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# *NICKEL-COPPER-COBALT ORES SU-1 AND UM-1: THEIR CHARACTERIZATION AND PREPARATION FOR USE AS STANDARD REFERENCE MATERIALS*

Members of the Staff of the Mineral Sciences Division

(Compiled by G.H. Faye, W.S. Bowman and Sutarno)

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BY

Members of the Staff of the Mineral Sciences Division
(Compiled by G.H. Faye, W.S. Bowman and Sutarno)

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## SYNOPSIS

Two nickel-copper-cobalt ores, SU-1 and UM-1, have been prepared and characterized for use as standard reference materials.

This report describes the nature and origin of SU-1 and UM-1, and gives information on procedures used for their preparation and for assessing their homogeneity. Twenty-five laboratories provided analytical results for nickel, copper and cobalt; the recommended values are, respectively: 1.51%, 0.87% and 0.063% for SU-1, and 0.88%, 0.43% and 0.035% for UM-1. The analytical results and the evaluation of statistical parameters for the three elements are reported for both ores.

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Mineral Sciences Division, Mines Branch, Department of Energy, Mines and Resources, Ottawa, Canada.

Direction des mines, bulletin technique TB 177

# MINERAIS SU-1 ET UM-1 DE NICKEL, DE CUIVRE ET DE COBALT: LEUR CARACTERISATION ET LEUR PREPARATION POUR SERVIR DE MATERIAUX TYPES DE REFERENCE

PAR

des membres du personnel de la Division des sciences minérales
(Compilé par G.H. Faye, W.S. Bowman et Sutarno)

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## RÉSUMÉ

On a préparé et caractérisé deux minerais de nickel, de cuivre et de cobalt, SU-1 et UM-1, pour les utiliser comme matériaux types de référence.

Dans ce rapport, les auteurs décrivent la nature et l'origine de SU-1 et UM-1, et donnent des renseignements sur les méthodes utilisées pour leur préparation et la vérification de leur homogénéité. Vingt-cinq laboratoires ont fourni des résultats d'analyse du nickel, du cuivre et du cobalt; les valeurs recommandées sont, respectivement, les suivantes: 1.51%, 0.87% et 0.063% pour SU-1, et 0.88%, 0.43% et 0.035% pour UM-1. Les résultats d'analyse et l'évaluation des paramètres statistiques pour les trois éléments sont donnés pour les deux minerais.

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Division des sciences minérales, Direction des mines, ministère de l'Energie, des Mines et des Ressources, Ottawa, Canada.

CONTENTS

# INTRODUCTION

This report describes the preparation and characterization of two nickel-copper-cobalt ores, SU-1 and UM-1, as standard reference materials. The work is the fourth facet of the Canadian Standard Reference Materials Project (CSRMP) to certify samples that are representative of major Canadian ore deposits. Previously issued SRM's are a molybdenum ore, PR-1, a zinc-tin-copper-lead ore, MP-1, and a copper-molybdenum ore, HV-1 (1, 2 & 3).

SU-1 and UM-1 were chosen primarily to fulfil a need for nickel-bearing standard reference ores, but also because they were available to the CSRMP already in a bottled condition (SU-1), or in a condition suitable for bottling (UM-1); therefore, preparative work associated with the distribution of these materials was expected to be minimal. That two nickel-copper-cobalt-bearing materials were selected for simultaneous certification is related to their appreciably different origins and mineralogy. These differences are expected to be of interest and value especially to laboratories involved in the earth sciences.

SU-1 is a composite of sample rejects collected, in 1958, at the Falconbridge Nickel Mines Limited, Falconbridge, Ontario (4), and is, therefore, considered to be representative of the Sudbury nickel-copper ores. SU-1 was originally intended as a reference material primarily for use by spectroscopists (4). It has been widely distributed to laboratories throughout the world, and numerous analytical results for minor and trace elements have been accumulated (5,6). Most of the analytical results, however, were obtained by emission spectroscopy and only single values for each element were obtained from each laboratory. Because of the wide range in the results for most elements, recommended values were not assigned previously. However, the work described in this report leads to the assignment of reliable

values for nickel, copper and cobalt, and to provisional values for the trace elements, silver, gold, platinum, palladium, zinc, cadmium and bismuth.

Although UM-1 is called an ore because of its relatively high base-metal-sulphide content, it is an ultramafic rock from the Giant Mascot Mine at Hope, British Columbia. UM-1 is one of a suite of three ultramafic rocks (the others being coded as UM-2 and UM-4) that have been termed geochemical standards for the determination of ascorbic acid/hydrogen peroxide-soluble nickel, copper and cobalt (7). Because UM-1 contains ore-grade concentrations of nickel, copper and cobalt, and was available to the CSRMP in a comminuted condition, it was chosen, along with SU-1, for the certification of these three elements.

Twenty-five laboratories analysed SU-1 and UM-1 for nickel, copper and cobalt. Most of the results were obtained by atomic-absorption methods; however, many laboratories voluntarily provided additional results by an alternative method of their choice. Statistical analyses were not conclusive in detecting any significant differences between the atomic-absorption results and those obtained by other methods

This report describes the procedures used to obtain recommended values for nickel, copper and cobalt in SU-1 and UM-1; these are, respectively: 1.51%, 0.87%, and 0.063% for SU-1, and 0.88%, 0.43%. and 0.035% for UM-1. Although only provisional values are available for platinum, palladium, gold and silver in SU-1 and UM-1, these should be very valuable to analysts and geochemists because of the dearth of reference materials containing measured quantities of the noble metals at low-ppm levels.

## PREPARATION AND MINERALOGICAL DESCRIPTION OF SU-1 AND UM-1

As was mentioned previously, SU-1 is a composite of sample rejects collected in 1958 at the Falconbridge Nickel Mines Limited, Falconbridge, Ontario (4). At that time, the material was thoroughly blended (24 hours in a rotating drum) and then riffled to obtain a number of portions of approximately 100 g each; these were placed in bottles by members of what was then called the Canadian Association for Applied Spectroscopy (4). An approximate screen analysis for SU-1 is given in Table 1 and its chemical composition is given in Table 2.

The rock from which UM-1 is derived (7) is "a peridotite consisting of 80-85% olivine, 3-5% orthopyroxene (bronzite), 5% augite, 5% pale yellow hornblende, 5-10% sulphides, and 1% chromite. The rock has a cumulate-type texture, with closely packed, subhedral grains of olivine surrounded by interstitial to slightly poikilitic pyroxenes, hornblende, and sulphides. Chromite occurs as small euhedral grains, some as inclusions in the olivine. There appears to be little alteration of the rock."

Using conventional milling equipment at the Mines Branch, UM-1 was pulverized with the intention of producing minus 200-mesh material (see Table 1). This product, weighing 143 lb, was tumbled in a 45-gallon, baffled mixing drum for 8 hours. After emptying the blender, the bulk ore was randomly sampled and tested for homogeneity by X-ray fluorescence analysis. Subsequently, UM-1 was placed in 642 bottles, each containing 100 g of material. An approximate screen analysis for UM-1 is given in Table 1; its chemical composition is given in Table 2. It is to be noted that there is a difference of approximately 3% between the total of the third column in Table 2 and the calculated total given for UM-1 in Reference (7).

TABLE 1

Approximate Screen Analyses of SU-1 and UM-1

| Mesh Size (Tyler) | | SU-1 | UM-1 |
|---|---|---|---|
| | +100 | 15 wt % | -- wt % |
| -100 | +200 | 18 | 15 |
| | -200 | 67 | 85 |

TABLE 2

Provisional Chemical and Spectrographic Analyses of SU-1 and UM-1

| | SU-1 (Wt %) | | UM-1 | |
|---|---|---|---|---|
| | Chemical[a] | Emission[b] Spectrographic (Lab 25) | Chemical[c] | Emission[b] Spectrographic (Lab 25) |
| O[d] | 31.2 (5) | | 36.5 (5) | |
| Si | 16.2 (8) | | 17.6 | |
| Al | 5.01 (9) | | 0.53 | 0.53 |
| Fe | 2. .9 (8) | | 13.4 | |
| Ca | 2.86 (9) | 2.8 | 1.67 | 1.5 |
| Mg | 2.47 (8) | 2.7 | 21.7 | |
| Na | 0.77 (7) | | 0.06 | |
| K | 0.53 (7) | | 0.02 | |
| Ti | 0.50 (9) | 0.46 | 0.06 | 0.044 |
| V | | 0.03 | | 0.009 |
| Cr | | 0.05 | 0.31 | 0.33 |
| Mn | 0.08 (9) | 0.10 | 0.12 | 0.14 |
| Ni | 1.51[e] | 1.55 | 0.88[e] | 0.85 |
| Cu | 0.87[e] | 0.91 | 0.43[e] | 0.49 |
| Co | 0.063[e] | 0.060 | 0.035[e] | 0.03 |
| Zn | 0.03[f] | | 0.01 | |
| Ba | | 0.020 | | <0.001 |
| Sr | | 0.011 | | 0.001 |
| Zr | | 0.014 | | <0.007 |
| S | 12.1 (6) | | 3.53 | |
| P | 0.04 (5) | | | |
| H (from $H_2O$) | 0.33 (4) | | 0.05 | |
| C (from $CO_2$) | | | 0.07 | |

a - except where indicated otherwise, the values are taken from Ref. (5) and the number in brackets is the number of determinations.

b - average of 10 replicate determinations made on 5 separate 40-mg aliquots from each of two bottles.

c - values taken from Ref. (7), except where indicated otherwise.

d - determined by neutron activation analysis in the Mineral Sciences Division.

e - from Table 11.

f - from Table 4.

## TESTS FOR HOMOGENEITY

Prior to the distribution of samples to participating laboratories, X-ray fluorescence methods were used to confirm that both SU-1 and UM-1 were sufficiently homogeneous to be used for the purpose for which they were intended.

