.05<>	<f0.975< th=""><th><f 0.99<="" th=""><th>&gt;F0.99</th></f></th></f0.975<>	<f 0.99<="" th=""><th>&gt;F0.99</th></f>	>F0.99
i	5				5		_	
1	6	-	-	-	ĕ	-	-	-
1	0	-	-	-	0	ĩ	-	-
e	8	-	-	-	1	T	-	-
lg	4	-	1	-	5	-	-	-
a	5	_	_	_	4	1	-	-
a	$\tilde{2}$	_	-	-	2	_	_	_
	-	-		-	-	_	-	_
	5	-	1	-	6	-	-	-
1	4	-	-	-	3	-	-	-
	4	_	-	_	4	_	_	-
[n	6		1	_	6	1		
0	ŏ	-	+	-	ě	-	-	-
· · · · · · · · · · · · · · · · · · ·	9	-	-	-	0	-	-	-
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(i	6				5			
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<b></b>	110							
Total	118	1	4	1	115	4	U	U

TABLE 2.—Number of calculated F ratios smaller or larger than the fractile of the F distribution indicated

several means can be considered to have been derived from the same population mean. We then consider the average of the several means so selected as the best estimate.

6

The question of which of the k sets of data, each with n determinations, have a common variance was resolved with Cochran's test for the homogeneity of variances (a brief discussion and tables are available in Dixon and Massey, 1951). Sample variances, with n-1=5 degrees of freedom, were calculated for all sets of data that included six determinations of an element by each of the several laboratories. The variances of the k groups of data, omitting the factor of the technique used, were listed in a column from the least to the greatest variance. Starting with the third least variance (the two lowest variances may be tested, where necessary, by the F ratio), the sums to the 3rd, 4th . . . kth variances  $(s^2)$  are listed in an adjacent column; Cochran's test, (Largest  $s^2$ )/(Sum  $s^2$ ), is then made with the largest variance. Because analysts did not use methods of equal precision for the same element, the kth (the greatest) variance can be a very significant part of the sum of the k-1 smaller variances, as shown in the tabulation for the calculations for cobalt in USGS-Nod-A-1. Many texts contain tables at probabilities p=0.05 and p=0.01for Cochran's test, where the allowable values are for k variances, each with  $\nu$  degrees of freedom. We used the test iteratively until a homogeneous set of variances was reached. For several elements in the samples, we accepted a test that was not significant at the greater ratios for p=0.01 so that a set of three or more homogeneous variances might be available.

No	Variances	Cumulative sum of	Largest $s^2$	Significant $(S)$ or		
	(×10 <sup>6</sup> )	$(\times 10^6)$ variances Sum $s^2$ ( $(\times 10^6)$		p = 0.05	p=0.01	
9	1,160	1,309	0.8862	S	S	
8	<b>97</b>	<b>´149</b>	.6510	ŝ	ŝ	
7	17	52	.3269	NS		
6	15	35				
5	8	20				
4	7	12				
3	3	5				
2	1					
1	1					

In the tabulation of the variances for cobalt the ratio of the seventh variance over the sum of the first seven  $(17 \times 10^{-6}/52 \times 10^{-6})$  does not exceed the allowable ratio for Cochran's test at p=0.05. The calculated ratio is not significant, and the lowest seven variances are accepted as a homogeneous set.

From this homogeneous set of variances, we then

calculate the standard deviation of the means of the n (=6) determinations. In calculations where the numbers of determinations are unequal, usually the individual variances multiplied by their respective degrees of freedom are summed, and the sum thus obtained is divided by the sum of the degrees of freedom. The procedure is less complicated where variances are calculated from equal numbers of determinations.

In the tabulation for the cobalt variances, the sum of the seven smaller variances,  $52 \times 10^{-6}$ , is divided by the number of variances, 7, to obtain the average of the set of homogeneous variances. When this result,  $7.4 \times 10^{-6}$ , is divided by 6, the number of determinations for each variance, and the square root is taken, we obtain  $1.11 \times 10^{-3}$  as the standard deviation of the mean of six determinations. As each variance has 5 degrees of freedom, we have 35 degrees of freedom associated with this standard deviation of the mean. Standard deviations for the other elements are calculated similarly.

The averages of the data are then tested by the Studentized range to determine if all or some of the means form a homogeneous group. The means of the several sets of data are ordered, the means being taken for the purpose of discrimination to more decimal places than are generally warranted. The order for the cobalt averages, each calculated to four digits, is 0.3900, 0.3363, 0.3265, 0.3215, 0.3128, 0.3127, 0.3080, 0.2813, and 0.2527. We may see that this ordered set of means shows appreciable variation and that we may need to use the procedure iteratively. The range of these nine averages is then divided by the standard deviation of the mean to yield an estimate of the Studentized range. Tables for the allowable values for percentage points of the Studentized range are given in Bennett and Franklin (1954, p. 188-189), and elsewhere, for samples of size n, each with  $\nu$  degrees of freedom, where these degrees of freedom are the sums of the degrees of freedom for the several laboratory variancs included in the homogeneous variance.

The range for the 9 cobalt means is 0.14, which yields, when divided by the standard deviation of the mean,  $1.11 \times 10^{-3}$ , an estimate of 126 for the Studentized range. This is far larger than the allowable values of 4.68 for the 5-percent point or 5.58 for the 1-percent point of the Studentized range for the set of 9 means with 35 degrees of freedom. The average, 0.39, is the extreme mean of the group, and it is omitted from further consideration. The range, 0.08, of the remaining eight means is also highly significant for values for 8 means and 35

degrees of freedom, and the extreme mean, 0.2527, is then dropped. After further iteration, we determined that the values in the order 0.3128, 0.3127, and 0.3080 form a homogeneous group, and their average, 0.311 percent cobalt, is taken as the best estimate. The average of the original 9 means is 0.319, a value not too far from the best estimate. The agreement between the two estimates is probably due to the almost symmetrical differences between the best estimate and the two extreme means, an event that happens infrequently for the groups of means of the remaining elements for the two samples. Best estimates for the remaining elements were calculated similarly.

#### SELECTION PROCESS AND LACK OF CLEAR CHOICES

During the selection process, we frequently arrived at some step where we had to make a decision, but criteria for these decisions may be absent or perhaps conflicting because of their subjectivity. Tables 3 and 4 contain a number of best estimates listed as means without upper and lower confidence limits, and some discussion of difficulties encountered in the selections seems warranted. The laboratory averages for determinations for Nod-A-1 that are discussed for several elements below may be found in table 32 and those for Nod-P-1 in table 33.

Silicon.—The highest laboratory average for Nod-A was obtained by atomic-absorption spectroscopy, and the two lowest averages by X-ray fluorescence. After the two extreme means were eliminated, averages of 1.82, 1.73, and 1.63 remained. If the middle value is paired with either extreme of these three, the Studentized range is not significant at p=0.01 for either pair. However, because the lowest average, 1.63, was obtained by X-ray fluorescence and the two higher means by gravimetry, the average of 1.82 and 1.73 was accepted as the best value.

The highest average for silicon in Nod-P, 6.97, was obtained by atomic-absorption spectroscopy, but this is the extreme mean that was eliminated first. The range for the four remaining means, 6.65, 6.64, 6.38, and 6.36, is unacceptably large as is the range when the two extreme averages are discarded. The two higher and the two lower means, when paired, do not differ within themselves. We cannot resort to the analytical method as a criterion for our choice because both gravimetry and X-ray fluorescence were the methods for each pair. The average of the four means is therefore accepted as a provisional estimate without confidence limits. Aluminum.—Of the six available averages for the aluminum content of Nod-A, only two means, 2.06 and 2.04, survived the selection process, and the average of 2.05 percent aluminum is accepted as the best value. The Studentized range for three means for Nod-P, 2.59, 2.56, and 2.41, did not exceed the tabled value for p=0.01, and their average is accepted as the best value. The acceptance of a wider range for means for Nod-P is due to the larger standard error of the mean (0.05 as against 0.03) and to the higher acceptable values for the range for three range for the ra

Magnesium.—All differences between adjacent pairs of means for magnesium in Nod-A proved significant at p=0.01 because of the small standard error of 0.01, and the average of the four means, 2.93, 2.89, 2.85, and 2.80, was accepted as a provisional estimate without limits. We had no problem in the selection for Nod-P.

Sodium.—The two lower of the three means for Nod-A were accepted, but there was no choice for the three means for Nod-P, and their average is taken without limits.

Titanium.—The small standard error, 0.004, for titanium in Nod-A results in the choice of two identical means, 0.32, and in the rejection of 0.28 and 0.29. The still smaller standard error, 0.002, for Nod-P leaves no choice, and the average of the four means is taken as a provisional estimate.

*Cobalt.*—The small standard error, 0.0011, for the means for cobalt in Nod-A resulted in the rejection of all but three of the nine means available. The rounded average of the three means, 0.311 percent cobalt, is accepted as the best value. The selection process, again with a small standard error of 0.0011, resulted in two pairs of means for Nod-P, 0.2287 and 0.2280, and 0.2203 and 0.2200, that differed between but not within themselves. As there is no criterion for a choice, the average of the four means is listed as a provisional estimate.

Nickel.—The selection of a best estimate for nickel in Nod-A presented no difficulties, and the average of 0.636 percent nickel, calculated from laboratory means, 0.6400, 0.6372, and 0.6300, whose Studentized range was acceptable, is taken as the best value. However, the selection for Nod-P produced surprises. The two lowest means, 1.243 and 1.276, were rejected first because they were far removed from the center of gravity of the distribution of the eight means. Further tests showed that the Studentized range for the three largest means, 1.40, 1.395 and 1.382 (average, 1.392) is not significant at p=0.01 but that the range for the next three lower means, 1.350, 1.328, and 1.32 (average, 1.333), is significant.

Without the benefit of the several tests with the Studentized range, one might normally accept the grand average, 1.337, of the eight means as the best estimate of central tendency. As the average, 1.392, of the three means deemed acceptable by the test is far removed from the grand average of the eight means, this grand average is accepted as the conditional best estimate without confidence limits. Future data from other analysts will undoubtedly resolve this dilemma.

#### SUMMARY ESTIMATES AND DISCUSSION

The best estimates obtained by the Studentized range and the provisional estimates obtained when the dispersion among the means was too large are given in tables 3 and 4. Because the elements to be determined were not specified, the number of means for either type of estimate is variable. The number of variances used for the standard error of the mean may be obtained by dividing the degrees of freedom shown in tables 3 and 4 by the number of degrees of freedom (five) for each laboratory variance. The number of laboratory means that were used for best estimates will differ because of the selection process; the numbers of means that survived this process are given in tables 3 and 4. The numbers of averages from which the provisional estimates without confidence limits were calculated are given in parentheses.

Laboratory averages for all elements in both samples are listed in tables 32 and 33 to facilitate comparisons of the estimates by different analysts. The column headings indicate the table number from which the averages were taken. The number of determinations included in these averages is noted in each column heading as "n=x," where x is the number. This provision is necessary because not all data reported by two analysts were used in the analysis of variance for reasons of symmetry, and two other analysts had insufficient time to complete the requested work. Nonsignificant zeros added where percentage of an element or oxide reported by analysts was converted to parts per million are indicated by the lowercase letter "o."

In either table the spread of the data for the major or minor elements produced no surprises, except perhaps for one or two elements. The hypothesis associated with the analysis of variance—that the content of an element differed among bottles of the sample—was not accepted for the determinations of cobalt, copper, and nickel by any analyst whose data were amenable to the analysis of variance; eight analysts or laboratories have therefore effectively inferred that these elements are homogeneously distributed among the bottles of samples.