Each laboratory (see pp. 10 and 11) received two randomly-selected bottles of both SU-1 and UM-1 and was requested to analyse five samples from each bottle for nickel, copper, and cobalt. The reported results (Table 6) were compared between bottles within each laboratory, using the *t*-test at a 5% significance level. The results of these tests are summarized in Table 3 and are illustrated in Figures 7 to 12. Table 3 shows that, for both SU-1 and UM-1, the majority of the laboratories did not find any evidence of a difference between the two bottles they received.

TABLE 3

Summary of the *t*-Tests on Results between Bottles for Each Laboratory

| Lab No. | SU-1 | | | UM-1 | | |
|---|---|---|---|---|---|---|
| | Nickel | Copper | Cobalt | Nickel | Copper | Cobalt |
| 1 | A | A | A | A | A | A |
| 2 | R | A | A | A | R | A |
| 3 | A | A | R | - | - | A |
| 4 | A | A | A | R | A | A |
| 5 | A | A | A | A | A | A |
| 6 | R | A | A | A | R | A |
| 7 | A | A | A | A | A | A |
| 8 | A | R | R | A | A | R |
| 9 | A | A | A | A | A | A |
| 10 | A | A | A | A | A | A |
| 11 | A | A | R | A | A | A |
| 12 | A | A | A | A | A | A |
| 13 | A | A | A | A | A | R |
| 14 | A | A | A | A | A | A |
| 15 | A | A | A | A | A | A |
| 16 | A | R | A | A | A | R |
| 17 | A | A | - | A | A | - |
| 18 | A | A | A | A | A | A |
| 19 | A | A | A | A | A | A |
| 20 | A | A | A | A | R | A |
| 21 | A | A | A | A | A | A |
| 22 | A | A | A | A | A | A |
| 23 | A | A | A | A | A | A |
| 24 | A | A | A | A | A | A |
| 25 | A | A | A | A | A | A |

A = Null hypothesis accepted, i.e., there is no evidence of inhomogeneity.

R = Null hypothesis rejected, i.e., there is evidence of inhomogeneity.

## PROVISIONAL RESULTS FOR TRACE ELEMENTS IN SU-1 AND UM-1

Although analytical results for minor and trace elements in SU-1 have been reported previously (5,6), these are generally not amenable to a statistical treatment that could yield reliable recommended values. Nevertheless, provisional results can be very useful for many purposes; therefore, these are presented in Table 4 for certain trace elements. The listed elements were chosen because, in addition to the earlier results by emission spectroscopy, recent results by atomic absorption-spectrophotometry are now available. The source of these results is also given in Table 4.

The precious metals values for SU-1 and UM-1 are of interest because of the relatively good agreement between results obtained by different methods, especially at such low levels of concentration. It is to be noted that the results reported by the Analytical Chemistry Section (Lab 21) for platinum, palladium and gold were obtained using the tin-collection scheme of fire assaying (8).

There has been much recent interest in standard reference materials that have been certified for one or more of the platinum-group metals. Three such materials, PTA (9) and PTC (10), have recently been placed on sale by the CSRMP - these contain the principal platinum metals, platinum and palladium, in the 3 to 12 ppm range. Therefore, the provisional values given for SU-1 and UM-1 will partly fill the need for reference materials containing these elements at the 0.1-ppm level.

TABLE 4

Provisional Analytical Results for Certain Trace Elements in SU-1 and UM-1

| Element | SU-1 (ppm) | No. of Results | UM-1 (ppm) | Method | Source |
|---|---|---|---|---|---|
| Ag | 4.1 | 5 | | a.a. | F.J. Langmyhr and L.T. Wold, private communication, 1973. |
| | 5.2 | 10 | | a.a. | Lab 21. |
| | 3.9 | 16 | | Emission spectrographic | Reference 5. |
| | 3.0 | 3 | | " | Reference 6. |
| | Mean 4.2 | | | | |
| Au | 0.10 | 5 | | Fire assay + a.a. | Lab 21. |
| | 0.11 | 2 | | See source ref. | Reference 5. |
| | | 5 | 0.08 | Fire assay + a.a. | Lab 21. |
| | Mean 0.10 | | | | |
| Bi | 5.2 | 5 | | a.a. | F.J. Langmyhr and L.T. Wold, private communication, 1973. |
| | 5.0 | 4 | | Emission spectrographic | Reference 5. |
| | Mean 5.1 | | | | |
| Cd | 1.6 | 5 | | a.a. | Reference 11. |
| Pt | 0.16 | 5 | | Fire assay + a.a. (8) | Lab 21. |
| | 0.17 | 2 | | See source ref. | Reference 5. |
| | | 5 | 0.07 | Fire assay + a.a. (8) | Lab 21. |
| | Mean 0.16 | | | | |
| Pd | 0.12 | 5 | | Fire assay + a.a. (8) | Lab 21. |
| | 0.18 | 2 | | See source ref. | Reference 5. |
| | | 5 | 0.07 | Fire assay + a.a. (8) | Lab 21. |
| | Mean 0.14 | | | | |
| Zn | 232 | 2 | | X-ray fluorescence | Reference 12 |
| | 298 | 11 | | See source ref. | Reference 5. |
| | 294 | 4 | | " | Reference 6. |
| | Mean 289 | | | | |

a.a. = atomic absorption

## THE CERTIFICATION OF SU-1 AND UM-1 FOR NICKEL, COPPER AND COBALT

The names of the laboratories that participated in the program to certify SU-1 and UM-1 are given below in alphabetical order. Each of these was arbitrarily assigned a code number so that analytical results could be recorded while preserving the anonymity of the laboratory. The code numbers bear no relation to the alphabetical order of the laboratory names.

The reader will have noted already that no attempt has been made to hide the identity of Lab 21 which is the Analytical Chemistry Section of the Mineral Sciences Division, Mines Branch.

Participating Laboratories

Analytical Chemistry Section, Mineral Sciences Division, Mines Branch, EMR, Ottawa, Ontario.

Assayers Limited, Rouyn, Quebec.

Bondar-Clegg and Company Limited, Ottawa, Ontario.

Bondar-Clegg and Company Limited, Vancouver, British Columbia.

Can-Test Limited, Vancouver, British Columbia.

Central Laboratories and Technical Services Division, Geological Survey of Canada, EMR, Ottawa, Ontario.

Chemex Labs Limited, North Vancouver, British Columbia.

Cominco, Trail, British Columbia.

Crest Laboratories (B.C.) Limited, Vancouver, British Columbia.

Extraction Metallurgy Division, Mines Branch, EMR, Ottawa, Ontario.

Falconbridge Nickel Mines Limited, Metallurgical Laboratories, Thornhill, Ontario.

General Testing Laboratories, Vancouver, British Columbia.

Hudson Bay Mining and Smelting Company Limited, Flin Flon, Manitoba.

International Nickel Company of Canada, Limited, Sheridan Park, Clarkson, Ontario.

Lakefield Research of Canada Limited, Lakefield, Ontario.

Loring Laboratories Limited, Calgary, Alberta.

Mineral Research Branch, Ministry of Natural Resources, Toronto, Ontario.

Noranda Mines Limited, Noranda, Quebec.

Resource Geophysics and Geochemistry Division, Geological Survey of Canada, EMR, Ottawa, Ontario.

Sherritt Gordon Mines Limited, Research and Development Division, Fort Saskatchewan, Alberta.

Sherritt Gordon Mines Limited, Mining and Milling Division, Lynn Lake, Manitoba.

Spectrochemistry Section, Mineral Sciences Division, Mines Branch, EMR, Ottawa, Ontario.

Swastika Laboratories Limited, Swastika, Ontario.

Technical Services Laboratories, Toronto, Ontario.

Thunder Bay Testing Limited, Thunder Bay, Ontario.

The participating laboratories, with the exception of those within the Mineral Sciences Division of the Mines Branch, each received two randomly-selected bottles of SU-1 and UM-1. These "outside" laboratories were requested to determine nickel, copper and cobalt in each bottle, in quintuplicate, by methods of their choice. Many laboratories voluntarily submitted analytical results obtained by two different methods (Tables 6, 12 and 13). However, as in previous facets (1-3) of the standards program, atomic-absorption spectrophotometry was the most popular technique for the determination of the three metals (see Tables 12 and 13).

Because the analytical methods were intended to give total values for nickel, copper and cobalt, many laboratories used hydrofluoric acid in their sample decomposition procedures for attacking the siliceous constituents of the ores SU-1 and UM-1. When they are known, the acid mixtures used by the participating laboratories are recorded in Table 5. See Table 14 for the correlation of the results with methods of decomposition.

TABLE 5

Acid Mixtures Used for Decomposition of SU-1 and UM-1

| | Nickel | | Copper | | Cobalt | |
|---|---|---|---|---|---|---|
| | a.a. | other | a.a. | other | a.a. | other |
| Lab. 1 | F,N,P | F,N,P | F,N,P | - | F,N,P | F,N,P |
| Lab. 2 | F,N,Cl,S | F,N,Cl,S | F,N,Cl,S | F,N,Cl,S | F,N,Cl,S | - |
| Lab. 3 | Cl,N,S, | N,$Br_2$ | Cl,N,S, | Cl,N,S | Cl,N,S | Cl,N,S |
| Lab. 4 | F,N,Cl,P | - | F,N,Cl,P | - | F,N,Cl,P | - |
| Lab. 5 | - | F,N,Cl,P | | F,N,Cl,P | - | F,N,Cl,P |
| Lab. 6 | F,N,Cl,P | | F,N,Cl,P | | F,N,Cl,P | - |
| Lab. 7 | F,N,Cl,P | | F,N,Cl,P | - | F,N,Cl,P | - |
| Lab. 8 | P | P | P | P | P | - |
| Lab. 9 | F,N,Cl,P | F,N,Cl,P | F,N,Cl,P | | F,N,Cl,P | - |
| Lab. 10 | - | total decomposition procedure | | | - | - |
| Lab. 11 | F,N,Cl,P | | F,N,Cl,P | - | F,N,Cl,P | - |
| Lab. 12 | N,P | N,P | N,P | N,P | N,P | - |
| Lab. 13 | F,N,P | | F,N,P | - | F,N,P | - |
| Lab. 14 | F,N,Cl,P | F,N,Cl,S | F,N,Cl,P | F,N,Cl,S | F,N,Cl,P | unknown |
| Lab. 15 | N,P | N,Cl,S | N,P | N,Cl,S | N,P | N,Cl,S |
| Lab. 16 | - | F,N,Cl,P | F,N,Cl,P | | F,N,Cl,P | - |
| Lab. 17 | - | - | - | - | - | - |
| Lab. 18 | F,N,Cl,$Br_2$ | | F,N,Cl,$Br_2$ | | F,N,Cl,$Br_2$ | - |
| Lab. 19 | N | N | N | N | N,Cl | - |
| Lab. 20 | P,N | F,N,S | P,N | P,N | P,N | - |
| Lab. 21 | F,N,Cl,P | | F,N,Cl,P | - | F,N,Cl,P | - |
| Lab. 22 | - | - F-containing acid mixture | | | - | - |
| Lab. 23 | F,N,P | unknown | F,N,P | unknown | F,N,P | - |
| Lab. 24 | N,Cl | | N,Cl | | N,Cl | - |
| Lab. 25 | - | - | - | - | - | - |

F = hydrofluoric
N = nitric
Cl= hydrochloric
P = perchloric
S = sulphuric
a.a. = atomic absorption

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## EVALUATION OF STATISTICAL PARAMETERS

The results reported by all participating laboratories are presented in Table 6. The following procedures were used to compute the best values for the statistical parameters.

A. All Results Treated as Though They Were Independent

Figures 1 to 6 show the cumulative distribution of the results for the three selected elements. The normal parameters, the median, mean, variance, standard deviation, skewness factor, and kurtosis coefficients were computed twice, first from all the results and then from those results that deviate from the overall mean ($\bar{x}..$) by no more than twice the standard deviation(s), i.e., $[(\bar{x}.. -2s) \leq x_{ij} \leq (\bar{x}.. +2s)]$. This rejection was considered necessary to prevent the possible introduction of bias to the estimated means. The results of these computations are presented in Table 7. It is seen that the results that deviate from the mean by more than twice the standard deviation are about 7% of the total frequency for SU-1 and about 5% for UM-1. This is a reasonable proportion for normally distributed independent variables. By rejecting these results, both the skewness and the kurtosis coefficient were brought nearer to the values of a normal distribution for most elements. The kurtosis coefficient, $\alpha_4 = m_4/m_2^2$, is a measure of the sharpness of the peak of the probability density curve; for an ideal distribution $\alpha_4 = 3$. The skewness factor, $\alpha_3 = m_3/\sqrt{m_2}^3$, is a measure of the symmetry of the curve. Ideally, $\alpha_3 = 0$.

In computing these quantities, use is made of the formula:

$$m_r = \frac{\sum_{i=1}^{k} \sum_{j=1}^{n_i} (x_{ij} - \bar{x}..)^r}{\sum_{i=1}^{k} n_i}$$

and is the mean of the $r^{th}$ moment of the x values about their mean.

In this and in other inter-laboratory programs conducted by the authors (1-3), it is evident that there were substantial variations between laboratories and that, therefore, the results were not completely independent of each other. Tables 8 and 9 show that most of the laboratories reported coefficients of variation lower than the over-all coefficient of variation. Figures 7 to 12 illustrate this point more clearly. In these figures, the average results by each laboratory for the first bottle were plotted against the average results for the second bottle (1-3, 13). The length of the arms of the crosses represent the estimated standard deviation of the results for the corresponding bottles. These figures show that the analytical results are strongly dependent on the laboratory from which they come. For this reason, the confidence limits, given under A in Table 10 for completeness, are unrealistic; their narrowness emphasizes this point. This type of analysis, however, gives a good estimate of the mean.

B. Analysis of Variance Technique

Having suspected the existence of inter-laboratory variations, the results were then treated as though they satisfied the following model (14):

$$x_{ij} = \mu + y_i + e_{ij}$$

where

$x_{ij}$ = the $j^{th}$ result reported by Laboratory i;

$\mu$ = the true value that will be estimated by the over-all mean $\bar{x}..$;

$y_i$ = the discrepancy between the mean of results from Laboratory i and the true value; and

$e_{ij}$ = the discrepancy of $x_{ij}$ from the mean of results from Laboratory i.

The assumption in this analysis is that both $y_i$ and $e_{ij}$ are normally distributed, with the means of zero and variances of $\omega^2$ and $\sigma^2$, respectively. The existence of $\omega^2$ can be detected

by comparing the ratio of "between-laboratory" mean squares to "within-laboratory" mean squares with the F statistic at the 95% confidence level and with the appropriate degree of freedom. The results of this analysis are presented in Table 10. The true value, $\mu$, in the above model can be estimated by the over-all mean, $\bar{x}..$, thus:

$$\bar{x}.. = \sum_{i=1}^{k} \sum_{j=1}^{n_i} x_{ij} \Big/ \sum_{i=1}^{k} n_i$$

with the variance of this over-all mean being given by:

$$V[\bar{x}..] = \frac{\sum_{i=1}^{k} n_i^2}{\left(\sum_{i=1}^{k} n_i\right)^2} \omega^2 + \frac{1}{\sum_{i=1}^{k} n_i} \sigma^2$$

where $n_i$ = the number of results reported by Laboratory i;

$k$ = the number of laboratories; and

$\omega$ and $\sigma$ can be estimated from "between-laboratory" and "within-laboratory" mean squares.

The 95% confidence intervals were then calculated according to the number of laboratories.

C. Weighted Mean to Give Minimum Variance

Further investigation of Tables 8 and 9 and Figures 7 to; 12 shows that there is a wide range in the degree of precision obtained by the various laboratories. For this reason, weighting the data by the weighting factor as a function of within-laboratory variance was tried. In this scheme, the results reported by each laboratory were considered as a set of independent variables with a mean of $\bar{x}_i.$ and a variance of $\sigma_i^2$. The weighted mean, $\bar{x}..$, was then computed from the following formula (14):

$$\bar{x}_{..} = \sum_{i=1}^{k} \frac{a_i}{n_i} \sum_{j=1}^{n_i} x_{ij}$$

in which

$$a_i = \frac{w_i}{\sum^{k} w_i}$$

and is the weighting factor for Laboratory i, and

$$w_i = \omega^2 + \frac{\alpha_i{}^2}{n_i}^{-1}$$

and is the reciprocal of the variance of $\bar{x}_{i.}$. This scheme will provide a mean value with a minimum variance of

$$V[\bar{x}_{..}] = \frac{1}{\sum_{i-1}^{k} w_i}$$

The results of these various schemes of computation are summarized in Table 10 under the corresponding notations, A, B, and C. The values of parameters under A were computed after rejecting all results that deviated from the over-all mean by more than twice the standard deviation. The parameters under B and C were computed after rejecting the entire results from laboratories whose laboratory means deviated from the over-all mean by more than twice the standard deviation.