 TABLE 3.—Summary of best estimates and confidence limits for averages of several elements in USGS-Nod-A-1

[Estimates in percent or in parts per million, as indicated. Estimates in brackets are averages, without limits, of the number of laboratory means shown in parentheses. Student's t<sub>0.45</sub> was used for confidence limits]

	Standard error	Degrees of	· · · · · · · · · · · · · · · · · · ·	Best va	alues and confidence	limits	Number of laboratory
Element	of mean	freedom	Lower limit	<	Mean	< Upper limit	for best values
			Perce	ent			
Si	0.022	20	1.737		1.775	1.813	2
Al	.032	25	1.995		2.05	2.105	2
Fe	.042	30	10.861		10.932	11.003	6
Mg	.0099	25			[2.87]		(4)
Ca	.039	30	10.964		11.03	11.096	4
Na	.0056	15	.765		.775	.785	2
К	.0086	30	.485		.50	.515	4
Ti	.004	20	.315		.32	.325	2
P	.007	15	.588		.60	.612	2
Mn	.050	25	18.459		18.545	18.631	4
Co	.0011	35	.309		.311	.313	3
Cu	.0010	35	.1082		.1099	.1116	7
Ni	.0024	35	.632		.636	.640	3
			Parts per	million			
Ba	30.8	15	1.616		1,670	1,724	3
Mo	8.7	30	433		448	463	3
Pb	8.2	25	831		846	861	2
Sr	13.7	20	1.724		1.748	1,772	3
V	6.2	10	,		[770]		(3)
Zn	4.6	30	579		587	595	4

TABLE 4.—Summary of best estimates and confidence limits for averages of several elements in USGS-Nod-P-1 [Estimates in percent or in parts per million, as indicated. Estimates in brackets are averages, without limits, of the number of laboratory means shown in parentheses. Student's to 05 was used for confidence limits]

		Standard error	Degrees of		Best va	alues and confidence	limits		Number of laboratory
E	ement	of mean	freedom	Lower limit	<	Mean	<	Upper limit	for best values
				Perce	ent				
Si		0.016	15			[6.508]			(4)
Al		.049	25	2.466		2.55		2.634	3
Fe		.031	30	5.727		5.78		5.833	4
Mg_		.0085	20	1,975		1.990		2.005	4
Ca		.012	30	2.167		2.187		2.207	6
Na _		.0043	10			1.64			(3)
К		.012	35	1.030		1.05		1.070	3
Ti		.002	10			۲.30]			(4)
P		.002	15	.199		<b>.</b> 203		.207	3
Mn _		.080	$\overline{20}$	29.002		29.14		29.278	3
Co		.0011	35			[.224]			(4)
Cu		.0049	30	1.143		1.151		1.159	`4
Ni		.0064	30			[1.337]			(8)
		······································		Parts per	million				
Ba		27.7	10			[3,350]			(3)
Mo		4.1	20			¯ [762]			(5)
Pb		5.8	25			<b>[</b> 555]			(6)
Sr		3.3	10			Γ <del>6</del> 821			(3)
V		10.3	15			15671			(3)
Zn		5.9	$\overline{25}$	1,585		1,595		1,605	2

In view of these inferences, one might have expected the laboratory averages for these elements of potential economic interest to be more closely grouped than the data in tables 32 and 33 indicate.

Data for several trace elements are sparsely distributed in tables 32 and 33. An average by a single laboratory is reported for 19 of the 32 trace elements listed, and 13 of these 19 were included in the suite of elements determined by instrumental neutron-activation analysis.

The trace elements molybdenum, vanadium, and zinc may be of economic interest, depending on the commercial process used for treatment. Although most laboratories that determined these elements by completing the requested work found the elements to be homogeneously distributed in the samples, the dispersion among laboratory means is too large. Further analytical work will be necessary to obtain better estimates for these elements, especially if the samples are to be used as reference materials for future determinations that may form the basis for an economic decision.

Conspicuous by their absence in tables 32 and 33 are estimates for gold and the platinum metals. Harriss and others (1968, table 2) have determined palladium, iridium, and gold in nodules taken from near the sites for Nod-A-1 and Nod-P-1.

After this paper had been completed, P. J. Arus-

cavage (written commun., 1979) determined platinum, palladium, and ruthenium in the two nodule samples. The three precious metals were determined by a method involving fire assay and flameless atomic-absorption spectroscopy with the same experimental design used while obtaining other data for these samples. His averages of six determinations, by samples and by elements, are given as follows:

#### Platinum-metal contents [In parts per billion]

Sample	Pt	Pd	Ru
USGS-Nod-A-1	453	2.5	18 4.7
USGS-Nod-P-1	123	5.6	

The possible recovery of the platinum metals and gold as byproducts of a commercial extraction process designed for an anticipated production of 1-3million tons of nodules per year (Pearson, 1975) might lower the unit cost of the mining and extraction operations and thereby make more attractive the economic decision to proceed.

Chemical determinations of several constituents in both nodule samples were furnished by Sarah T. Neil of the U.S. Geological Survey, Menlo Park, Calif., after the manuscript had been completed. We corrected these determinations, which were on sample portions not dried, to a dry basis  $(105^{\circ} \text{ C})$  using the determinations of  $H_2O^-$  for each portion of sample. Gravimetric factors were used to convert data reported as oxides to elements.

The data by Neil are shown in tables 30 and 31, and her averages are listed with all other averages in tables 32 and 33. The analysis of variance, under the usual assumptions for the design with a single variable of classification, results in F ratios that are not significant at  $F_{0.95}$  for 34 of the possible 35 tests; the remaining ratio is not significant at  $F_{0.975}$ . Although data for two elements (titanium and sodium) in Nod-A and for three (titanium, sodium, and calcium) in Nod-P are outside the range of data provided by other analysts, we feel that these additional data would not significantly affect calculated best values. These data, plus others published later, will be used in calculations to revise or establish best values.

Anne E. Childress of the U.S. Geological Survey determined niobium in the two samples with the same experimental design used by the other analysts. Analyses of variance of the data showed that niobium was distributed homogeneously in the bottles of both samples. Childress obtained averages of 43.2 parts per million niobium for six determinatins for Nod-A-1 and 21.2 parts per million for Nod-P-1. These estimates were confirmed by Esma Y. Campbell, who found averages for two determinations of 43.8 parts per million niobium for Nod-A-1 and of 21.4 parts per million for Nod-P-1.

The contents of some lithophilic elements such as the rare earths, zirconium, hafnium, niobium, thorium, and uranium, shown in tables 32 and 33, are similar to the contents of these elements in fractionated crustal rocks. The relatively high abundances of these elements in the nodules, which contain high amounts of chalcophilic elements, pose an interesting problem for future geochemical research because of the low abundance of lithophilic elements in sea water and in basaltic rocks typical of the ocean floor.

#### COMPARISONS WITH DATA FROM MARSDEN SQUARES 049 AND 116

Because of the differences among data published for elements determined in manganese nodules, we have shown estimates for our nodule samples and those of the distribution of data on samples from the Marsden Squares that include the collection areas for our samples. Monget and others (1976), in their compilation, have listed the published data by the number of the Marsden Square, the rectangles enclosed by each  $10^{\circ}$  of longitude and latitude.

Our estimates for Nod-A-1 are listed in table 5 with estimates of the distribution of data from Marsden Square 116, and our estimates for Nod-P-1, together with estimates for Marsden Square 049, are given in table 6. We feel that our data and those from the squares should not be formally compared, because of the extremely low sampling density in the two squares—one sample per 100,000  $km^2$  in square 049 and one sample per 20,000  $km^2$ in square 116. The USGS samples were probably taken from test sites with areas of less than 35  $km^2$ , whereas Marsden Square 049 covers about 1,550,000  $km^2$  and square 116 in the Atlantic Ocean, about 500,000  $km^2$  after the land area included in the square is subtracted.

The ranges shown in the data for the samples from each Marsden square are very wide. Although some dispersion may be partly due to analytical methods and error, the major part may be due to compositional differences among single nodules that were analyzed. In addition to the average for manganese, the averages for copper and nickel, the two elements of considerable economic interest in the two USGS samples, agree well with, or exceed, the averages of data for the respective squares, but our cobalt averages are one-quarter to one-third lower. The manganese contents of the USGS samples are both about 6 percent (absolute) higher than the averages of similar data for samples from the corresponding squares.

#### AVAILABILITY OF SAMPLES

Because of the limited amounts of the two samples, we must have restrictions on their distribution until commercial exploitation begins and more material is available for future reference samples. Requests are anticipated from those who wish to use the samples as standards and from those who, in addition, wish to confirm or improve the estimates given here. Information on the availability of the samples may be obtained from either author.

The powder density of the processed material is less than that of powdered igneous rocks, and a full bottle is estimated to contain not more than 30 grams. The units of issue will be packaged routinely from the randomized stock of the samples, and some bottles may be only half filled as a result of our normal sampling procedure. Neither the authors nor this laboratory has hand specimens of the nodules from which the two samples were made.

#### ESTIMATES FOR MARSDEN-SQUARE AND USGS SAMPLES

 TABLE 5.—Comparison of best estimates for several elements in USGS-Nod-A-1 with estimates of the distribution of the elements for data in samples from Marsden Square 116

[Data for Marsden Square from Monget and others (1976). Estimates in percent or in parts per million, as indicated. n, number of determinations;  $\overline{x}$ , average; s.d., standard deviation]

			Esti	mates of data from Mars	sden Square 116		
Element	USGS - Nod-A-1		_	ad	R	ange	
	100-11-1	n	x	s.u.	Lower	< Upper	
			Percent				
Si	1.78	20	1.76	1.48	0.3	5.9	
Al	2.05	20	1.6	.88	.1	2.8	
Fe	10.93	<b>24</b>	11.38	4.4	1.6	20.0	
Mg	2.87	16	1.97	.67	1.09	3.70	
Ca	11.03	23	14.30	7,12	1.9	28.7	
Na	.77						
К	.50	17	.30	.10	.14	.54	
Ti	.32	21	.27	.15	.06	.68	
Р	.60	17	2.08	2.35	.05	7.0	
Mn	18.54	23	12.64	3.75	6.9	21.5	
Co	.311	9	.44	.05	.38	.55	
Cu	.110	23	.116	.062	.03	.26	
Ni	.636	24	.412	.156	.13	.77	
			Parts per mi	llion			
Ba	1,670						
Мо	448	6	1,107	1,570	350	4,300	
Pb	<b>846</b>	8	1,658	295	1,200	2,100	
Sr	1,748	20	1,660	684	676	3,380	
V	770						
Zn	587	8	508	94	390	670	

 TABLE 6.—Comparison of best estimates for several elements in USGS-Nod-P-1 with estimates of the distribution of the elements for data in samples from Marsden Square 049

[Data for Marsden Square from Monget and others (1976). Estimates in percent or in parts per million, as indicated. n, number of determinations;  $\overline{x}$ , average; s.d., standard deviation]

			Esti	mates of data from Mars	den Square 049		
Element	USGS - Nod-P-1				]	Range	
	1004-1-1	n	x	s.a. —	Lower	< Upper	
			Percent				
Si	6.51	12	7.62	1.59	5.4	11.2	
Al	2.55	10	3.43	.57	2.8	4.6	
Fe	5.78	18	7.69	2.24	2.4	10.4	
Mg	1.99	4	1.90	.11	1.7	2.0	
Ca	2.19	$1\overline{4}$	1.4	.24	.9	1.8	
Na	1.64						
К	1.05	9	.93	.22	.69	1.40	
Ti	.30	11	.46	.15	.29	.74	
P	.20	$\frac{1}{2}$	.12	(average)			
Mn	29.14	18	23.8	4.10	18.5	32.9	
Co	.224	17	.307	.091	.10	.44	
Cu	1.15	17	1.00	.22	.47	1.4	
Ni	1.33	18	1.18	.32	.47	1.9	
			Parts per mil	lion			
Ba	2.350						
Мо	762	-9	520	152	380	900	
Рb	555	9	911	482	280	1,700	
Sr	682	8	795	182	510	990	
V	567	3	587	121	460	700	
Zn	1,505	8	856	337	400	1,400	

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TABLES 7-33

#### 14 USGS ROCK STANDARDS: MANGANESE-NODULE SAMPLES USGS-NOD-A-1, USGS-NOD-P-1

 TABLE 7.—Determinations of several elements in USGS-Nod-A-1 by instrumental neutron-activation analysis by P. A.

 Baedecker, U.S. Geological Survey<sup>1</sup>

[Determinations in parts per million or in percent, as indicated. d.f., degrees of freedom; S, significant; NS, not significant; Neg., negative bottle variance obtained, F ratios tested at  $F_{0.95}$  or the fractile in parentheses. Analytical method: Baedecker, Rowe and Steinnes (1977)]

		Bottle			Standard	d deviation	
Element	30/25	52/1	53/18	Mean	Bottle $(d.f.=2)$	Error (d.f.=3)	F ratio
			Р	ercent			
Fe	$\begin{array}{c} 10.75\\ 10.98 \end{array}$	10.86 10.89	$\begin{array}{c} 11.05\\ 10.77\end{array}$	10.88	Neg.	0.15	0.05 NS
			Parts	per million			
Ba	1,458 1,738	$1,451 \\ 1,551$	1,457 1,454	1,518	Neg.	120	.72 NS
Се	676 670	672 673	667 651	668	6.2	7.0	2.58 NS
Co	2,522 2,530	$2,506 \\ 2,544$	$2,550 \\ 2,512$	2,527	Neg.	22.2	.04 NS
Cr	$\begin{array}{c} 26.4 \\ 27.3 \end{array}$	$\begin{array}{c} 30.7 \\ < 67.7 \end{array}$	$19.1 \\ < 65.1$				
Eu	$\begin{array}{r} 4.49 \\ 4.45 \end{array}$	$\begin{array}{c} 4.54 \\ 4.52 \end{array}$	$\begin{array}{r} 4.48 \\ 5.39 \end{array}$	4.48	.04	.04	2.74 NS
Gd	$\begin{array}{c} 28.1 \\ 25.1 \end{array}$	33.2 32.6	$\begin{array}{c} 20.8\\ 19.4 \end{array}$	26.5	6.3	1.4	43.4 S(0.99)
Hf	6.4 6.4	6.6 5.4	6.9 5.5	6.2	Neg.	.75	.14 NS
La	134 129	133 131	139 129	132.5	Neg.	4.6	.16 NS
Lu	2.10 2.12	2.10	2.25	2.16	.03	.06	1.42 NS
Nd	86 83	82 85	93 83	85.3	Neg.	4.4	.57 NS
Sb	33.0 34.6	$33.6 \\ 32.5$	33.0 34.3	33.5	Neg.	.95	.35 NS
Sc	11.09 11.36	$11.10 \\ 11.23$	$11.32 \\ 11.27$	11.23	Neg.	.12	.55 NS
Sm	18.5 21.6	$\begin{array}{c} 21.4 \\ 21.8 \end{array}$	20.8 21 4	20.9	Neg.	1.3	.74 NS
Tb	$4.54 \\ 6.00$	4.36 4.81	5.76 $3.74$	4.87	Neg.	1.03	.24 NS
Th	$\begin{array}{c} 25.4\\ 25.7\end{array}$	$\begin{array}{c} 25.6\\ 25.3 \end{array}$	$26.4 \\ 26.6$	25.8	.56	.19	18.3 NS(0.99)
Tm	$.78 \\ 1.52$	$\begin{array}{c} 1.62 \\ < 1.88 \end{array}$	2.29 2.30	1.72	² .77	³ .37	9.57 NS
U	$5.8\\5.5$	$\begin{bmatrix} 6.7\\ 5.5\end{bmatrix}$	$\begin{array}{c} 6.5 \\ 5.7 \end{array}$	5.95	Neg.	.60	.37 NS
Yb	$\begin{array}{c} 16.0\\ 17.1 \end{array}$	15.9 15.9	$\begin{array}{c} 15.6 \\ 17.1 \end{array}$	16.3	Neg.	.76	.38 NS

<sup>1</sup> Reston, VA 22092. <sup>2</sup> d.f.=1. <sup>3</sup> d.f.=2.