TABLE 6

Results of Analysis of SU-1

| | Nickel (%) | | Copper (%) | | Cobalt (%) | |
|---|---|---|---|---|---|---|
| SU-1, LAB-1 | (a.a.) | (color.) | (a.a.) | | (a.a.) | (color.) |
| | 1.54 | 1.54 | 0.840 | | 0.060 | 0.060 |
| | 1.52 | 1.52 | 0.860 | | 0.061 | 0.059 |
| | 1.52 | 1.52 | 0.860 | | 0.059 | 0.059 |
| | 1.52 | 1.56 | 0.860 | | 0.060 | 0.061 |
| | 1.54 | | 0.860 | | 0.061 | |
| | 1.54 | | 0.860 | | 0.060 | |
| | 1.54 | | 0.860 | | 0.059 | |
| | 1.52 | | 0.860 | | 0.060 | |
| | 1.52 | | 0.860 | | 0.060 | |
| | 1.54 | | 0.860 | | 0.061 | |
| Sample Wt, g | 0.5 | - | 0.5 | | 0.5 | - |
| SU-1, LAB-2 | (a.a.) | (polar.) | (a.a.) | (polar.) | (a.a.) | |
| | 1.52 | 1.51 | 0.85 | 0.85 | 0.064 | |
| | 1.53 | 1.49 | 0.87 | 0.87 | 0.064 | |
| | 1.53 | 1.50 | 0.86 | 0.87 | 0.068 | |
| | 1.50 | 1.48 | 0.87 | 0.87 | 0.068 | |
| | 1.48 | 1.50 | 0.86 | 0.87 | 0.065 | |
| | 1.49 | 1.49 | 0.85 | 0.88 | 0.066 | |
| | 1.52 | 1.48 | 0.84 | 0.87 | 0.065 | |
| | 1.45 | 1.48 | 0.84 | 0.87 | 0.065 | |
| | 1.45 | 1.49 | 0.85 | 0.88 | 0.068 | |
| | 1.49 | 1.49 | 0.85 | 0.87 | 0.065 | |
| Sample Wt, g | 2 | 2 | 2 | 2 | 2 | |
| SU-1, LAB-3 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) | (color.) |
| | 1.44 | 1.39 | 0.849 | 0.848 | 0.051 | 0.051 |
| | 1.50 | 1.46 | 0.843 | 0.844 | 0.052 | 0.052 |
| | 1.48 | 1.46 | 0.850 | 0.839 | 0.054 | 0.050 |
| | 1.54 | 1.55 | 0.841 | 0.838 | 0.051 | 0.051 |
| | 1.53 | 1.57 | 0.848 | 0.851 | 0.052 | 0.051 |
| | 1.47 | 1.50 | 0.857 | 0.838 | 0.050 | 0.050 |
| | 1.46 | 1.49 | 0.851 | 0.847 | 0.050 | 0.051 |
| | 1.47 | 1.47 | 0.849 | 0.850 | 0.051 | 0.052 |
| | 1.49 | 1.54 | 0.853 | 0.844 | 0.047 | 0.049 |
| | 1.39 | 1.46 | 0.854 | 0.851 | 0.050 | 0.050 |
| | 1.44 | | 0.849 | | | |
| | 1.51 | | 0.848 | | | |
| Sample Wt, g | 0.5 | 1 | 0.5 | 1 | 0.5 | 1 |
| SU-1, LAB-4 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 1.53 | | 0.88 | | 0.066 | |
| | 1.55 | | 0.88 | | 0.066 | |
| | 1.55 | | 0.88 | | 0.068 | |
| | 1.53 | | 0.88 | | 0.070 | |
| | 1.53 | | 0.88 | | 0.070 | |
| | 1.53 | | 0.88 | | 0.068 | |
| | 1.54 | | 0.88 | | 0.068 | |
| | 1.53 | | 0.88 | | 0.068 | |
| | 1.53 | | 0.87 | | 0.067 | |
| | 1.54 | | 0.87 | | 0.068 | |
| Sample Wt, g | 1 | | 1 | | 1 | |
| SU-1, LAB-5 | | (vol.) | | (vol.) | | (color.) |
| | | 1.52 | | 0.86 | | 0.063 |
| | | 1.52 | | 0.87 | | 0.062 |
| | | 1.53 | | 0.88 | | 0.060 |
| | | 1.53 | | 0.88 | | 0.061 |
| | | 1.53 | | 0.86 | | 0.062 |
| | | 1.52 | | 0.86 | | 0.062 |
| | | 1.50 | | 0.86 | | 0.061 |
| | | 1.51 | | 0.88 | | 0.062 |
| | | 1.52 | | 0.88 | | 0.062 |
| | | 1.54 | | 0.87 | | 0.062 |
| Sample Wt, g | | - | | - | | - |

| | Nickel (%) | | Copper (%_ | | Cobalt (%) |
|---|---|---|---|---|---|
| SU-1, LAB-6 | (a.a.) | | (a.a.) | | (a.a.) |
| | 1.53 | | 0.885 | | 0.068 |
| | 1.52 | | 0.880 | | 0.068 |
| | 1.51 | | 0.882 | | 0.068 |
| | 1.51 | | 0.880 | | 0.068 |
| | 1.51 | | 0.887 | | 0.069 |
| | 1.50 | | 0.886 | | 0.069 |
| | 1.49 | | 0.883 | | 0.068 |
| | 1.50 | | 0.883 | | 0.069 |
| | 1.50 | | 0.883 | | 0.067 |
| | 1.50 | | 0.885 | | 0.069 |
| Sample Wt, g | - | | - | | - |
| SU-1, LAB-7 | (a.a.) | | (a.a.) | | (a.a.) |
| | 1.55 | | 0.896 | | 0.070 |
| | 1.55 | | 0.896 | | 0.070 |
| | 1.55 | | 0.896 | | 0.070 |
| | 1.55 | | 0.908 | | 0.070 |
| | 1.54 | | 0.896 | | 0.070 |
| | 1.55 | | 0.896 | | 0.068 |
| | 1.54 | | 0.896 | | 0.070 |
| | 1.54 | | 0.896 | | 0.070 |
| | 1.54 | | 0.888 | | 0.070 |
| | 1.55 | | 0.896 | | 0.068 |
| Sample Wt, g | 0.5 | | 0.5 | | 0.5 |
| SU-1, LAB-8 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) |
| | 1.48 | 1.49 | 0.88 | 0.86 | 0.069 |
| | 1.48 | 1.49 | 0.88 | 0.87 | 0.069 |
| | 1.49 | 1.49 | 0.88 | 0.86 | 0.069 |
| | 1.49 | 1.49 | 0.88 | 0.87 | 0.069 |
| | 1.49 | 1.47 | 0.87 | 0.86 | 0.069 |
| | 1.48 | 1.50 | 0.88 | 0.88 | 0.065 |
| | 1.49 | 1.50 | 0.88 | 0.89 | 0.066 |
| | 1.47 | 1.51 | 0.89 | 0.90 | 0.065 |
| | 1.48 | 1.50 | 0.88 | 0.88 | 0.065 |
| | 1.49 | 1.51 | 0.88 | 0.88 | 0.065 |
| Sample Wt, g | 2 | 2 | 2 | 2 | 2 |
| SU-1, LAB-9 | (a.a.) | (grav.) | (a.a.) | | (a.a.) |
| | 1.49 | 1.50 | 0.845 | | 0.064 |
| | 1.45 | 1.50 | 0.854 | | 0.066 |
| | 1.46 | 1.50 | 0.854 | | 0.066 |
| | 1.48 | 1.49 | 0.858 | | 0.067 |
| | 1.49 | 1.49 | 0.853 | | 0.065 |
| | 1.45 | 1.50 | 0.857 | | 0.065 |
| | 1.45 | 1.50 | 0.845 | | 0.065 |
| | 1.48 | 1.50 | 0.857 | | 0.066 |
| | 1.45 | 1.51 | 0.854 | | 0.066 |
| | 1.46 | 1.51 | 0.850 | | 0.066 |
| Sample Wt, g | 1 | 2 | 1 | | 1 |
| SU-1, LAB-10 | (a.a.) | | (a.a.) | | (a.a.) |
| | 1.49 | | 0.891 | | 0.0634 |
| | 1.51 | | 0.908 | | 0.0640 |
| | 1.52 | | 0.882 | | 0.0625 |
| | 1.52 | | 0.900 | | 0.0645 |
| | 1.52 | | 0.882 | | 0.0635 |
| | 1.51 | | 0.900 | | 0.0645 |
| | 1.50 | | 0.891 | | 0.0650 |
| | 1.52 | | 0.882 | | 0.0640 |
| | 1.50 | | 0.882 | | 0.0630 |
| | 1.52 | | 0.882 | | 0.0645 |
| | 1.49 | | 0.900 | | 0.0630 |
| | 1.52 | | 0.900 | | 0.0635 |
| Sample Wt, g | 1 | | 1 | | 1 |
| SU-1, LAB-11 | (a.a.) | | (a.a.) | | (a.a.) |
| | 1.47 | | 0.860 | | 0.065 |
| | 1.48 | | 0.850 | | 0.066 |
| | 1.48 | | 0.862 | | 0.066 |
| | 1.47 | | 0.860 | | 0.066 |
| | 1.47 | | 0.851 | | 0.065 |
| | 1.48 | | 0.850 | | 0.064 |
| | 1.47 | | 0.857 | | 0.064 |
| | 1.48 | | 0.851 | | 0.064 |
| | 1.47 | | 0.857 | | 0.064 |
| | 1.48 | | 0.880 | | 0.066 |
| Sample Wt, g | - | | - | | - |

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| | Nickel (%) | | Copper (%) | | Cobalt (%) | |
|---|---|---|---|---|---|---|
| SU-1, LAB-12 | (a.a.) | (vol.) | (a.a.) | (vol.) | (a.a.) | |
| | 1.38 | 1.50 | 0.84 | 0.87 | 0.059 | |
| | 1.41 | 1.46 | 0.84 | 0.87 | 0.060 | |
| | 1.41 | 1.50 | 0.84 | 0.86 | 0.059 | |
| | 1.39 | 1.50 | 0.84 | 0.86 | 0.057 | |
| | 1.41 | 1.48 | 0.84 | 0.87 | 0.060 | |
| | 1.38 | 1.48 | 0.83 | 0.87 | 0.057 | |
| | 1.45 | 1.49 | 0.82 | 0.87 | 0.061 | |
| | 1.45 | 1.49 | 0.83 | 0.87 | 0.061 | |
| | 1.37 | 1.50 | 0.83 | 0.86 | 0.058 | |
| | 1.44 | 1.50 | 0.84 | 0.86 | 0.060 | |
| Sample Wt, g | - | - | - | - | - | |
| SU-1, LAB-13 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 1.474 | | 0.794 | | 0.069 | |
| | 1.395 | | 0.799 | | 0.073 | |
| | 1.447 | | 0.799 | | 0.066 | |
| | 1.447 | | 0.836 | | 0.072 | |
| | 1.474 | | 0.775 | | 0.078 | |
| | 1.447 | | 0.819 | | 0.077 | |
| | 1.421 | | 0.824 | | 0.068 | |
| | 1.500 | | 0.811 | | 0.074 | |
| | 1.474 | | 0.794 | | 0.072 | |
| | 1.500 | | 0.840 | | 0.068 | |
| Sample Wt, g | 0.1 | | 0.1 | | 0.1 | |
| SU-1, LAB-14 | (a.a.) | (grav.) | (a.a.) | (electro.) | (a.a.) | (color.) |
| | 1.56 | 1.56 | 0.896 | 0.89 | 0.064 | 0.063 |
| | 1.56 | 1.56 | 0.892 | 0.90 | 0.060 | 0.063 |
| | 1.57 | 1.56 | 0.888 | 0.89 | 0.062 | 0.062 |
| | 1.55 | 1.58 | 0.888 | 0.885 | 0.062 | 0.061 |
| | 1.57 | 1.56 | 0.892 | 0.89 | 0.061 | 0.060 |
| | 1.57 | 1.58 | 0.892 | 0.89 | 0.061 | 0.061 |
| | 1.54 | 1.55 | 0.894 | 0.895 | 0.061 | 0.063 |
| | 1.57 | 1.56 | 0.890 | 0.885 | 0.064 | 0.060 |
| | 1.56 | 1.56 | 0.890 | 0.885 | 0.064 | 0.063 |
| | 1.56 | 1.57 | 0.894 | 0.89 | 0.060 | 0.064 |
| Sample Wt, g | 0.5 | 1 | 0.5 | 1 | 0.5 | 1 |
| SU-1, LAB-15 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) | (color.) |
| | 1.56 | 1.52 | 0.88 | 0.90 | 0.064 | 0.058 |
| | 1.58 | 1.51 | 0.90 | 0.90 | 0.060 | 0.059 |
| | 1.56 | 1.51 | 0.88 | 0.87 | 0.062 | 0.056 |
| | 1.54 | 1.50 | 0.89 | 0.87 | 0.060 | 0.060 |
| | 1.56 | 1.50 | 0.88 | 0.89 | 0.062 | 0.058 |
| | 1.56 | 1.49 | 0.88 | 0.90 | 0.064 | 0.059 |
| | 1.56 | 1.48 | 0.88 | 0.87 | 0.064 | 0.059 |
| | 1.54 | 1.52 | 0.89 | 0.87 | 0.062 | 0.060 |
| | 1.54 | 1.50 | 0.88 | 0.90 | 0.060 | 0.058 |
| | 1.58 | 1.49 | 0.89 | 0.89 | 0.062 | 0.058 |
| Sample Wt, g | 1 | 2 | 1 | 1 | 1 | 1 |
| SU-1, LAB-16 | | (grav.) | (a.a.) | | (a.a.) | |
| | | 1.53 | 0.833 | | 0.056 | |
| | | 1.53 | 0.835 | | 0.059 | |
| | | 1.53 | 0.835 | | 0.060 | |
| | | 1.54 | 0.838 | | 0.060 | |
| | | 1.55 | 0.843 | | 0.060 | |
| | | 1.52 | 0.848 | | 0.057 | |
| | | 1.52 | 0.853 | | 0.058 | |
| | | 1.53 | 0.858 | | 0.059 | |
| | | 1.54 | 0.859 | | 0.060 | |
| | | 1.54 | 0.860 | | 0.060 | |
| Sample Wt, g | | - | - | | - | |
| SU-1, LAB-17 | | (x.r.f.) | | (x.r.f.) | | |
| | | 1.446 | | 0.861 | | |
| | | 1.481 | | 0.874 | | |
| | | 1.443 | | 0.859 | | |
| | | 1.473 | | 0.865 | | |
| | | 1.474 | | 0.862 | | |
| | | 1.472 | | 0.868 | | |
| | | 1.450 | | 0.852 | | |
| | | 1.440 | | 0.854 | | |
| | | 1.462 | | 0.855 | | |
| | | 1.449 | | 0.853 | | |
| Sample Wt, g | | - | | - | | |

| | Nickel (%) | | Copper (%) | | Cobalt (%) | |
|---|---|---|---|---|---|---|
| SU-1, LAB-18 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 1.48 | | 0.86 | | 0.067 | |
| | 1.50 | | 0.83 | | 0.064 | |
| | 1.49 | | 0.86 | | 0.069 | |
| | 1.48 | | 0.86 | | 0.069 | |
| | 1.50 | | 0.87 | | 0.065 | |
| | 1.55 | | 0.86 | | 0.067 | |
| | 1.46 | | 0.83 | | 0.064 | |
| | 1.48 | | 0.83 | | 0.067 | |
| | 1.46 | | 0.84 | | 0.068 | |
| | 1.52 | | 0.85 | | 0.068 | |
| Sample Wt, g | - | | - | | - | |
| SU-1, LAB-19 | (a.a.) | (grav.) | (a.a.) | (polar.) | | (color.) |
| | 1.51 | 1.52 | 0.845 | 0.854 | | 0.067 |
| | 1.50 | 1.51 | 0.845 | 0.853 | | 0.067 |
| | 1.51 | 1.52 | 0.850 | | | 0.065 |
| | 1.50 | 1.52 | 0.850 | | | 0.068 |
| | 1.51 | | 0.841 | | | 0.067 |
| | 1.51 | | 0.844 | | | 0.066 |
| | | | 0.850 | | | 0.066 |
| | | | 0.850 | | | 0.067 |
| | | | | | | 0.068 |
| | | | | | | 0.067 |
| Sample Wt, g | 1 | 1 | 1 | 1 | | 2 |
| SU-1, LAB-20 | (a.a.) | (grav.) | (a.a.) | (color.) | (a.a.) | |
| | 1.53 | 1.50 | 0.843 | 0.855 | 0.063 | |
| | 1.54 | 1.50 | 0.850 | 0.848 | 0.063 | |
| | 1.55 | 1.52 | 0.850 | 0.861 | 0.064 | |
| | 1.55 | 1.49 | 0.843 | 0.845 | 0.062 | |
| | 1.52 | 1.50 | 0.837 | 0.853 | 0.064 | |
| | 1.56 | 1.48 | 0.842 | | 0.063 | |
| | 1.54 | 1.50 | 0.835 | | 0.064 | |
| | 1.55 | 1.48 | 0.846 | | 0.063 | |
| | 1.54 | 1.49 | 0.847 | | 0.064 | |
| | 1.56 | 1.50 | 0.841 | | 0.063 | |
| Sample Wt, g | - | 0.25 | 2 | - | - | |
| SU-1, LAB-21 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 1.48 | | 0.86 | | 0.065 | |
| | 1.52 | | 0.86 | | 0.066 | |
| | 1.52 | | 0.85 | | 0.066 | |
| | 1.53 | | 0.87 | | 0.067 | |
| | 1.51 | | 0.87 | | 0.066 | |
| | 1.52 | | 0.86 | | 0.066 | |
| | 1.51 | | 0.86 | | 0.067 | |
| | 1.52 | | 0.86 | | 0.066 | |
| | 1.54 | | 0.87 | | 0.065 | |
| | 1.53 | | 0.89 | | 0.065 | |
| Sample Wt, g | 1 | | 1 | | 1 | |
| SU-1, LAB-22 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) | |
| | 1.54 | 1.546 | 0.864 | 0.861 | 0.054 | |
| | 1.53 | 1.524 | 0.884 | 0.851 | 0.052 | |
| | 1.53 | 1.501 | 0.878 | 0.854 | 0.052 | |
| | 1.53 | 1.544 | 0.876 | 0.854 | 0.050 | |
| | 1.51 | 1.528 | 0.876 | 0.856 | 0.054 | |
| | 1.53 | 1.512 | 0.858 | 0.871 | 0.050 | |
| | 1.53 | 1.500 | 0.856 | 0.861 | 0.054 | |
| | 1.54 | 1.491 | 0.860 | 0.854 | 0.054 | |
| | 1.52 | 1.512 | 0.856 | 0.859 | 0.050 | |
| | 1.52 | 1.524 | 0.862 | 0.859 | 0.050 | |
| Sample Wt, g | 0.5 | 0.5 | 0.5 | 2 | 0.5 | |

| | Nickel (%) | | Copper (%) | | Cobalt (%) |
|---|---|---|---|---|---|
| SU-1, LAB-23 | (a.a.) | (grav.) | (a.a.) | (color.) | (a.a.) |
| | 1.514 | 1.525 | 0.885 | 0.880 | 0.063 |
| | 1.520 | 1.485 | 0.847 | 0.875 | 0.064 |
| | 1.526 | 1.517 | 0.878 | 0.875 | 0.064 |
| | 1.526 | 1.547 | 0.867 | 0.878 | 0.066 |
| | 1.536 | 1.517 | 0.870 | 0.887 | 0.066 |
| | 1.514 | 1.517 | 0.867 | 0.876 | 0.063 |
| | 1.512 | 1.518 | 0.860 | 0.882 | 0.063 |
| | 1.520 | 1.513 | 0.856 | 0.880 | 0.066 |
| | 1.520 | 1.509 | 0.863 | 0.857 | 0.065 |
| | 1.514 | | 0.850 | 0.864 | 0.064 |
| Sample Wt, g | - | - | - | - | - |
| SU-1, LAB-24 | (a.a.) | | (a.a.) | | (a.a.) |
| | 1.42 | | 0.81 | | 0.055 |
| | 1.41 | | 0.79 | | 0.056 |
| | 1.39 | | 0.79 | | 0.058 |
| | 1.41 | | 0.80 | | 0.056 |
| | 1.40 | | 0.81 | | 0.057 |
| | 1.39 | | 0.78 | | 0.055 |
| | 1.40 | | 0.80 | | 0.056 |
| | 1.42 | | 0.79 | | 0.054 |
| | 1.39 | | 0.81 | | 0.057 |
| | 1.43 | | 0.81 | | 0.055 |
| Sample Wt, g | - | | - | | - |
| SU-1, LAB-25 | | (spect.) | | (spect.) | (spect.) |
| | | 1.53 | | 0.90 | 0.0513 |
| | | 1.60 | | 0.91 | 0.0638 |
| | | 1.55 | | 0.91 | 0.0640 |
| | | 1.49 | | 0.85 | 0.0606 |
| | | 1.45 | | 0.92 | 0.0606 |
| | | 1.53 | | 0.92 | 0.0660 |
| | | 1.65 | | 0.96 | 0.0606 |
| | | 1.56 | | 0.91 | 0.0580 |
| | | 1.49 | | 0.93 | 0.0587 |
| | | 1.62 | | 0.91 | 0.0523 |
| Sample Wt, g | | 0.04 | | 0.04 | 0.04 |