 $[ Determinations in parts per million or in percent, as indicated. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratios tested at <math>F_{0.95}$ . Analytical method: Baedecker, Rowe, and Steinnes (1977)]

III III		Bottle		_	Standar	d deviation	
Element	7/1	17/19	48/11	Mean	Bottle (d.f.=2)	$\frac{\text{Error}}{(d,f,=3)}$	F. ratio
			Р	ercent			
Fe	$5.80 \\ 5.76$	$5.94 \\ 5.15$	6.03 5.85	5.76	Neg.	0.33	0.72 NS
			Parts	per million			
За	2,512	2,617	2,519	2,498	Neg.	<sup>2</sup> 290	.95 NS
Ce	2,700	2,009	287	2,458	6.5	9.9	1.85 NS
Co	1,789 1,802	1,813 1,775	1,806 1,865	1,808	11.4	29.1	1.30 NS
)r	21.0	<57.5	<56.0				
2u	6.16 6.18	< 57.5 6.67	6.59 7.06	6.57	.32	.19	6.44 NS
Gd	$26.4 \\ 25.9$	24.4 33.6	36.1 30.1	29.4	1.5	4.5	1.21 NS
If	4.3	4.2	4.2	4.3	Neg.	.10	.50 NS
⊿a	126	4.4 130 102	127	120	Neg.	<sup>2</sup> 13.7	.26 NS
u	1.89	1.95	1.86	1.85	Neg.	<sup>2</sup> .16	.55 NS
1d	106 104	113	111 126	112.8	5.4	6.4	2.40 NS
Sb	49.5	51.5	50.1 52.9	50.1	Neg.	2.4	.58 NS
Sc	9.54 $9.53$	40.4 9.65 8.67	9.61 9.84	9.47	Neg.	.41	.98 NS
Sm	28.3	29.6	31.1	30.4	.64	1.2	1.61 NS
ГЬ	30.9 5.18	30.3 5.71	32.0 5.23	5.31	Neg.	.35	.34 NS
ſh	5.65 17.3	5.02 16.3	5.06 16.8	17.0	Neg.	.61	.35 NS
Гт	16.5 1.75	17.2 1.70	1.54	1.77	Neg.	.17	.19 NS
J	1.91 $2.7$	$1.82 \\ 3.4 \\ 4.2$	1.91	3.5	.30	.53	1.64 NS
Yb	$\begin{array}{c} 3.2\\ 14.2\\ 14.2\end{array}$	4.3 14.8 11 9	4.1 15.0	13.8	Neg.	<sup>2</sup> 1.4	.34 NS

<sup>1</sup> Reston, VA 22092. <sup>2</sup> d.f.=2.

Flowenter		Bottle				Standard	deviation	-
oxide	1	2	3	- Mean	Mean <sup>2</sup>	Bottle (d.f.=2)	Error (d.f.=3)	ratio
SiO <sub>2</sub>	3.98 3.83	3.92 3.91	3.90 3.85	3.90	1.82	Neg.	0.06	0.21 NS
Al <sub>2</sub> O <sub>3</sub>	$3.32 \\ 3.35$	$2.99 \\ 3.40$	$3.43 \\ 3.33$	3.30	1.75	Neg.	.17	.62 NS
$Fe_2O_3(T)$	$15.57 \\ 15.49$	$15.40 \\ 15.49$	$15.49 \\ 15.74$	15.53	10.86	.03	.11	1.12 NS
Mg	2.80	2.88 2.85	2.85	2.85	2.85	.01	.03	1.49 NS
Ca	$11.10 \\ 11.25$	11.38 11.20	11.10 10.98	11.17	11.17	.10	.11	2.71 NS
Na	.79	.82	.79	.80	.80	.006	.011	1.50 NS
К	.50	.51	.58	.50	.50	.02	.04	1.27 NS
TiO <sub>2</sub>	.54 .54	.53 .53	.53 .54	.53 5	.32	.004	.004	3.00 NS
<b>P</b> <sup>5</sup> O <sup>2</sup>	.96 .93	.99 1.06	.76 1.12	.97	.42	Neg.	.15	.20 NS
Mn	$18.35 \\ 18.31$	$18.36 \\ 18.41$	$18.49 \\ 18.50$	18.40	18.40	.08	.02	20.2 NS(0.99)
Co	.328	.334 .326	.323	.326	.326	.002	.003	2.01 NS
Cu	.112	.112	.112	.112	.112	.0006	.0011	1.50 NS
Ni	.649 .639	.653 .644	.650 .654	.648	.648	Neg.	.0057	.98 NS

 TABLE 9.—Determinations of several elements and oxides in USGS-Nod-A-1 by John Marinenko, U.S. Geological Survey<sup>1</sup>

 [Determinations in percent. T, total; d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratios tested at  $F_{0.05}$  or the fractile indicated in parentheses]

<sup>1</sup> Reston, VA 22092. <sup>2</sup> As percent element.

Analytical methods:-SiO<sub>2</sub>, gravimetry; Al<sub>2</sub>O<sub>3</sub>, fluorimetric analysis; Total Fe and Mn, volumetric analysis; Mg, Ca, Na, K, Co, Cu, and Ni, atomicabsorption spectrometry; and TiO<sub>2</sub> and P<sub>2</sub>O<sub>5</sub>, spectrophotometry.

Element el	Bottle					Stand	ard deviation	
Element or oxide	1	2	3	Mean	Mean <sup>2</sup>	Bottle (d.f.=1)	Error (d.f.=2)	- F ratio
SiO <sub>2</sub>	14.48	14.45 13.82 14.46	14.17 13.90	14.08	6.64	Neg.	0.34	0.08 NS
Al <sub>2</sub> O <sub>3</sub>	3.90	3.30 3.94 4.02	$\begin{array}{c} 3.44\\ 3.78\end{array}$	3.62	1.97	Neg.	.36	.0008 NS
Fe <sub>2</sub> O <sub>3</sub> (T)	8.23	$8.36 \\ 8.14 \\ 8.10$	$\substack{\textbf{8.15}\\\textbf{8.15}}$	8.20	5.73	Neg.	.11	.83 NS
Mg	2.00	$1.98 \\ 1.99 \\ 1.95$	$\begin{array}{c} 1.96 \\ 2.02 \end{array}$	1.99	1.98	Neg.	.03	.03 NS
Ca	2.20	2.25 2.21 2.21	$\begin{array}{c} 2.22 \\ 2.14 \end{array}$	2.20	2.20	.02	.04	1.25 NS
Na	1.62	$1.82 \\ 1.64 \\ 1.63$	$\begin{array}{c} 1.74\\ 1.61 \end{array}$	1.70	1.68	Neg.	.11	.24 NS
К	.96	1.04 .97 .98	$\begin{array}{c} 1.03\\ .98 \end{array}$	1.00	.99	Neg.	.04	0.0 NS
TiO <sub>2</sub>	.51	.52 .50 .52	.52 .50	.51	.31	Neg.	.01	0.0 NS
P <sub>2</sub> O <sub>5</sub>	.41	.43 .42 .22	.41 .43	.42	.17	Neg.	.011	.20 NS
Mn	29.49	28.66 29.42 29.42	$\begin{array}{c} 29.10\\ 29.04 \end{array}$	29.06	29.19	Neg.	.38	.006 NS
Co	.219	.226 .217 .218	.223 .219	.221	.220	Neg.	.005	.01 NS
Cu	1.20	1.19 1.20 1.21	1.18 1.19	1.19	1.20	.005	.007	2.00 NS
Ni	1.37	$     1.41 \\     1.39 \\     1.37 $	$\begin{array}{c} 1.41 \\ 1.42 \end{array}$	1.41	1.40	.007	.011	1.80 NS

 TABLE 10.—Determinations of several elements and oxides in USGS-Nod-P-1 by John Marinenko, U.S. Geological Survey<sup>1</sup>

 [Determinations in percent. T, total; d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratios tested at F0.05. Estimates of the mean, standard deviation, and F ratio calcuated for the symmetrical data for bottles 2 and 3]

<sup>1</sup> Reston, VA 22092.

 $^{2}\ \mathrm{Average}$  of six determinations, as the element, used for best values.

Analytical methods:-SiO<sub>2</sub>, gravimetry; Al<sub>2</sub>O<sub>3</sub>, fluorimetric analysis; Total Fe and Mn, volumetric analysis; Mg, Ca, Na, K, Co, Cu, and Ni, atomicabsorption spectrometry; andTiO<sub>2</sub> and P<sub>2</sub>O<sub>5</sub>, spectrophotometry.

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 TABLE 11.—Determinations of U and Th in USGS-Nod-A-1 and USGS-Nod-P-1 by delayed neutron activation analysis by

 H. T. Millard, Jr., and C. M. Ellis, U.S. Geological Survey

[Determinations in parts per million. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratio tested at F0.85. Analytical method described by H. T. Millard, Jr., 1976]

					Standard	deviation	E
Element		Bottle		Mean	Bottle (d.f.=2)	Error (d.f.=3)	ratio
		· · · · · · · · · · · · · · · · · · ·	USGS-	Nod-A-1			
	17/13	18/01	24/32				
Th	$24.84 \\ 25.37$	27.30 35.93	$\frac{25.79}{24.88}$	27.35	2.71	3.55	2.17 NS
U	$\begin{array}{c} 6.84 \\ 6.91 \end{array}$	$\begin{array}{c} 6.84 \\ 6.97 \end{array}$	$\begin{array}{c} 6.85\\ 6.74\end{array}$	6.86	.02	.08	1.14 NS
			USGS-	Nod-P-1			
	13/12	22/29	55/04				
Th	$17.80 \\ 16.47$	15.65 19.80	19.11	17.65	Neg.	1.96	.12 NS
U	$\begin{array}{c} 4.18\\ 4.36\end{array}$	$\begin{array}{c} 4.36\\ 4.31\end{array}$	$4.09 \\ 4.37$	4.28	Neg.	.14	.30 NS

<sup>1</sup>Stop 424, Box 25046 DFC, Lakewood, CO 80225.

TABLE 12.—Determinations of several elements in USGS-Nod-A-1 by the National Physical Research Laboratory <sup>1</sup> [Determinations in percent. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratio tested at F0.85]

		Bottle			Standar	d deviation	F
Element	22/1	51/12	63/24	Mean	Bottle (d.f.=2)	Error (d.f.=3)	ratio
A1	1.8 1.3	2.0 $1.5$	1.9 1.5	1.66	Neg.	0.33	0.20 NS
Fe	$\begin{array}{c} 10.0\\9.1 \end{array}$	$\begin{array}{c} 10.3 \\ 8.9 \end{array}$	$\begin{array}{c}10.3\\9.6\end{array}$	9.7	Neg.	.74	.17 NS
К	.13 .18	.18 .18	.19 .14	.17	Neg.	.03	.38 NS
Mn	$13.3 \\ 13.4$	$14.6\\12.9$	14.0 $14.2$	13.7	Neg.	.70	.57 NS
Ba	.32 .21	.36 .22	.23 .29	.27	Neg.	.08	.09 NS
Co	.30 .31	.32 .30	.32 .30	.31	Neg.	.01	.11 NS
Cu	.11 $.12$	.11	.11	.11	.000	.004	1.0 NS
Mo	.034 .039	.038 .034	.039 .034	.036	Neg.	.003	.02 NS
Ni	$.64\\.64$	$\begin{array}{c} .64\\ .63\end{array}$	$.64 \\ .65$	.64	.007	.004	7.0 NS
Pb	.10 .091	.094 .094	.094 .095	.095	Neg.	.004	.08 NS
Sr	.19 .14	.20 .16	.21 .18	.18	Neg.	.03	.54 NS
Zn	$.057 \\ .058$	.061 .059	.059 .059	.059	Neg.	.0009	3.8 NS

<sup>1</sup> CSIR, P.O. Box 395, Pretoria 0001, South Africa.