TABLE 6

Results of Analysis of UM-1

| | Nickel (%) | | Copper (%) | | Cobalt (%) | |
|---|---|---|---|---|---|---|
| UM-1, LAB-1 | (a.a.) | (color.) | (a.a.) | | (a.a.) | (color.) |
| | 0.92 | 0.92 | 0.425 | | 0.031 | 0.032 |
| | 0.92 | 0.92 | 0.425 | | 0.031 | 0.030 |
| | 0.93 | 0.91 | 0.425 | | 0.031 | 0.031 |
| | 0.92 | 0.91 | 0.430 | | 0.031 | 0.029 |
| | 0.93 | | 0.430 | | 0.030 | |
| | 0.93 | | 0.430 | | 0.031 | |
| | 0.93 | | 0.430 | | 0.031 | |
| | 0.92 | | 0.435 | | 0.031 | |
| | 0.92 | | 0.425 | | 0.031 | |
| | 0.92 | | 0.435 | | 0.031 | |
| Sample Wt, g | 0.5 | - | 0.5 | | 0.5 | - |
| UM-1, LAB-2 | (a.a.) | (polar.) | (a.a.) | (polar.) | (a.a.) | |
| | 0.89 | 0.86 | 0.45 | 0.44 | 0.033 | |
| | 0.89 | 0.87 | 0.45 | 0.44 | 0.033 | |
| | 0.89 | 0.86 | 0.43 | 0.44 | 0.034 | |
| | 0.89 | 0.86 | 0.43 | 0.44 | 0.033 | |
| | 0.89 | 0.86 | 0.45 | 0.44 | 0.033 | |
| | 0.88 | 0.87 | 0.44 | 0.45 | 0.033 | |
| | 0.88 | 0.87 | 0.44 | 0.45 | 0.033 | |
| | 0.88 | 0.87 | 0.45 | 0.45 | 0.033 | |
| | 0.89 | 0.87 | 0.45 | 0.45 | 0.033 | |
| | 0.88 | 0.87 | 0.45 | 0.45 | 0.033 | |
| Sample Wt, g | 2 | 2 | 2 | 2 | 2 | |
| UM-1, LAB-3 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) | (color.) |
| | 0.886 | 0.879 | 0.441 | 0.447 | 0.036 | 0.037 |
| | 0.877 | 0.882 | 0.442 | 0.440 | 0.036 | 0.036 |
| | 0.885 | 0.880 | 0.442 | 0.443 | 0.036 | 0.038 |
| | 0.887 | 0.883 | 0.447 | 0.450 | 0.034 | 0.036 |
| | 0.881 | 0.888 | 0.439 | 0.442 | 0.034 | 0.035 |
| | | | | | 0.036 | 0.037 |
| | | | | | 0.036 | 0.037 |
| | | | | | 0.038 | 0.036 |
| | | | | | 0.037 | 0.035 |
| | | | | | 0.037 | 0.037 |
| | | | | | 0.036 | 0.037 |
| | | | | | 0.036 | 0.036 |
| | | | | | 0.037 | 0.036 |
| | | | | | 0.038 | 0.035 |
| | | | | | 0.037 | 0.037 |
| Sample Wt, g | 1 | 1 | 1 | 1 | 0.5-1 | 1 |
| UM-1, LAB-4 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 0.89 | | 0.43 | | 0.037 | |
| | 0.89 | | 0.44 | | 0.037 | |
| | 0.89 | | 0.43 | | 0.037 | |
| | 0.90 | | 0.43 | | 0.037 | |
| | 0.89 | | 0.43 | | 0.037 | |
| | 0.90 | | 0.43 | | 0.037 | |
| | 0.90 | | 0.43 | | 0.037 | |
| | 0.90 | | 0.43 | | 0.037 | |
| | 0.90 | | 0.43 | | 0.037 | |
| | 0.90 | | 0.43 | | 0.037 | |
| Sample Wt, g | 1 | | 1 | | 1 | |

| | Nickel (%) | | Copper (%) | | Cobalt (%) |
|---|---|---|---|---|---|
| UM-1, LAB-5 | | (vol.) | | (vol.) | (color.) |
| | | 0.91 | | 0.44 | 0.033 |
| | | 0.91 | | 0.42 | 0.031 |
| | | 0.92 | | 0.43 | 0.032 |
| | | 0.93 | | 0.44 | 0.033 |
| | | 0.89 | | 0.44 | 0.032 |
| | | 0.88 | | 0.44 | 0.033 |
| | | 0.88 | | 0.44 | 0.032 |
| | | 0.91 | | 0.44 | 0.031 |
| | | 0.90 | | 0.44 | 0.032 |
| | | 0.93 | | 0.42 | 0.032 |
| Sample Wt, g | | - | | - | - |
| UM-1, LAB-6 | (a.a.) | | (a.a.) | | (a.a.) |
| | 0.908 | | 0.435 | | 0.038 |
| | 0.901 | | 0.436 | | 0.038 |
| | 0.904 | | 0.436 | | 0.038 |
| | 0.899 | | 0.436 | | 0.038 |
| | 0.906 | | 0.435 | | 0.038 |
| | 0.899 | | 0.446 | | 0.038 |
| | 0.906 | | 0.442 | | 0.038 |
| | 0.903 | | 0.442 | | 0.038 |
| | 0.909 | | 0.444 | | 0.038 |
| | 0.900 | | 0.440 | | 0.038 |
| Sample Wt, g | - | | - | | - |
| UM-1, LAB-7 | (.a.a.) | | (a.a.) | | (a.a.) |
| | 0.860 | | 0.435 | | 0.036 |
| | 0.860 | | 0.432 | | 0.036 |
| | 0.870 | | 0.435 | | 0.036 |
| | 0.860 | | 0.437 | | 0.036 |
| | 0.860 | | 0.435 | | 0.036 |
| | 0.860 | | 0.435 | | 0.036 |
| | 0.860 | | 0.435 | | 0.036 |
| | 0.860 | | 0.432 | | 0.036 |
| | 0.860 | | 0.435 | | 0.036 |
| | 0.860 | | 0.437 | | 0.036 |
| Sample Wt, g | 0.5 | | 0.5 | | 0.5 |
| UM-1, LAB-8 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) |
| | 0.85 | 0.89 | 0.44 | 0.44 | 0.034 |
| | 0.85 | 0.89 | 0.44 | 0.44 | 0.034 |
| | 0.86 | 0.89 | 0.44 | 0.44 | 0.033 |
| | 0.86 | 0.88 | 0.44 | 0.44 | 0.034 |
| | 0.86 | 0.88 | 0.44 | 0.44 | 0.034 |
| | 0.89 | 0.87 | 0.44 | 0.44 | 0.036 |
| | 0.89 | 0.87 | 0.44 | 0.44 | 0.036 |
| | 0.89 | 0.87 | 0.44 | 0.44 | 0.036 |
| | 0.89 | 0.88 | 0.44 | 0.44 | 0.037 |
| | 0.89 | 0.87 | 0.44 | 0.44 | 0.036 |
| Sample Wt, g | 2 | 2 | 2 | 2 | 2 |
| UM-1, LAB-9 | (a.a.) | (grav.) | (a.a.) | | (a.a.) |
| | 0.852 | 0.870 | 0.435 | | 0.037 |
| | 0.852 | 0.870 | 0.435 | | 0.037 |
| | 0.852 | 0.870 | 0.432 | | 0.037 |
| | 0.852 | 0.864 | 0.435 | | 0.037 |
| | 0.856 | 0.851 | 0.428 | | 0.037 |
| | 0.848 | 0.870 | 0.435 | | 0.036 |
| | 0.848 | 0.864 | 0.426 | | 0.037 |
| | 0.848 | 0.872 | 0.430 | | 0.037 |
| | 0.848 | 0.872 | 0.435 | | 0.037 |
| | 0.848 | 0.864 | 0.436 | | 0.036 |
| Sample Wt, g | - | - | - | | - |

| | Nickel (%) | | Copper (%) | | Cobalt (%) | |
|---|---|---|---|---|---|---|
| UM-1, LAB-10 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 0.860 | | 0.440 | | 0.0360 | |
| | 0.865 | | 0.450 | | 0.0365 | |
| | 0.860 | | 0.445 | | 0.0360 | |
| | 0.870 | | 0.450 | | 0.0360 | |
| | 0.860 | | 0.445 | | 0.0355 | |
| | 0.855 | | 0.450 | | 0.0355 | |
| | 0.860 | | 0.450 | | 0.0360 | |
| | 0.865 | | 0.445 | | 0.0365 | |
| | 0.860 | | 0.450 | | 0.0360 | |
| | 0.865 | | 0.450 | | 0.0365 | |
| | 0.850 | | 0.455 | | 0.0355 | |
| | 0.860 | | 0.445 | | 0.0355 | |
| Sample Wt, g | 1 | | 1 | | 1 | |
| UM-1, Lab-11 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 0.878 | | 0.420 | | 0.035 | |
| | 0.878 | | 0.418 | | 0.034 | |
| | 0.881 | | 0.418 | | 0.034 | |
| | 0.878 | | 0.418 | | 0.034 | |
| | 0.871 | | 0.421 | | 0.034 | |
| | 0.878 | | 0.418 | | 0.034 | |
| | 0.878 | | 0.424 | | 0.034 | |
| | 0.881 | | 0.421 | | 0.034 | |
| | 0.871 | | 0.414 | | 0.034 | |
| | 0.871 | | 0.424 | | 0.034 | |
| Sample Wt, g | - | | - | | - | |
| UM-1, LAB-12 | (a.a.) | (vol.) | (a.a.) | (vol.) | (a.a.) | |
| | 0.80 | 0.86 | 0.40 | 0.43 | 0.031 | |
| | 0.79 | 0.85 | 0.40 | 0.42 | 0.031 | |
| | 0.79 | 0.86 | 0.40 | 0.41 | 0.033 | |
| | 0.79 | 0.86 | 0.40 | 0.43 | 0.033 | |
| | 0.79 | 0.85 | 0.40 | 0.42 | 0.034 | |
| | 0.80 | 0.87 | 0.41 | 0.43 | 0.031 | |
| | 0.80 | 0.88 | 0.40 | 0.43 | 0.031 | |
| | 0.79 | 0.88 | 0.41 | 0.42 | 0.031 | |
| | 0.79 | 0.88 | 0.41 | 0.41 | 0.032 | |
| | 0.78 | 0.87 | 0.41 | 0.43 | 0.032 | |
| Sample Wt, g | - | - | - | - | - | |
| UM-1, LAB-13 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 0.833 | | 0.420 | | 0.035 | |
| | 0.833 | | 0.420 | | 0.034 | |
| | 0.833 | | 0.413 | | 0.036 | |
| | 0.857 | | 0.413 | | 0.036 | |
| | 0.857 | | 0.413 | | 0.033 | |
| | 0.833 | | 0.408 | | 0.037 | |
| | 0.848 | | 0.413 | | 0.037 | |
| | 0.833 | | 0.408 | | 0.036 | |
| | 0.857 | | 0.413 | | 0.037 | |
| | 0.881 | | 0.417 | | 0.038 | |
| Sample Wt, g | 0.1 | | 0.1 | | 0.1 | |
| UM-1, LAB-14 | (a.a.) | (grav.) | (a.a.) | (electro.) | (a.a.) | (color.) |
| | 0.892 | 0.896 | 0.436 | 0.43 | 0.033 | 0.034 |
| | 0.902 | 0.898 | 0.436 | 0.44 | 0.036 | 0.034 |
| | 0.898 | 0.904 | 0.438 | 0.44 | 0.034 | 0.034 |
| | 0.896 | 0.904 | 0.440 | 0.435 | 0.034 | 0.034 |
| | 0.902 | 0.900 | 0.438 | 0.44 | 0.032 | 0.034 |
| | 0.902 | 0.900 | 0.442 | 0.44 | 0.035 | 0.033 |
| | 0.896 | 0.904 | 0.440 | 0.43 | 0.035 | 0.033 |
| | 0.898 | 0.898 | 0.438 | 0.43 | 0.032 | 0.034 |
| | 0.898 | 0.896 | 0.438 | 0.435 | 0.032 | 0.035 |
| | 0.902 | 0.900 | 0.438 | 0.435 | 0.033 | 0.035 |
| Sample Wt, g | 1 | 1 | 1 | 1 | 1 | 1 |

| | Nickel (%) | | Copper (%) | | Cobalt (%) | |
|---|---|---|---|---|---|---|
| UM-1, LAB-15 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) | (color.) |
| | 0.88 | 0.88 | 0.42 | 0.45 | 0.036 | 0.032 |
| | 0.88 | 0.90 | 0.43 | 0.45 | 0.034 | 0.032 |
| | 0.88 | 0.90 | 0.42 | 0.46 | 0.034 | 0.030 |
| | 0.86 | 0.88 | 0.44 | 0.44 | 0.032 | 0.030 |
| | 0.86 | 0.87 | 0.44 | 0.45 | 0.034 | 0.030 |
| | 0.86 | 0.90 | 0.44 | 0.46 | 0.036 | 0.028 |
| | 0.86 | 0.90 | 0.42 | 0.45 | 0.036 | 0.030 |
| | 0.88 | 0.87 | 0.43 | 0.44 | 0.034 | 0.032 |
| | 0.86 | 0.88 | 0.44 | 0.46 | 0.034 | 0.030 |
| | 0.86 | 0.88 | 0.44 | 0.45 | 0.034 | 0.030 |
| Sample Wt, g | 1 | 2 | 1 | 1 | 1 | 1 |
| UM-1, LAB-16 | | (grav.) | (a.a.) | | (a.a.) | |
| | | 0.90 | 0.409 | | 0.034 | |
| | | 0.91 | 0.411 | | 0.034 | |
| | | 0.91 | 0.412 | | 0.034 | |
| | | 0.91 | 0.413 | | 0.035 | |
| | | 0.91 | 0.413 | | 0.035 | |
| | | 0.90 | 0.405 | | 0.035 | |
| | | 0.90 | 0.408 | | 0.035 | |
| | | 0.90 | 0.408 | | 0.035 | |
| | | 0.91 | 0.411 | | 0.035 | |
| | | 0.91 | 0.415 | | 0.036 | |
| Sample Wt, g | - | - | - | | - | |
| UM-1, LAB-17 | | (x.r.f.) | | (x.r.f.) | | |
| | | 0.923 | | 0.476 | | |
| | | 0.963 | | 0.491 | | |
| | | 0.938 | | 0.487 | | |
| | | 0.952 | | 0.491 | | |
| | | 0.943 | | 0.482 | | |
| | | 0.941 | | 0.475 | | |
| | | 0.963 | | 0.486 | | |
| | | 0.939 | | 0.477 | | |
| | | 0.967 | | 0.484 | | |
| | | 0.950 | | 0.482 | | |
| Sample Wt,g | | - | | - | | |
| UM-1, LAB-18 | (a.a.) | | (a.a.) | | (a.a.) | |
| | 0.86 | | 0.42 | | 0.034 | |
| | 0.86 | | 0.43 | | 0.034 | |
| | 0.88 | | 0.42 | | 0.035 | |
| | 0.85 | | 0.43 | | 0.035 | |
| | 0.85 | | 0.43 | | 0.037 | |
| | 0.86 | | 0.42 | | 0.034 | |
| | 0.87 | | 0.41 | | 0.034 | |
| | 0.89 | | 0.42 | | 0.035 | |
| | 0.84 | | 0.43 | | 0.037 | |
| | 0.78 | | 0.43 | | 0.037 | |
| Sample Wt, g | - | | - | | - | |
| UM-1, LAB-19 | (a.a.) | (grav.) | (a.a.) | (polar.) | | (color.) |
| | 0.882 | 0.890 | 0.424 | 0.428 | | 0.037 |
| | 0.878 | 0.885 | 0.424 | 0.428 | | 0.038 |
| | 0.885 | 0.890 | 0.428 | | | 0.035 |
| | 0.882 | 0.885 | 0.429 | | | 0.038 |
| | 0.878 | | 0.424 | | | 0.037 |
| | 0.885 | | 0.424 | | | 0.037 |
| | | | 0.428 | | | 0.035 |
| | | | 0.429 | | | 0.038 |
| | | | | | | 0.038 |
| | | | | | | 0.037 |
| Sample Wt, g | 1 | 1 | 1 | 1 | | 2 |

| | Nickel (%) | | Copper (%) | | Cobalt (%) |
|---|---|---|---|---|---|
| UM-1, LAB-20 | (a.a.) | | (a.a.) | (color.) | (a.a.) |
| | 0.895 | | 0.424 | 0.429 | 0.033 |
| | 0.900 | | 0.424 | 0.426 | 0.033 |
| | 0.898 | | 0.421 | 0.424 | 0.033 |
| | 0.900 | | 0.421 | 0.423 | 0.033 |
| | 0.903 | | 0.421 | 0.428 | 0.033 |
| | 0.888 | | 0.429 | | 0.033 |
| | 0.896 | | 0.432 | | 0.033 |
| | 0.894 | | 0.429 | | 0.033 |
| | 0.898 | | 0.431 | | 0.032 |
| | 0.896 | | 0.431 | | 0.033 |
| Sample Wt, g | - | | 2 | - | - |
| UM-1, LAB-21 | (a.a.) | | (a.a.) | | (a.a.) |
| | 0.88 | | 0.43 | | 0.035 |
| | 0.87 | | 0.43 | | 0.035 |
| | 0.88 | | 0.44 | | 0.035 |
| | 0.88 | | 0.44 | | 0.036 |
| | 0.90 | | 0.43 | | 0.036 |
| | 0.90 | | 0.44 | | 0.036 |
| | 0.88 | | 0.44 | | 0.034 |
| | 0.89 | | 0.45 | | 0.033 |
| | 0.90 | | 0.44 | | 0.035 |
| | 0.90 | | 0.43 | | 0.036 |
| Sample Wt, g | 1 | | 1 | | 1 |
| UM-1, LAB-22 | (a.a.) | (grav.) | (a.a.) | (vol.) | (a.a.) |
| | 0.872 | 0.862 | 0.444 | 0.443 | 0.028 |
| | 0.869 | 0.892 | 0.440 | 0.431 | 0.028 |
| | 0.869 | 0.870 | 0.440 | 0.441 | 0.028 |
| | 0.870 | 0.862 | 0.440 | 0.435 | 0.028 |
| | 0.872 | 0.870 | 0.446 | 0.443 | 0.028 |
| | 0.867 | 0.882 | 0.436 | 0.433 | 0.028 |
| | 0.869 | 0.862 | 0.444 | 0.428 | 0.028 |
| | 0.864 | 0.858 | 0.444 | 0.426 | 0.028 |
| | 0.869 | 0.878 | 0.442 | 0.423 | 0.028 |
| | 0.870 | 0.878 | 0.440 | 0.428 | 0.028 |
| Sample Wt, g | 0.5 | 0.5 | 0.5 | 2 | 0.5 |
| UM-1, LAB-23 | (a.a.) | (grav.) | (a.a.) | (color.) | (a.a.) |
| | 0.871 | 0.895 | 0.427 | 0.436 | 0.035 |
| | 0.872 | 0.892 | 0.427 | 0.440 | 0.034 |
| | 0.873 | 0.889 | 0.427 | 0.437 | 0.034 |
| | 0.868 | 0.892 | 0.427 | 0.433 | 0.034 |
| | 0.870 | 0.889 | 0.429 | 0.439 | 0.034 |
| | 0.875 | 0.886 | 0.427 | 0.438 | 0.034 |
| | 0.874 | 0.883 | 0.427 | 0.433 | 0.034 |
| | 0.876 | 0.897 | 0.427 | 0.424 | 0.034 |
| | 0.871 | 0.890 | 0.427 | 0.420 | 0.034 |
| | 0.871 | 0.889 | 0.429 | 0.436 | 0.035 |
| Sample Wt, g | - | - | - | - | - |
| UM-1, LAB-24 | (a.a.) | | (a.a.) | | (a.a.) |
| | 0.88 | | 0.40 | | 0.032 |
| | 0.90 | | 0.42 | | 0.031 |
| | 0.92 | | 0.42 | | 0.032 |
| | 0.89 | | 0.40 | | 0.032 |
| | 0.90 | | 0.41 | | 0.032 |
| | 0.89 | | 0.41 | | 0.032 |
| | 0.88 | | 0.42 | | 0.033 |
| | 0.92 | | 0.42 | | 0.031 |
| | 0.92 | | 0.41 | | 0.032 |
| | 0.90 | | 0.41 | | 0.032 |
| Sample Wt, g | - | | - | | - |