Analytical method:—A mixture of 20 mL HF and 10 mL concentrated HCl was added to 1-g portions of the samples and the mixtures taken to dryness on a hotplate. This procedure was repeated. Twenty mL of 50 percent HCl was added to the residue and evaporated to a volume of 5 mL. These were transferred to a 25-mL volumetric flask and made to volume with distilled water. Aliquots of these solutions were diluted to suitable concentrations for the determinations of the elements. These determinations were made using a Techtron AA5<sup>1</sup> atomic-absorption instrument with the recommended analytical conditions.

<sup>1</sup> Any trade names in this publication are used for descriptive purposes only and do not constitute endorsement by the U.S. Geological Survey.

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Bottle Standard deviation Element Bottle (d.f.=2) Mean Error (d.f.=3) ratio 37/2 44/538/28  $1.5 \\ 2.0$ 1.9 1.9 0.21 Al \_\_\_\_\_ 1.80.27 NS Neg. 1.8 1.95**.2** Fe \_\_\_\_\_ 5.25.25.4Neg. .32.64 NS 5.75.24.8.29 .23 .29 .28 К \_\_\_\_\_ .26 Neg. .04 .52 NS .28 .3525.925.426.3Mn \_\_\_\_\_ 25.5Neg. .94 .12 NS 24.2 .70 25.2 26.0Ba ..... .67 .58 .67 .04 .03 4.98 NS .73 .69 .65 Co ..... .22 .21 .24 .22 .004 .009 1.40 NS .22 .22 .22 Cu \_\_\_\_\_ 1.21.21.31.2.000 .04 1.00 NS 1.2 1.2 1.2.068 .063 .063 .004 4.50 NS Mo ..... .063 .003 .058 .068 .058 Ni \_\_\_\_\_ .000. 1.4 1.4 .04 1.00 NS 1.4 1.41.5 1.4 1.4 Pb \_\_\_\_\_ .054 .052 .051 .052 Neg . .002 .71 NS .053 .053 .051 .11 .11 .17 .000 Sr \_\_\_\_\_ .099 .004 1.00 NS **.1**1 .108 **.1**1 .11 Zn \_\_\_\_\_ .17.15.16 .000 .01 (No test) .15.15 .17

TABLE 13.—Determinations of several elements in USGS-Nod-P-1 by the National Physical Research laboratory <sup>1</sup> [Determinations in percent. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratio tested at  $F_{0.95}$ ]

<sup>1</sup> CSIR, P.O. Box 395, Pretoris 0001, South Africa.

Analytical method:—A mixture of 20 mL HF and 10 mL concentrated HCl was added to 1-g portions of the samples and the mixtures taken to dryness on a hotplate. This procedure was repeated. Twenty mL of 50 percent HCl was added to the residue and evaporated to a volume of 5 mL. These were transferred to a 25-mL volumetric flask and made to volume with distilled water. Aliquots of these solutions were diluted to suitable concentrations for the determinations of the elements. These determinations were made using a Techtron AA5 atomic-absorption instrument with the recommended analytical conditions.

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Flowert		Bottle			Standard	l deviation	F	
or oxide	1	2	3	Mean	Bottle $(d.f.=2)$	Error (d.f.=3)	ratio	
				Percent				
Si	1.45	1.20	1.35	1.42	Neg.	0.16	0.16 NS	
Al	$1.44 \\ 2.32 \\ 2.41$	$\begin{array}{c} 1.54\\ 2.18\\ 2.25\end{array}$	1.56 2.11 2.34	2.27	.04	.10	1.28 NS	
Fe	11.24 10.90	$10.78 \\ 10.99$	$10.86 \\ 11.12$	10.98	Neg.	.19	.45 NS	
Ca	11.40	11.35 11.32	11.32 11 41	11.42	.07	.13	1.64 NS	
Mg	3.27 3.36	3.11 3.14	3.18	3.20	.10	.05	9.12 NS	
К	.46 .51	.44 .47	.51 .50	.48	.02	.02	2.17 NS	
Ті	.29	.29 .30	.29	.29	Neg.	.006	.5 NS	
Р	.57	.58	.60	.59	Neg.	.02	.1 NS	
Mn	18.89 18.65	$18.71 \\ 18.65$	$18.56 \\ 18.39$	18.64	.12	.12	3.04 NS	
CO <sub>2</sub>	11.66 11.70	11.80 11.55	11.44 11.70	11.64	Neg.	.15	.35 NS	
Со	.44	.37	.41	.39	.02	.02	3.08 NS	
Cu	.18	.19	.18	.18	Neg.	.006	.5 <sub>.</sub> NS	
Ni	.60 .57	.57 .52	.57 .57	.57	.01	.02	1.44 NS	
			Pa	rts per million				
As	315 300	280 300	285 310	298	Neg.	14.4	.74 NS	
Ba	1,695	1,625 1,645	1,665 1.715	1,671	26	23	3.70 NS	
Мо	460 450	460 465	465 460	460	2.5	5.0	1.5 NS	
Pb	1,310	1,315 1.315	1,325	1,323	13	14	2.66 NS	
Rb	10 10	10 10	10 10	10			(No test)	
Sr	1,760	1,715	1,775	1,728	Neg.	41	.09 NS	
Υ	115	110	115	115	1.4	2.9	1.5 NS	
Zn	880 870	865 875	865	870	2.9	5.8	1.5 NS	
Zr	320 310	310 320	320 320	317	Neg.	5.8	.5 NS	

 TABLE 14.—Determinations of several elements in USGS-Nod-A-1 by S. E. Calvert, Institute of Oceanographic Sciences<sup>1</sup>

 [Determinations in percent or parts per million, as indicated. d.f., degrees of freedom; NS, not significant; Neg., negative variance obtained. F ratio tested at F<sub>0.95</sub>]

<sup>1</sup> Brook Rd., Wormley, Godalming, Surrey, GU8 5UB, England.

Analytical methods:—(1) Major elements Si to Mn, plus Co, Cu, and Ni, were determined by X-ray fluorescence on samples fused with LizB<sub>1</sub>O<sub>7</sub> and La<sub>2</sub>O<sub>3</sub>; the method follows that of Norrish and Hutton (1969); (2) trace elements As to Zr were determined by X-ray fluorescence on powders pressed into discs at 15 tons; the method follows that of Anderman and Kemp (1958); (3) CO<sub>2</sub> was determined gravimetrically after treating the sample with hot 10 percent HCl.

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		Bottle			Standard	deviation	
Element — or oxide	1	2	3	Mean	Bottle (d.f.=2)	Error (d.f.=3)	ratio
				Percent			
Si	6.67 6.54	6.62 6.62	6.74 6.69	6.65	0.04	0.06	2.20 NS
Al	2.51	2.51 2.78	2.58 2.65	2.59	Neg.	.11	.65 NS
Fe	4.80	4.63	4.83	4.76	.11	.05	11.0 NS(0.975)
Ca	2,18	4.02 2.15 2.17	2.20	2.16	Neg.	.04	.04 NS
Mg	2.12 2.00 2.12	2.01	2.12	2.08	Neg.	.11	.06 NS
К	$\begin{array}{c} \textbf{2.12}\\ \textbf{1.13}\\ \textbf{1.14} \end{array}$	2.19 1.15 1.18	$1.10 \\ 1.15$	1.14	.01	.02	1.48 NS
Ti	.30	.30	.30	.30			(No test)
Р	.30 .22 21	.21	.50 .21 .21	.21	Neg.	<b>.0</b> 06	.5 NS
Mn	29.66 29.75	29.15 28.50	29.65 28.50	29.06	Neg.	.61	.16 NS
CO <sub>2</sub>	1.21	1.91	1.14	1.39	.46	.06	<sup>2</sup> 102
Co	.30	.28	.28	.28	.008	.004	9.0 NS
Cu	1.02 1.07	1.11	1.12 05	1.04	Neg.	.10	.01 NS
Ni	1.07 1.26 1.38	1.40 1.11	1.34 1.17	1.28	Neg.	.14	.13 NS
•			Pa	rts per million			
As	40	50 50	25 35	39	9.6	4.6	9.8 NS(0.975)
Ba	3,405 2,455	3,500 2,425	3,460 2,475	3,453	Neg.	37	.60 NS
Мо	820	825 815	835	823	Neg.	7.6	.50 NS
Pb	825 700 740	700	690 795	715	Neg.	27	.14 NS
Rb	20 20 20	$\begin{array}{c} 740\\ 20\\ 25\end{array}$	20 20 20	21	.000	2.0	1.0 NS
Sr	755	765	760	751	Neg.	12 <b>.2</b>	.11 NS
Y	90	90 90	85	89	.000	2.0	1.0 NS
Zn	90 1,975	90 1,985	90 1,990	1,998	Neg.	20	.12 NS
Zr	2,010 280 280	2,010 285 275	2,015 280 280	280	Neg.	4.1	.0 NS

TABLE 15.—Determinations of several elements in USGS-Nod-P-1 by S. E. Calvert, Institute of Oceanographic Sciences<sup>1</sup> [Determinations in percent or parts per million, as indicated. d.f., degrees of freedom; NS, not significant; Neg., negative variance obtained. F ratio tested at  $F_{0.95}$  or the fractile indicated in parentheses]

 $^1$  Brook Rd., Wormley, Godalming, Surrey, GU8 5UB, England.  $^2\,F$  ratio is extremely significant.

Analytical methods:—(1) Major elements Si to Mn, plus Co, Cu, and Ni, were determined by X-ray fluorescence on samples fused with LizBtO7 and LazO2; the method follows that of Norrish and Hutton (1969); (2) trace elements As to Zr were determined by X-ray fluorescence on powders pressed into discs at 15 tons; the method follows that of Anderman and Kemp (1958); (3) CO<sub>2</sub> was determined gravimetrically after treating the sample with hot 10 percent HCl.

		Bottle			Standard o	leviation	
Element	5/29	18/15	28/11	Mean	Bottle (d.f.=2)	Error (d.f.=3)	ratio
				Percent			
Si	1.98	1.95 2.15	1.98	2.005	Neg.	0.09	0.6 NS
Al	2.04	2.11	2.09	2.060	Neg.	.04	.43 NS
Fe	11.20	10.80	11.00 11.60	11.08	.13	.26	1.49 NS
Mg	2.80	2.84	2.79	2.803	.01	.02	1.75 NS
Ca	11.00	11.00 10.90	11.00	10.97	Neg.	.06	.5 NS
Na	.76	.80 77	.75	.767	.01	.01	3.1 NS
К	.51 50	.52	.53	.518	.012	.004	19 NS(0.99)
Ti	.27 28	.27 29	.26	.275	Neg.	.012	.33 NS
Mn	$17.60 \\ 17.90$	$18.10 \\ 17.70$	$17.90 \\ 17.60$	17.80	Neg.	.24	.26 NS
,			Par	ts per million			
Ba	1,700	1,580 1,670	1,640 1.740	1,668	Neg.	55.5	0.91 NS
Be	5.5 5.5	5.8 5.8	5.3 5.5	5.57	.20	.08	13.0 NS(0.975)
Co	3,100 3,170	3,090 3,180	3,100 3,130	3,128	Neg.	48	.12 NS
Cr	25 20	25 25	20 25	23	Neg.	2.9	.5 NS
Cu	$1,050 \\ 1.050$	$1,060 \\ 1.050$	$1,040 \\ 1.060$	1,052	Neg.	9.1	.2 NS
Li	77 76	76 76	77 76	76.3	Neg.	.58	.5 NS
Mo	460	443	465	457.3	Neg.	13.1	.82 NS
Ni	440 6,300 6,400	468 6,300	468 6,200	6,300	29	58	.5 NS
Pb	798 805	0,300 778 890	0,300 778 778	792.8	3.9	17.4	1.10 NS
Sr	1,680	1,650	1,660	1,660	Neg.	28.9	.78 NS
v	578 500	580	590 575	586.3	Neg.	1 <b>2.</b> 9	.35 NS
Zn	561 548	565 565	553 560	558.7	3.6	6.0	1.71 NS

 TABLE 16.—Determinations of several elements in USGS-Nod-A-1 by R. T. T. Rantala, Bedford Institute of Oceanography<sup>1</sup>

 [Determinations in percent or in parts per million, as indicated. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained.

 F ratio tested at F<sub>0.95</sub> or the fractile indicated in parentheses]

<sup>1</sup> Dartmouth, N.S., Canada, B2Y 4A2.

Analytical method.—Samples were decomposed by acid in teflon bombs as described by Rantala and Loring (1973); the elements were determined by flame atomic-absorption spectrometry as described by Rantala and Loring (1975), with slight variations due to dilutions used.

 TABLE 17.—Determinations of several elements in USGS-Nod-P-1 by R. T. T. Rantala, Bedford Institute of Oceanography<sup>1</sup>

 [Determinations in percent or in parts per million, as indicated. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained.

 F ratio tested at  $F_{0.05}$  or the fractile indicated in parentheses]

<u> </u>		Bottle			Standar	d deviation	
Element	29/23	52/24	57/10	Mean	Bottle $(d.f.=2)$	Error (d.f.=3)	ratio
				Percent			
Si	6.85 6.88	6.90 6.95	6.85 7.38	6.968	Neg.	0.22	0.72 NS
Al	2.42 2.38	2.37 2.45	2.44 2.38	2.407	Neg.	.04	.03 NS
Fe	5.76 5.74	5.76 5.88	5.94 5.80	5.813	.03	.08	1.27 NS
Mg	1.96 1.98	1.97 1.99	1.97 1.98	1.975	Neg.	.01	.33 NS
Ca	2.16 2.16	2.16 2.12	2.18 2.16	2.157	.008	.018	1.4 NS
Na	1.63 1.64	1.62 1.65	1.62 1.62	1.630	Neg.	.013	.90 NS
К	1.04	1.05 1.04	1.04	1.040	.005	.010	1.5 NS
Ti	.28	.26	.28	.277	.010	.006	6.5 NS
Mn	28.90 28.90	29.20 29.40	29.30 29.30	29.17	.22	.08	16.0 NS(0.975)
			Pa	rts per million			
Ba	2,710	2,940	2,800	2,862	Neg.	103	0.38 NS
Be	2.8	2,000	2.8	2.8	.06	.11	1.5 NS
Co	2,240 2,260	2,270 2,340	2,290 2,280	2,280	18	30	1.72 NS
Cr	20 20	20 20	20 20	20			
Cu	11,300 11,300	11,400 11,300	11,500	11,370	64	58	3.50 NS
Li	$142 \\ 142$	143 140	141 143	141.8	Neg.	1.5	.08 NS
Mo	795	803	785	801.7	Neg.	12.9	.32 NS
Ni	800 13,000	812 13,200	815 13,400	13,280	Neg.	220	.24 NS
Рв	428 415	13,500 430	13,200 442	436.2	12	10	4.10 NS
Sr	415 618	442 628	460 665	638.2	10.4	15.8	1.86 NS
v	625 483	655 478	638 470	479.8	8.6	5.7	5.46 NS
Zn	$\begin{array}{r} 490 \\ 1,460 \\ 1.460 \end{array}$	$\begin{array}{r} 490 \\ 1,490 \\ 1.460 \end{array}$	$\begin{array}{r} 468 \\ 1,470 \\ 1.460 \end{array}$	1,467	Neg.	12.9	.70 NS

<sup>1</sup> Dartmouth, N.S., Canada, B2Y 4A2.

Analytical method:-Samples were decomposed by acid in tefion bombs as described by Rantala and Loring (1973); the elements were determined by flame atomic-absorption spectrometry, described by Rantala and Loring (1975), with slight variations due to dilutions used.

		Bottle			Standard	deviation		
Element	9/30	12/3	36/17	Mean	Bottle $(d.f.=2)$	Error (d.f.=3)	ratio	
			]	Percent				
Si	1.64	1.56	1.71	1.63	0.04	0.04	2.7 NS	
Al	1.85	1.87	1.86	1.86	.02	.08	1.13 NS	
Fe	10.37	10.25	10.32	10.29	.04	.03	3.39 NS	
Mg	10.31 2.77	2.73	2.72	2.74	.025	.009	16.2 NS(0.99)	
Ca	11.33	11.28	11.30	11.30	.008	.062	1.03 NS	
К	.35 .42	.38 .40	.40 .44	.40	Neg.	.03	.62 NS	
Ti	.32	.32	.32	.32	.008	.015	1.50 NS	
Mn		18.62 18.61	18.61	18.63	Neg.	.04	.91 NS	
Р	.64	.58	.62	.60	.02	.02	2.28 NS	
Co	.278	.285	.278	.281	.002	.002	1.77 NS	
Cu	.108	.107	.108	.107	Neg.	.0009	.20 NS	
Ni	.624 .616	.639 .621	.623 .625	.625	Neg.	.008	.78 NS	
			• Parts	per million				
Мо	561 561	535 540	562 625	567	19	30	1.80 NS	
Zn	496 578	549 579 569	619 603	574	28	34	2.32 NS	

 TABLE 18.—Determinations of several elements in USGS-Nod-A-1 by the National Institute for Metallurgy<sup>1</sup>

 [Determinations in percent, or in parts per million, as indicated. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratio tested at F<sub>0.95</sub> or the fractile indicated in parentheses. Si through P reported as the oxide but converted to the element]

<sup>1</sup> Private Bag X3015, Randburg, 2125, South Africa.

Analytical methods:-

1. MnO, 0.75 g of Nod-P and 1 g of Nod-A were dissolved in HCl and HNO3 and taken to fumes with H2SO4. After cooling, water was added and the solutions diluted in a volumetric flask. Mn was determined on an aliquot by potentiometric titration with KMnO4.

 SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, CaO, K<sub>2</sub>O, TiO<sub>2</sub>, MnO, and P<sub>2</sub>O<sub>5</sub> were determined by X-ray fluorescence on a disc made by fusing 0.5-0.6-g samples with 7.5 g of a flux of 10 percent Li<sub>2</sub>B<sub>1</sub>O<sub>7</sub>, 51 percent Na<sub>2</sub>B<sub>1</sub>O<sub>7</sub>, and 39 percent Na<sub>2</sub>CO<sub>3</sub>.

3. For Co, Cu, Mo, Ni, and Zn, 0.4-g samples were dissolved with 10 mL HNO3, 20 mL HF, and 3 mL HClO4 in platinum dishes. Solutions were fumed to incipient dryness and 3 mL HCl added. Solutions were warmed and 3-4 drops of H2O2 added to oxidize Mn. The resulting clear solutions were transferred to volumetric flasks and diluted to the mark. Appropriate dilutions of these solutions were measured by atomic-absorption spectrometry.

4. The Canadian sample, CSRM-SU-1, was used as a control sample; results are listed as follows:

	National Institute for Metallurgy	Certified Value	
Coppm	568, 553, 581, 569	630	
Cupercent	0.845, 0.845, 0.846, 0.838	0.87	
Moppm	<40, <40, <40, <40		
Nipercent	1.46, 1.46, 1.45, 1.46	1.51	
Znppm	319, 311, 304, 312	294	

 TABLE 19.—Determinations of several elements in USGS-Nod-P-1 by the National Institute for Metallurgy<sup>1</sup>

 [Determinations in percent, or in parts per million, as indicated. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratios tested at F0.05 or the fractile indicated in parentheses. Si through P reported as the oxide but converted to the element]

		Bottle			Standard	deviation	F
Element	18/6	24/30	58/11	- Mean	Bottle (d.f.=2)	Error (d.f.=3)	r ratio
			I	Percent			
Si	6.30	6.37	6.61	6.36	0.06	0.14	1.34 NS
Al		6.41 2.61 2.57	6.30 2.66 2.50	2.56	Neg.	.08	.83 NS
Fe	2.44 5.60 5.56	5.60 5.54	5.49 5.36	5.52	.075	.06	4.09 NS
Mg	1.88	1.77	1.82 1.74	1.80	Neg.	.06	.14 NS
Ca	2.21	2.20	2.18	2.20	.019	.009	9.8 NS(0.975)
К	.96 .94	.90 .95	.95 .88	.93	Neg.	.04	.50 NS
Ti	.31 26	.31 31	.29 26	.29	.006	.024	1.15 NS
Mn	29.96 29.91	29.96 29.94	29.91 29.90	29.93	.02	.02	2.10 NS
Р	.22 .20	.21 .20	.20 .19	.20	.003	.010	1.16 NS
Co	.200 .202	.203 .199	.201 .200	.201	Neg.	.0019	.48 NS
Cu	$\begin{array}{c} 1.16 \\ 1.16 \end{array}$	$\begin{array}{c} 1.16 \\ 1.15 \end{array}$	$\begin{array}{c} 1.17\\ 1.16\end{array}$	1.16	.003	.006	1.5 NS
Ni	$1.31 \\ 1.32$	$\begin{array}{c} 1.33 \\ 1.32 \end{array}$	$1.32 \\ 1.32$	1.32	.003	.006	1.50 NS
Zn	.148 .147	.149 .157	.148 .158	.151	Neg.	.0052	.73 NS
			Parts	per million			
Mo	990 1,000	980 970	970 980	982	10.4	7.1	5.33 NS

<sup>1</sup> Private Bag X3015, Randburg, 2125, South Africa.

Analytical methods:-

1. For MnO, 0.75 g of Nod-P and 1 g of Nod-A were dissolved in HCl and HNO<sub>3</sub> and taken to fumes with H<sub>2</sub>SO<sub>4</sub>. After cooling, water was added and the solutions diluted in a volumetric flask. Mn was determined on an aliquot by potentiometric titration with KMnO<sub>4</sub>.

 SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>5</sub>, MgO, CaO, K<sub>2</sub>O, TiO<sub>2</sub>, MnO, and P<sub>2</sub>O<sub>5</sub> were determined by X-ray fluorescence on a disc made by fusing 0.5-0.6-g samples with 7.5 g of a flux of 10 percent Li<sub>2</sub>B<sub>1</sub>O<sub>7</sub>, 51 percent Na<sub>2</sub>B<sub>1</sub>O<sub>7</sub>, and 39 percent Na<sub>2</sub>CO<sub>3</sub>.

3. For Co, Cu, Mo, Ni, and Zn, 0.4 g samples were dissolved with 10 mL HNO<sub>3</sub> 20 mL HF, and 3 mL HClO<sub>4</sub> in platinum dishes. Solutions were funed to incipient dryness and 3 mL HCl added. Solutions were warmed and 3-4 drops of H<sub>2</sub>O<sub>2</sub> added to oxidize Mn. The resulting clear solutions were transferred to volumetric flasks and diluted to the mark. Appropriate dilutions of these solutions were measured by atomic-absorption spectrometry.

4. The Canadian sample, CSRM-SU-1, was used as a control sample; results are listed as follows:

	National Institute for Metallurgy	Certified Value
Coppm	568, 553, 581, 569	630
Cupercent	0.845, 0.845, 0.846, 0.838	0.87
Moppm	<40, <40, <40, <40	
Nipercent	1.46, 1.46, 1.45, 1.46	1.51
Znppm	319, 311, 304, 312	294

			Bottle			Standard	deviation	F
Element	Method <sup>2</sup>	10/6	13/24	49/29	Mean	Bottle (d.f.=2)	Error (d.f=3)	ratio
Si	GR	1.72 1.70	1.71	1.72 1.77	1.73	Neg.	0.03	0.72 NS
Fe(T)	SA	10.92	10.88	10.91	10.91	Neg.	.03	.57 NS
Mg	AA	2.90	2.92	2.95	2.93	.02	.02	1.97 NS
Ca	GR	$11.02 \\ 11.02$	11.03 11.00	$11.05 \\ 10.90$	11.00	Neg.	.062	.31 NS
Ca	AA	11.08 10.98	$11.06 \\ 11.15$	$11.15 \\ 11.01$	11.07	Neg.	.079	.46 NS
К	FE	.46 .47	.45 $.46$	.46 $.46$	.46	.003	.006	1.5 NS
Mn(T)	РТ	$18.50 \\ 18.51$	$18.51 \\ 18.48$	18,54 18,51	18.51	.009	.018	$1.47~\mathrm{NS}$
Р	SA	.52	.53	.52	.53	Neg.	.006	.50 NS
Ba	ES	.256 .230	.260 .237	.204	.232	.022	.014	5.86 NS
Co	AA	.312	.315	.312	.313	Neg.	.0013	.70 NS
Со	SA	.322	.321	.322	.322	.0008	.0004	9.00 NS
Cu	AA	.107 .112	.108 .109	.108 .112	.109	Neg.	.003	.17 NS
Мо	ES	.0527 0534	$.0546 \\ 0497$	.0487	.0514	.0015	.0020	2.04 NS
Ni	SA	.635	.635	.633	.637	Neg.	.004	.15 NS
Pb	AA	.085	.082	.083	.084	.002	.001	3.77 NS
Sr	AA	.18 .17	.18 .18	.17 .18	.177	Neg.	.0058	.50 NS
Tl	ES	$.0045 \\ .0077$	.0073 .0040	.0073 .0057	.00608	Neg.	.0020	.09 NS
v	ES	$.0964 \\ .0951$	.1013 .0878	$.0848 \\ .0843$	.0916	.0047	.0055	2.46 NS
Zn	AA	.061 .063	.061 .061	$.061 \\ .064$	.062	Neg.	.0015	.54 NS

TABLE 20.—Determinations of several elements in USGS-Nod-A-1 by the Institute of Geological Sciences<sup>1</sup>[Determinations in percent. T, total; d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratio tested at F0.85.Analysts: N. Cogger, chemical analysis, and Linda Ault, spectrographic analysis]

<sup>1</sup> 64/78 Gray's Inn Rd., London, WC1X 8NG, England.

.

<sup>2</sup> Analytical methods:-AA, atomic-absorption spectrometry; ES, emission spectrography; FE, flame-emission spectrophotometry; GR, gravimetry; PT, potentiometric titrimetry; and SA, solution absorptiometry.

 TABLE 21.—Determinations of several elements in USGS-Nod-P-1 by the Institute of Geological Sciences<sup>1</sup>

 [Determinations in percent. T, total; d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained; F ratio tested at F0.65.

 Analysts: N. Cogger, chemical analysis, and Linda Ault, spectrographic analysis]

			Botle			Standard	leviation	TE .
Element	Method <sup>2</sup>	25/31	57/3	58/27	Mean	Bottle $(d.f.=2)$	Error (d.f.=3)	ratio
Si	GR	6.36	6.36	6.44	6.38	0.02	0.02	2.40 NS
Fe(T)	SA	6.06	6.05 6.09	6.03 6.03	6.05	.02	.02	2.40  NS
Mg	AA	2.01	2.01	1.99	2.00	Neg.	.03	.45 NS
Ca	AA	2.20	2.26	2.04 2.21	2.20	.02	.04	1.62 NS
К	FE	1.00	1.10	1.04	1.05	Neg.	.039	.63 NS
Mn(T)	РТ	29.80	29.95	29.87	29.86	Neg.	.065	.46 NS
Р	SA	.20	29.85 .21	.20	.202	~0	.004	1.00 NS
Ba	ES	.20 .397	.20 .377	.383	.374	Neg.	.025	.15 NS
Co	AA	.229	.229	.226	.227	.0009	.0013	1.90 NS
Co	SA	.228 .231	.220	.220	.231	.001	.002	1.48 NS
Cu	AA	1.15	1.17	1.13	1.15	.006	.011	1.50 NS
Мо	ES	.0706	.0678	.0631	.0678	.0026	.0015	7.41 NS
Ni	SA	1.34	1.36	1.34	1.35	.01	.00	Indet.
Pb	AA	.046	.048	.048	.047	.0004	.0009	1.40 NS
Sr	AA ES	.0177	.0161	.0183	.09 .0154	Neg.	.0033	.08 NS
V	ES	.0140 .0588	.0155 .0556	.0111 .0557	.0548	Neg.	.0027	.48 NS
Zn	AA	.0539 .164 .165	.0529 .167 .167	.0520 .164 .167	.166	.0009	.0013	1.90 NS

<sup>1</sup> 64/78 Gray's Inn Rd., London, WC1X 8NG, England.

<sup>2</sup> Analytical methods:—AA, atomic-absorption spectrometry; ES, emission spectrography; FE, flame-emission spectrophotometry; GR, gravimetry, PT, potentiometric titrimetry; and SA, solution absorptiometry.

# TABLE 22.—Determinations of several elements by atomic-absorption spectrometry in USGS-Nod-A-1 and USGS-Nod-P-1 by David Piper, U.S. Geological Survey 1

[Determinations in percent. Data are averages of two determinations]

	_	USGS-N	od-A-1		USGS-Nod-P-1			
Element	Bottle				Bottle			
	35/11	45/21	45/24	Average	2/15	33/11	44/10	Average
<sup>r</sup> e	10.6	10.5	10.7	10.6	6.49	6.31	6.54	6.45
In	17.1	16.9	16.8	16.9	27.6	27.1	27.3	27.3
Co	.30	.30	.31	.30	.22	.22	.22	.22
Cu	.088	.087	.088	.088	1.11	1.11	1.11	1.11
Vi	.61	.61	.61	.61	1.28	1.25	1.25	1.26
'n	.047	.044	.047	.046	.14	.14	.14	.14

<sup>1</sup> Stop 97, 345 Middlefield Rd., Menlo Park, CA 94025.

		USGS-N	lod-A-1		USGS-Nod-P-1			
Element4		Bottle			-	Bottle		
	4/27	28/21	32/6	- Average	26/19	50/25	51/26	Average
<sup>7</sup> e	10.4	10.3	10.0	10.2	5.13	5.26	4.74	5.04
/In	17.6	17.0	17.3	17.3	27.2	26.9	29.4	27.8
Co	.28	.28	.29	.28	.20	.20	.20	.20
Cu	.11	.11	.11	.11	1.12	1.14	1.12	1.13
Ni	.58	.59	.59	.59	1.24	1.26	1.21	1.24

TABLE 23.—Determinations of several elements by flame atomic-absorption spectrometry in USGS-Nod-A-1 and USGS-Nod-P-1 by Lockhead Ocean Laboratory <sup>1</sup>

[Determinations in percent]

<sup>1</sup> 3380 North Harbor Dr., San Diego, CA 92101.

#### TABLE 24.—Complete analyses of USGS-Nod-A-1 by M. Saunders, Grant Institute of Geology<sup>1</sup> [Determinations in percent]

Oxide or		Bottle		Avera	ge as	Math of 9
element	21/21	28/13	54/28	Oxide	Element	Method 2
SiO <sub>2</sub>	3.91	3.96	3.90	3.92	1.83	С
Al <sub>2</sub> O <sub>3</sub>	4.34	4.36	4.35	4.35	3.26	Α
$Fe_2O_3$	15.82	15.75	15.72	15.76	11.02	С
MgO	4.76	4.75	4.71	4.74	2.86	Α
CaO	15.70	15.68	15.69	15.69	11.22	G
Na₂O	1.09	1.12	1.12	1.11	.82	F
K <sub>2</sub> O	.55	.55	.56	.55	.46	F
TiO <sub>2</sub>	.47	.46	.47	.47	.28	С
$P_2 O_5$	1.23	1.22	1.21	1.22	.53	С
MnO	23.81	23.97	23.97	23.92	18.51	т
CO <sub>2</sub>	11.59	11.56	11.59	11.61		G
CoO	.43	.44	.45	.44	.34	Α
Cu0	.139	.138	.140	.139	.111	Α
NiO	.90	.91	.88	.90	.71	Α
Excess O	5.31	5.30	5.29		5.30	Т
H₂O+				~7.9		G
Ba0	.19	.20	.20	.20	.18	Α
Cl	.54	.54	.54		.54	G
<b>MoO</b> <sub>3</sub>	.062	.061	.060	.061	.041	Α
РЬО	.100	.104	.107	.104	.096	Α
Sr0	.203	.208	.197	.203	.172	Α
V <sub>2</sub> O <sub>5</sub>	.108	.110	.110	.109	.061	Α
ZnO	.082	.080	.080	.081	.065	Α
Subtotal	99.23	99.47	99.24			
Less 0=Cl	.12	.12	.12			
Total	99.11	99.35	99.12			

<sup>1</sup> University of Edinburgh, West Mains Rd., Edinburgh EH9 3JW, Scotland.

<sup>2</sup> Analytical methods:—A, atomic-absorption spectrometry; C, colorimetry; F, flame photometry; G, gravimetry; and T, titrimetry.

Notes:—Both dried nodule samples rapidly absorbed moisture during weighing. When exposed to the atmosphere overnight. Nod-A-1 absorbed between 11 and 13 percent H<sub>2</sub>O and Nod-P-1, between 8 and 10 percent H<sub>2</sub>O. Inconsistent data were obtained for  $H_2O + by$  two methods, and the results are reported as an approximate value.

Oxide or		Bottle		Avera	ge as	35.433.9
element	31/2	1/5	47/15	Oxide	Element	• Method <sup>2</sup>
SiO <sub>2</sub>	14.61	14.60	14.63	14.61	. 6.82	G
Al <sub>2</sub> O <sub>3</sub>	5.12	5.19	5.12	5.14	3.85	A
Fe <sub>2</sub> O <sub>3</sub>	8.50	8.48	8.41	8.46	5.91	С
MgO	3.29	3.32	3.27	3.29	1.98	Ã
CaO	2.91	2.87	2.91	2.90	2.07	A
Na <sub>2</sub> O	2.21	2.26	2.23	2.23	1.65	F
K₂O	1.23	1.21	1.21	1.22	1.01	F
ΓiO <sub>2</sub>	.48	.47	.46	.47	.28	С
P <sub>2</sub> O <sub>5</sub>	.48	.48	.48	.48	.21	С
MnO	38.64	38.67	38.65	38.65	29.92	Т
CO <sub>2</sub>	.69	.77	.82	.76		G
CoO	.32	.34	.34	.33	.26	А
CuO	1.71	1.70	1.66	1.69	1.35	Α
NiO	1.98	2.01	1.99	1.99	1.56	Α
Excess O	8.42	8.40	8.42		8.41	т
H <sub>2</sub> O+				~8.2		G
BaO	.33	.32	.32	.32	.29	Α
Cl	.15	.14	.14		.14	С
MoO <sub>3</sub>	.113	.113	.108	.111	.074	Α
PbO	.058	.060	.057	.058	.054	Α
Sr0	.091	.086	.090	.089	.075	Α
$V_2O_5$	.092	.094	.095	.094	.053	Α
ZnO	.208	.210	.213	.210	.169	Α
Subtotal	99.83	99.99	99.82			
Less O=Cl	.03	.03	.03			
Total	99.80	99.96	99.79			

 TABLE 25.—Complete analyses of USGS-Nod-P-1 by M. Saunders, Grant Institute of Geology<sup>1</sup>

 [Determinations in percent]

<sup>1</sup>University of Edinburgh, West Mains Rd., Edinburgh EH9 3JW, Scotland.

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<sup>2</sup> Analytical methods:—A, atomic-absorption spectrometry; C, colorimetry; F, flame photometry; G, gravimetry; and T, titrimetry.

Notes:-Both dried nodule samples rapidly absorbed moisture during weighing. When exposed to the atmosphere overnight, Nod-A-1, absorbed between 11 and 13 percent H<sub>2</sub>O and Nod-P-1, between 8 and 10 percent H<sub>2</sub>O. Inconsistent data were obtained for  $H_2O + by$  two methods, and the results are reported as an approximate value.

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#### 30 USGS ROCK STANDARDS: MANGANESE-NODULE SAMPLES USGS-NOD-A-1, USGS-NOD-P-1

#### TABLE 26.—Determinations of several elements in USGS-Nod-A-1 by David Felix, Ocean Mining Laboratory 1

[Determinations in percent or in parts per million, as indicated. Data for day 1 for all elements are not included in the analysis of variance. S, significant; NS, not significant; VS, very significant (calculated  $F > F_{0.055}$ ). Means and F ratios from the analysis of variance for the complete sets of three data on days 2 and 3. Degrees of freedom for mean sums of squares: bottles, 2; days, 1; and residual (error), 2. F ratios tested at  $F_{0.055}$  or the fractile indicated in parentheses. For any element except Pb, the upper mean is for all data and the lower is for the six data used in the analysis of variance and for best values]

Tal	Dec		Bottle			F ratio		
Element	Day	1	2	3	Mean —	Bottles	Days	
· · · · · · · · · · · · · · · · · · ·				Percent		_		
Al	1	$\begin{array}{c} 1.78\\ 2.14\end{array}$	1.84 $2.00$	1.81 $2.00$	$\begin{array}{c} 1.96 \\ 2.04 \end{array}$	<1 NS	16.54 NS(0.99)	
Fe	$3_{}$ $1_{}$ $2_{}$	$2.03 \\ 11.2 \\ 11.1$	$2.04 \\ 11.3 \\ 11.2$	$2.01 \\ 11.2 \\ 11.2$	$11.38 \\ 11.43$	2.0 NS	16.0 NS(0.99)	
Mn	$ \frac{3}{1} \\ \frac{1}{2} $	$11.6 \\ 18.2 \\ 18.0$	$12.0 \\ 18.5 \\ 19.0 \\$	$11.6 \\ 18.5 \\ 18.2$	17.84 17.57	1.82 NS	32.65 S(0.995)	
Co	3 1	16.8 .330	16.8 .329	16.6 .329	.3340		32.00 D(0.999)	
Cu	2 3 1	.337 .336 .10	.340 .334 .11	.338 .333 .11	.3363 .110	<1 NS	30.5 S(0.995)	
N;	2 3	.11 .11 .67	.12 .11	.11 .11	.112	(No test)	(No test)	
MI	2 3	.67 .66 .66	.68 .65 .66	.67 .65 .66	.657	<1 NS	11.45 NS(0.99)	
			I	Parts per million				
Cr	1 2	13 21	14 22	13 22	$\begin{array}{c} 20.8\\ 24.5\end{array}$	<1 NS	160.9 VS	
Mo <sup>2</sup>	3 1 2	28 420 430	26 420 430	$\begin{array}{c} 28\\ 420\\ 440\\ 420\end{array}$	$\begin{array}{c} 425.6\\ 428.3\end{array}$	4.19 NS	13.44 NS(0.99)	
Pb <sup>2</sup> V <sup>2</sup>	a 1	420 1,260 850	420 1,280 870	430 1,260 850	$1,267 \\ 830.0$	(No test)	(No test)	
7 9	2	800 830	800 840	800 830	816.6	3.08 NS	73.19 VS	
2n *	$\begin{array}{c}1 \\ 2 \\ 3 \\ \end{array}$	630 600 580	620 620 580	600 590 580	600.0 591.7	2.71 NS	11.29 NS(0.99)	

<sup>1</sup> Kennecott Exploration, Inc., 3377 Carmel Mt. Rd., San Diego, CA 92121.

<sup>2</sup> Data for element converted from percent, and nonsignificant zero added.

Analytical method:---1-g portions of the dried samples were digested in teflon beakers with hot HCl, HClO4, and HF to form dense perchloric fumes. After making to volume, appropriate dilutions were made to bring concentrations of each metal to its optimum range for atomic absorption. Determinations were made without background corrections on a Perkin-Elmer model 305 atomic-absorption unit. In-house standards and analyzed samples, plus standards prepared from spectrographic-quality metals, were used to bracket the unknowns. Calibration curves were not used.

TABLE 27.—Determinations of several elements in USGS-Nod-P-1 by David Felix, Ocean Mining Laboratory 1

[Determinations in percent, or in parts per million, as indicated. Data for day 1 and the second listed determination for day 3 for all elements are not included in the analysis of variance. S, significant; NS, not significant; VS, very significant (calculated  $F > F_{0.005}$ ). Means and F ratios from the analysis of variance for the complete sets of three data on days 2 and 3. Degrees of freedom for mean sums of squares: bottles, 2; days, 1; and residual (error), 2. F ratios tested at  $F_{0.05}$  or the fractile indicated in parentheses. For any element except Pb, the upper mean is for all data and the lower is for the six data used in the analysis of variance and for best values.]

$\begin{array}{c c c c c c c c c c c c c c c c c c c $		D	······································	Bottle		Maan	F	ratio
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Element	Day	1	2	3	Mean —	Bottles	Days
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$					Percent			
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Al	1	2.45		2.42	2.819		
$ \begin{array}{c} \begin{array}{c} 3 \\ 3 \\ 3 \\ 2 \\ 2 \\ 2 \\ 2 \\ 3 \\ 2 \\ 2 \\$		2	2.40	2.39	2.40	2.893	1.13 NS	158 NS(0.995)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		3	3.54	3.26	3.37			
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		3		3.14				
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Fe	1	6.2		6.1	6.33		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		2	6.8	6.8	7.2	6.47	<1 NS	49 NS(0.99)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		3	6.0	6.0	6.0			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	М.,	3		5.9		90.94		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Mn	1	30.4	07.0	30.2	29.34	CI NO	200 VG (0 00F)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		2	27.4	27.0	21.0	20.01		320 VS(0.995)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		0 9	50.4	30.3 20.2	30.0			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Co	0 1		30.3	240	9401		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	00	9	249	238	240	2410	<1 NS	175 NS
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		3	241	243	242	.2110		1110 115
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		3		.239				
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Cu	1	1.17		1.17	1.163		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		2	1.14	1.14	1.14	1.158	<1 NS	100.5 NS(0.995)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		3	1.18	1.18	1.17		•	
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		3		1.18				
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	Ni	1	1.41		1.40	1.388		
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		2	1.37	1.37	1.38	1.382	1.3 NS	3.42 NS
$\begin{array}{c c c c c c c c c c c c c c c c c c c $		3	1.39	1.40	1.38			
$\begin{tabular}{ c c c c c c c c c c c c c c c c c c c$		3		1.40				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$				Pa	arts per million	l		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Cr	1	30		18	18.9		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		2	19	19	19	17.5	<sup>3</sup> (No test)	³ (No test)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		3	16	16	16			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		3		17				
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Mo <sup>2</sup>	1	700		720	672.2		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		2	650	650	650	658.3	<1 NS	24.47 NS(0.975)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		3	670	670	660			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	<b>T</b> )] 9	3		680		000.0		DO OF MO(O OFF)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	PD*	2	650	640	640	698.3	< 1 NS	30.25 NS(0.975)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		J	740	790	730			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	<b>V7</b> 2	0 1	500	130	500	669 9		
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	v	9	650	650	690	003.3 679.9	<1 NG	970 NS(0075)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		3	700	600	790	019.9		21.0 100(0.915)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		3	100	750	140			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	Zn <sup>2</sup>	1	1.670	100	1.660	1.638		
$3_{}$ 1,640 1,630 1,640 3 1,660		2	1.620	1.620	1,600	1.625	<1 NS	6.97 NS
3 1 660		3	1.640	1.630	1.640	_,0_0	1. AIN	
		3		1,660				

<sup>1</sup> Kennecott Exploration, Inc., 3377 Carmel Mt. Rd., San Diego, CA 92121.

<sup>2</sup> Data for element converted from percent, and nonsignificant zero added.

<sup>3</sup> All variation in the analysis of variance due to days.

Analytical method:---l-g portions of the dried samples were digested in teflon beakers with hot HCl, HClO<sub>4</sub>, and HF to form dense perchloric fumes. After making to volume, appropriate dilutions were made to bring concentrations of each metal to its optimum range for atomic absorption. Determinations were made without background corrections on a Perkin-Elmer model 305 atomic-absorption unit. In-house standards and analyzed samples, plus standards prepared from spectrographic-quality metals, were used to bracket the unknowns. Calibration curves were not used.

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 TABLE 28.—Determinations of several elements in USGS-Nod-A-1 by atomic-absorption spectrometry by Wayne Mountjoy, James G. Crock, and George Riddle, U.S. Geological Survey<sup>1</sup>

[Determinations in percent or in parts per million, as indicated. Alkalies, alkaline earths, and iron were determined by Mountjoy, and other elements by Crock and Riddle. d.f., degrees of freedom; NS, not significant. F ratios tested at  $F_{0.85}$  or the fractile indicated in parentheses]

	·····	Bottle			_	F ratio					
Element	Run	1	2	3	Mean	Bottle (2 d.f.)	Run (1 d.f.)				
Percent											
Fe	1	10.8	10.9	10.8	10.88	2.00 NS	4.00 NS				
Mg	12	2.90	2.90	2.87 2.87	2.893	16.0 NS	(No test)				
Ca	1 2	11.2 11.0	11.1 11.1	11.0 11.1	11.08	<1 NS	<1 NS				
Na	1	.77	.79	.79	.783	<1 NS	(No test)				
К	1	.518	.508	.511	.510	<1 NS	<1 NS				
Cu	1	.1070	.1060	.1070	.1072	1.78 NS	1.21 NS				
Ni	1 = 2 = 2	.5690 .5720	.5800 .5750	.5600 .5750	.5718	1.05 NS	<1 NS				
			I	arts per million							
Ag Cd	1 9	 6 6	777	7	$<^{5}_{6.5}$	<1 NS	<1 NS				
Li	1	72 71	72	72 72	72	1.0 NS	No test				
Pb	2 1 2	845 850	855 855	73 845 855	850.8	2.32 NS	3.04 NS				
Rb <sup>2</sup>	1	9 19	9	10	11	2.94 NS	98 NS(0.99)				
Sr	1 2	1,510 1,490	1,500 1,500	1,500 1,500	1,500	(No test)	<1 NS				
Zn	1 $2$ $2$ $2$ $2$ $2$ $2$ $2$ $2$ $2$ $2$	590 588	608 590	593 597	594.3	<1 NS	<1 NS				

<sup>1</sup> Stop 928, Box 25046 DFC, Lakewood, CO 80225.

<sup>2</sup> Determinations near the limit of estimation of the method.

TABLE 29.—Determinations of several elements in USGS-Nod-P-1 by atomic-absorption spectrometry by Wayne Mountjoy,James G. Crock, and George Riddle, U.S. Geological Survey 1

[Determinations in percent or in parts per million, as indicated. Alkalies, alkaline earths, and iron were determined by Mountjoy, and other elements by Crock and Riddle. d.f., degrees of freedom; NS, not significant. F ratios tested at  $F_{0.85}$  or the fractile indicated in parentheses]

		Bottle				F ratio		
Element	Run	1	2	3	Mean	Bottle (2 d.f.)	Run (1 d.f.)	
				Percent				
Fe	1	5.82	5.80	5.81	5.845	1,2 NS	7.3 NS	
Mg	1	2.01	2.01	2.01	2.005	1.0 NS	3.0 NS	
Ca	1 2	2.17 2.20	2.20 2.19	2.00 2.20 2.21	2,195	1.0 NS	<1 NS	
Na	1 2	$1.62 \\ 1.61$	$1.62 \\ 1.62$	1.60 1.61	1.613	2.0 NS	(No test)	
К	1 2	1.08 1.04	1.08 1.04	1.07 1.04	1.058	<1 NS	100.5 NS(0.995)	
Cu	1 2	1.08 1.08	1.08 1.08	$1.06 \\ 1.08$	1.077	2.0 NS	2.0 NS	
Ni	$\begin{array}{c} 1 \\ 2 \\ \end{array}$	$\begin{array}{c} 1.25 \\ 1.25 \end{array}$	$1.21\\1.23$	$\begin{array}{c} 1.26 \\ 1.26 \end{array}$	1.243	7.13 NS	<1 NS	
				Parts per millior	1			
Ag					<5			
Cd	1	22	22	23	22.3	(No test)	(No test)	
Li	1	139	139	139	137.5	1.0 NS	6.75 NS	
Рb	$\frac{2}{1}$	$138 \\ 485 \\ 400$	136 480 495	$134 \\ 490 \\ 400$	488.3	<1 NS	2.24 NS	
Rb	1	22 24	455 24 24	21 23	23.0	3.08 NS	4.15 NS	
Sr	12		655 660	645 $660$	655.8	<1 NS	6.23 NS	
Zn	$1_{}$ $2_{}$	1,580 1,590	1,570 1,600	1,600 1,600	1,590	1.29 NS	2.29 NS	

<sup>1</sup> Stop 928, Box 25046 DFC, Lakewood, CO 80225.

TABLE 30.—Chemical determinations of several constituents in USGS-Nod-A-1 by Sarah T. Neil, U.S. Geological Survey<sup>1</sup> [Determinations in percent. Data, except those for F, Cl, S, Ni, Co, and Cu, were reported as oxides on sample portions not dried. All data were calculated to the dry basis by the individual determinations of H<sub>2</sub>O- (at 105°C), given in parentheses as the last entries in the table. Oxides were then converted to elements by gravimetric factors. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratios tested at F<sub>0.95</sub>]

	Bottle				Standard	<b>D</b>	
or oxide	14/32	33/15	38/10	Mean	Bottle (d.f.=2)	Error (d.f.=3)	ratio
Si	1.66 1.72	$1.66 \\ 1.68$	1.71	1.69	Neg.	0.03	0.64 NS
Al	1.94	1.90 1.90	1.90	1.93	0.02	.03	2.02 NS
Fe	10.99	11.09 10.99	10.99	11.01	Neg.	.04	.90 NS
Mg	2.86	2.92	2.91 2.90	2.91	Neg.	.03	.97 NS
Са	$11.28 \\ 11.30$	11.27 11.26	$11.31 \\ 11.26$	1 <b>1.2</b> 8	Neg.	.02	.70 NS
Na	.88 .88	.88 .87	.87 .92	.88	Neg.	.02	.50 NS
К	$.51 \\ 50$	.49 .49	.49 .53	.50	Neg.	.02	.76 NS
H <sub>2</sub> O+	8.26 8.37	8.29 8.40	8.38 8.41	8.35	Neg.	.06	.78 NS
Ti	.37	.38	.38	.38	.00	.00	1.00 NS
Р	.53	.54	.54	.54	.00	.00	3.00 NS
CO <sub>2</sub>	11.80 12.09	$12.12 \\ 12.06$	$12.18 \\ 12.14$	12.07	.07	.12	1.62 NS
F	.10 .10	.10 .10	.10 .10	.10			(No test)
Cl	.42	.41	.42	.43	Neg.	.02	.20 NS
S	.40	.45	.39	.40	.03	.02	7.19 NS
Mn	18.03 17.80	18.17 18.31	$18.21 \\ 18.24$	18.13	.17	.11	5.50 NS
Ni	.65	.65	.65	.65	Neg.	.01	.50 NS
Co	.35	.34 .34	.34 .35	.34	Neg.	.01	.20 NS
Cu	.12	.10 .12	.12	.11	Neg.	.01	.50 NS
(H <sub>2</sub> O—)	(13.45) (13.35)	(14.12) (14.02)	(13.82) (13.79)				

<sup>1</sup> Menlo Park, CA 94025.

Analytical methods:-

Gravimetry: Total H2O (Penfield); H2O-; SiO2; Al2O3 (weighed as AlPO4); CaO (weighed as CaCO3); MgO (weighed as Mg2P2O7); and total S (weighed as BaSO4).

Other methods: Na2O and K2O, by flame photometry; TiO2, by spectrophotometry with Tiron; P2O5 by spectrophotometry with molybdovanadate method; Ni, Co, and Cu, by atomic-absorption spectrometry; total Fe, by spectrophotometry with orthophenanthroline; F, by F- electrode; Cl, by spectrophotometry using Fe(CNS)3; CO2, as total C, by combustion with Leco WR-12; and total Mn, volumetrically with sodium bismuthate. TABLE 31.—Chemical determinations of several constituents in USGS-Nod-P-1 by Sarah T. Neil, U.S. Geological Survey 1

[Determinations in percent. Data, except those for F, Cl, S, Ni, Co, and Cu, were reported as oxides on sample portions not dried. All data were calculated to the dry basis by the individual determinations of H<sub>2</sub>O- (at 105°C), given in parentheses as the last entries in the table. Oxides were then converted to elements by gravimetric factors. d.f., degrees of freedom; NS, not significant; Neg., negative bottle variance obtained. F ratios tested at F<sub>0.05</sub> or the fractile indicated in parentheses.]

Floment	Bottle				Standard		
or oxide	21/10	23/3	36/8	Mean	Bottle (d.f.=2)	Error (d.f.=3)	ratio
Si	6.60 6.56	6.57 6.55	6.56 6.55	6.57	0.00	0.02	1.00 NS
Al	2.47	2.47	2.48 2.49	2.47	.01	.02	2.17 NS
Fe	5.95 5.95	5.95 5.92	5.97 5.94	5.95	Neg.	.02	.72 NS
Mg	2.04 2.08	2.03 2.06	2.06 2.18	2.08	.01	.05	1.12 NS
Ca	$2.26 \\ 2.27$	2.26 2.32	2.37 2.40	2.31	.06	.03	10.46 NS(0.975)
Na	$\begin{array}{c} 1.74 \\ 1.72 \end{array}$	$\begin{array}{c} 1.73 \\ 1.73 \end{array}$	$\begin{array}{c} 1.72 \\ 1.74 \end{array}$	1.73	Neg.	.01	.00 NS
К	1.06	1.05	1.04	1.05	Neg.	.01	.17 NS
H <sub>2</sub> O+	8.47 8.47	8.59	8.62 8.72	8.57	.09	.05	8.85 NS
Ti	.36	.38	.37 .37	.37	.00	.01	1.00 NS
Р	.20 .20	.21 .22	.21 .21	.21	.01	.00	7.00 NS
CO <sub>2</sub>	.81 .81	.83 .84	.80 .85	.82	Neg.	.02	.73 NS
F	.04 .03	.03 .04	.03 .04	.04	Neg.	.01	.00 NS
Cl	.17 11	.10 12	.12 11	.12	Neg.	.03	.76 NS
S	.20	.20	.20 .22	.20	.01	.01	1.46 NS
Mn	$29.78 \\ 29.67$	29.79 29.88	$29.74 \\ 29.58$	29.74	.06	.09	2.05 NS
Ni	$1.35 \\ 1.37$	$\begin{array}{c} 1.40 \\ 1.37 \end{array}$	$1.37 \\ 1.38$	1.37	.01	.02	1.36 NS
Co	.25 .25	.25 .26	.24 .24	.25	.01	.00	7.00 NS
Cu	$\begin{array}{c} 1.18\\ 1.18\end{array}$	$\begin{array}{c} 1.15 \\ 1.20 \end{array}$	$1.19 \\ 1.15$	1.18	Neg.	.03	.07 NS
(H <sub>2</sub> O—)	(6.70) (6.70)	(7.19) (7.25)	(6.39) (6.29)				

<sup>1</sup> Menlo Park, CA 94025.

Analytical methods:—

Gravimetry: Total H2O (Penfield); H2O-; SiO2; Al2O3 (weighed as AlPO4); CaO (weighed as CaCO3); MgO (weighed as Mg2P2O7); and total S (weighed as BaSO4).

Other methods: Na<sub>2</sub>O and K<sub>2</sub>O, by flame photometry; TiO<sub>2</sub>, by spectrophotometry with Tiron; P<sub>2</sub>O<sub>5</sub> by spectrophotometry with molybdovanadate method; Ni, Co, and Cu, by atomic-absorption spectrometry; total Fe, by spectrophotometry with orthophenanthroline; F, by F<sup>-</sup> electrode; Cl, by spectrophotometry using Fe(CNS)s; CO<sub>2</sub>, as total C, by combustion with Leco WR-12; and total Mn, volumetrically with sodium bismuthate.

#### TABLE 32.—Averages of determinations of elements

[Averages in percent or in parts per million as indicated. Averages are indicated by the table number and the number of determinations (n) for surviving the selection process

						10				
Table No.	7	28	9	22	11	12	14			
Element	<u>n=6</u>	n=6	n=6	n=3	n=6	<u>n=6</u>	<u>n=6</u>			
Percent										
Si			1.82				1.42			
A1			1.75			1.7	2.27			
Fe	10.88	10.88	10.86	10.6		9.7	10.98			
Mg		2.89	2.85				3.20			
Ca		11.08	11.17				11.42			
Ca										
Na		.78	.80							
K		.510	.50			.17	.48			
Ti			.32	10.0		10 7	.29			
Mn			18.40	16.9		13.7	18.64			
r	9597		.42				.09			
00	1202.		.820	.00		.01	.09			
Co		1070								
Cu		.1072	.112	.088		.11	.18			
		.5718	.648	10.		.04	.57			
H-O-L							11.04			
Excess $0$										
			Parts per mil	lion						
Ag		< 5	~							
As			~~~~				298			
Ba	1,518					2,700	1,671			
Be										
Cd		6.5								
Ce	800									
Cl										
Cr	25.9									
Eu	4.48									
GQ	20.0									
111 T.9	132.5									
	104.0									
	0.10	72								
	2.16					260				
Nd	85.3					900	400			
Ph	00.0	851				950	1 323			
Rb		11				·	10			
Sh	33 5									
Sc	11.23									
Sm	20.9									
Sr		1,500				1,800	1,728			
Tb	4.87									
Th	25.8				27.35					
T1										
Tm	1 1.72									
Ų	5.95				6.86					
V		*******								
I Vh	16.9						115			
±N	10.9									
Zn		594		460		590	870			
Zr							317 -			

<sup>1</sup> Average of four. <sup>2</sup> Averag

<sup>2</sup> Average of three.

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#### in USGS-Nod-A-1 by all contributing analysts

each average. Lower case letter "o" indicates nonsignificant zeros added to values for trace-element averages. Bold-faced numbers indicate averages and estimates calculated from them]

	16	18	20	24	26	23	30	"Best"
-	n=6	n=6	n=6	n=3	n=6	n=3	n=6	value
		· · · · · · · · · · · · · · · · · · ·		Percent				
Si	2.00	1.63	1.73	1.83			1.69	1.78
A1	2.06	1.86		3.26	2.04		1.93	2.05
Fe	11.08	10.29	10.91	11.02	11.4	10.2	11.01	10.93
Mø	2.80	2.74	2.93	2.86			2.91	2.87
Ca	10.97	11.30	11.00	11.22			11.28	11.03
Ca			11.07					
Na	.77			.82			.88	.775
K	.52	.40	.46	.46			.50	.50
Ti	.28	.32	•10	.28			.38	.32
Mn	17.80	18.63	18.51	18.51	17.6	17.3	18.13	18.54
P	11100	.60	.53	.53		-110	.54	60
Co	.3128	.281	.313	.34	.336	.28	.34	.311
Co			.322					
Ču	.1052	.107	.109	.111	.112	.11	.11	.11
Ni	.6300	.625	.637	.71	.657	.59	.65	.636
CO <sub>2</sub>				11.61			12.07	
H <sub>0</sub> 0+				~7.9			8.35	
Excess O				5.30				
			Pa	arts per million	·····			
•								
Ag								
AS	1 669		9 290	1 800				1 670
Ba	56		2,020	1,000				1,070
C4	0.0							
Ce								
Cl				5,400			4,300	
<u>Cr</u>	23				24			
Eu								
Gd								
Hf								
La								
Li	76.3							
Lu								
Mo	457	567	514	<b>41</b> 0	428			448
Ph	793		840	060	<sup>2</sup> 1 967			846
Rb	100		040	900	1,207			040
<b>C</b> 1							*	
SD								
Sm Sm								
Sr Sr	1 660		1 770	1 790				1 749
Th	1,000		1,770	1,720				1,140
Th								
***								
Tl			61					
Tm								
U	E00		010					
V	980		916	610	817			770
I Vh								
1V								
Zn	559	574	<b>62</b> 0	650	<b>592</b>			587
<b>4</b> 4								

#### TABLE 33.—Averages of determinations of elements in

[Averages in percent or in parts per million as indicated. Averages are indicated by the table number and the number of determinations (n) for surviving the selection process

Table No.	8	29	10	22	11	13	15			
Element	n=6	n=6	n=6	n=3	n=6	n=6	n=6			
Percent										
Si			6.64				6.65			
Al	 E 770		1.97	C 45		1.8	2.59			
re Ma	5.76	5.84 2.00	5.73 1 0 9	6.40		5.2	4.76			
Ca		2.20	2.20				2.16			
Na		1.61	1.68							
<u>K</u>		1.06	.99			.28	1.14			
T1			.31	97.9		95 5	.30			
MIN P			29.19	21.5		20.0	29.00			
Co	.1808		.220	.22		.22	.28			
Co										
Cu		1.08	1 20	1 11		1.2	1 04			
Ni		1.24	1.40	1.26		1.4	1.28			
CO <sub>3</sub>							1.39			
H <sub>2</sub> O+										
Excess O										
			Parts per mil	lion			·····			
Ag		<5								
As							39			
Ba	1 2,498					6,700	3,453			
Be										
Cd		22.3								
Ce	289									
Cl										
Cr	$\sim 15$									
Cd	0.57									
Hf	43									
La	<sup>1</sup> 120									
——т;		197 5								
La La	11.85	191.9								
Mo	1.00					630	823			
Nd	112.8									
Pb		488				52o	715			
Rb		23					21			
Sb	50.1									
Sc	9.47									
Sm	30.4	050				1.00				
Sr	5 91	656				1,080	751			
10 Th	0.01 17 0				17 65					
m	17.0				11.00					
11 Tm										
	2.77				1 98					
V	0.0				4.20					
Ý							89			
Yb	<sup>1</sup> 13.8									
Zn		1.590				1 600	1 998			
Zr						1,000	280			

<sup>1</sup> Average of four.

#### USGS-Nod-P-1 by all contributing analysts

each average. Lower case letter "o" indicates nonsignificant zeros added to values for trace-element averages. Bold-faced numbers indicate averages and estimates calculated from them]

· · · · · · · · · · · · · · · · · · ·	17	19	21	25	27	23	31	"Best"
	n=6	n=6	n=6	n=3	n=6	n=3	n=6	value
	· · · · · · · · · · · · · · · · · · ·			Percent				
Si	6.97	6.36	6.38	6.82			6.57	6 51
A1	2.41	2.56	0.00	3.85	2.89		2.47	2.55
Fe	5.81	5.52	6.05	5.91	6.47	5.04	5.95	5.78
Me	1.98	1.80	2.00	1.98			2.08	1.99
Ca	2.16	2.20	2.20	2.07			2.31	2.19
Na	1.63			1.65			1.73	1.64
Κ	1.04	.93	1.05	1.01			1.05	1.05
Ti	.28	.29		.28			.37	.30
Mn	29.17	29.93	29.86	29.92	28.87	27.8	29.74	29.14
Р		.20	.20	.21			.21	.20
Co	.2280	.201	.227	.26	.241	.20	.25	.224
Co			.231					
Cu	1.137	1.16	1.15	1.35	1.16	1.13	1.18	1.15
Ni	1.328	1.32	1.35	1.56	1.38	1.24	1.37	1.34
CO2				.76			.82	
H <sub>2</sub> O+				~8.2			8.57	
Excess O				8.41				
			Pa	rts per million				
•								<u> </u>
Ag								
Ro	2 862		3 740	2.900				3 350
Be	2.8		0,110	2,000				0,000
Cd	2.0							
Ce								
C				1.400			1 900	
Cn				1,400	175		1,200	
F11	20				11.0			
Gd								
Hf								
T.a								
T:	140							
	142							
Mo	802	982	678	740	658			762
Nd	002	002	010	110				102
Ph	436		470	540	698			555
<b>R</b> b								
Sb								
Sc								
Sm								
Sr	638		~900	750				680
Tb								
Th								
Tl			154					
Tm								
<u>U</u>								
<u>V</u>	480		548	530	673			570
Y								
Yb								
Zn	1,467	1,510	1,660	1,690	1,625			1,595
Zr								

,