| | Nickel (%) | Copper (%) | Cobalt (%) |
|---|---|---|---|
| UM-1, LAB-25 | (spect.) | (spect.) | (spect.) |
| | 0.865 | 0.510 | 0.0330 |
| | 0.870 | 0.540 | 0.0330 |
| | 0.810 | 0.432 | 0.0335 |
| | 0.810 | 0.400 | 0.0350 |
| | 0.875 | 0.595 | 0.0330 |
| | 0.878 | 0.454 | 0.0345 |
| | 0.788 | 0.452 | 0.0328 |
| | 0.800 | 0.460 | 0.0340 |
| | 0.885 | 0.525 | 0.0350 |
| | 0.875 | 0.520 | 0.0328 |
| Sample Wt, g | 0.04 | 0.04 | 0.04 |

SU-1, NICKEL

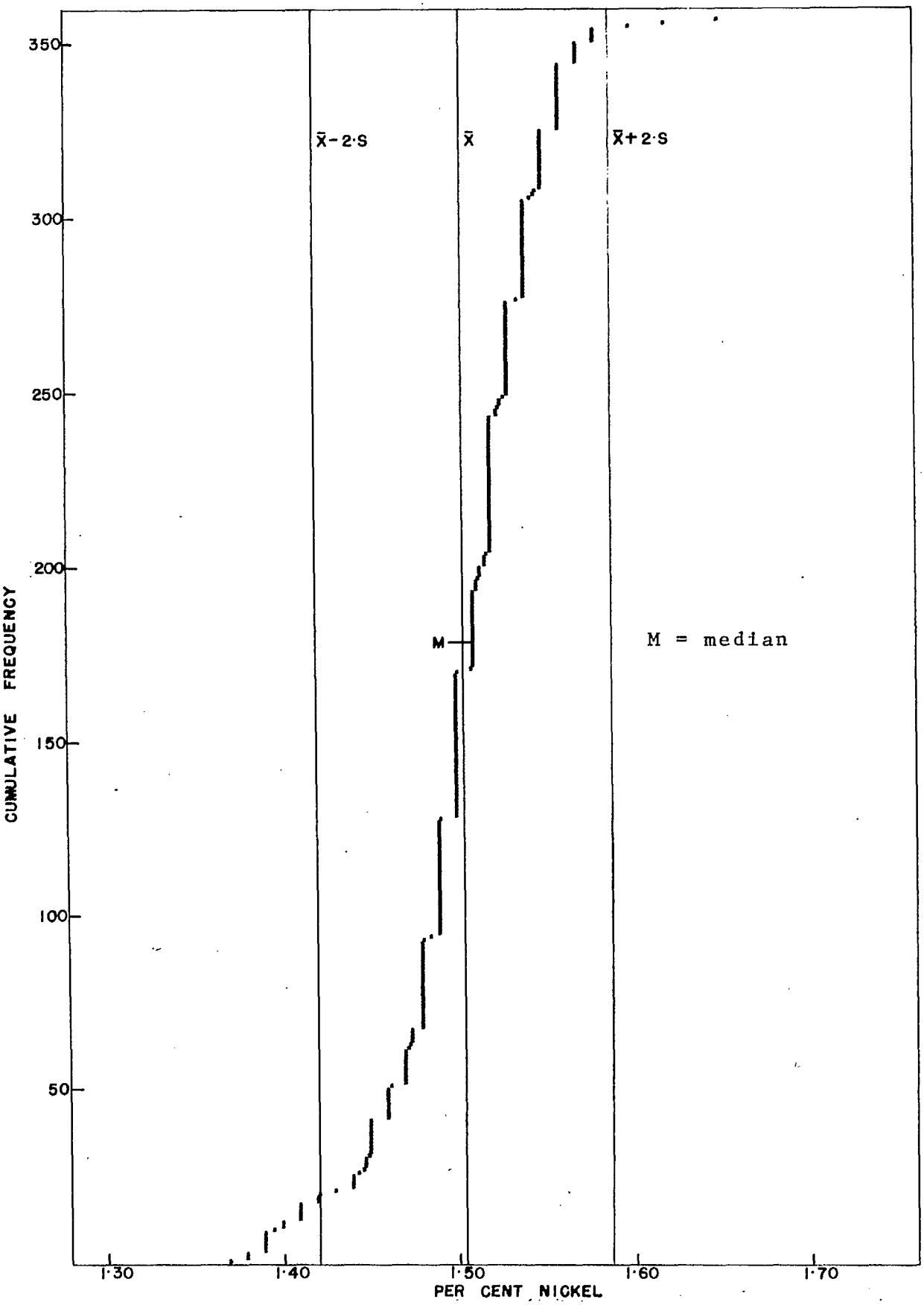


Figure 1. Cumulative Distribution for Nickel Results. /28

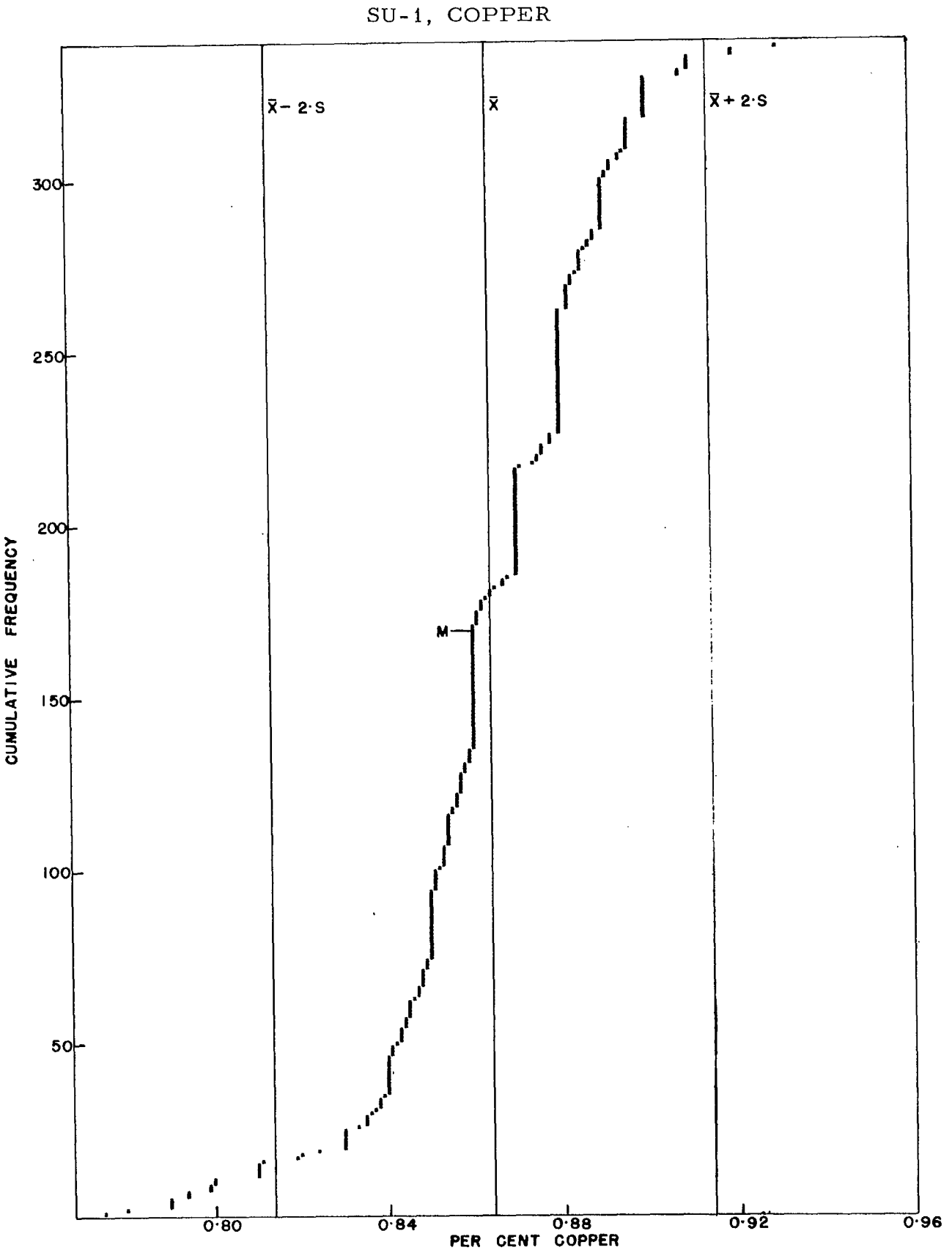


Figure 2. Cumulative Distribution for Copper Results.

SU-1, COBALT

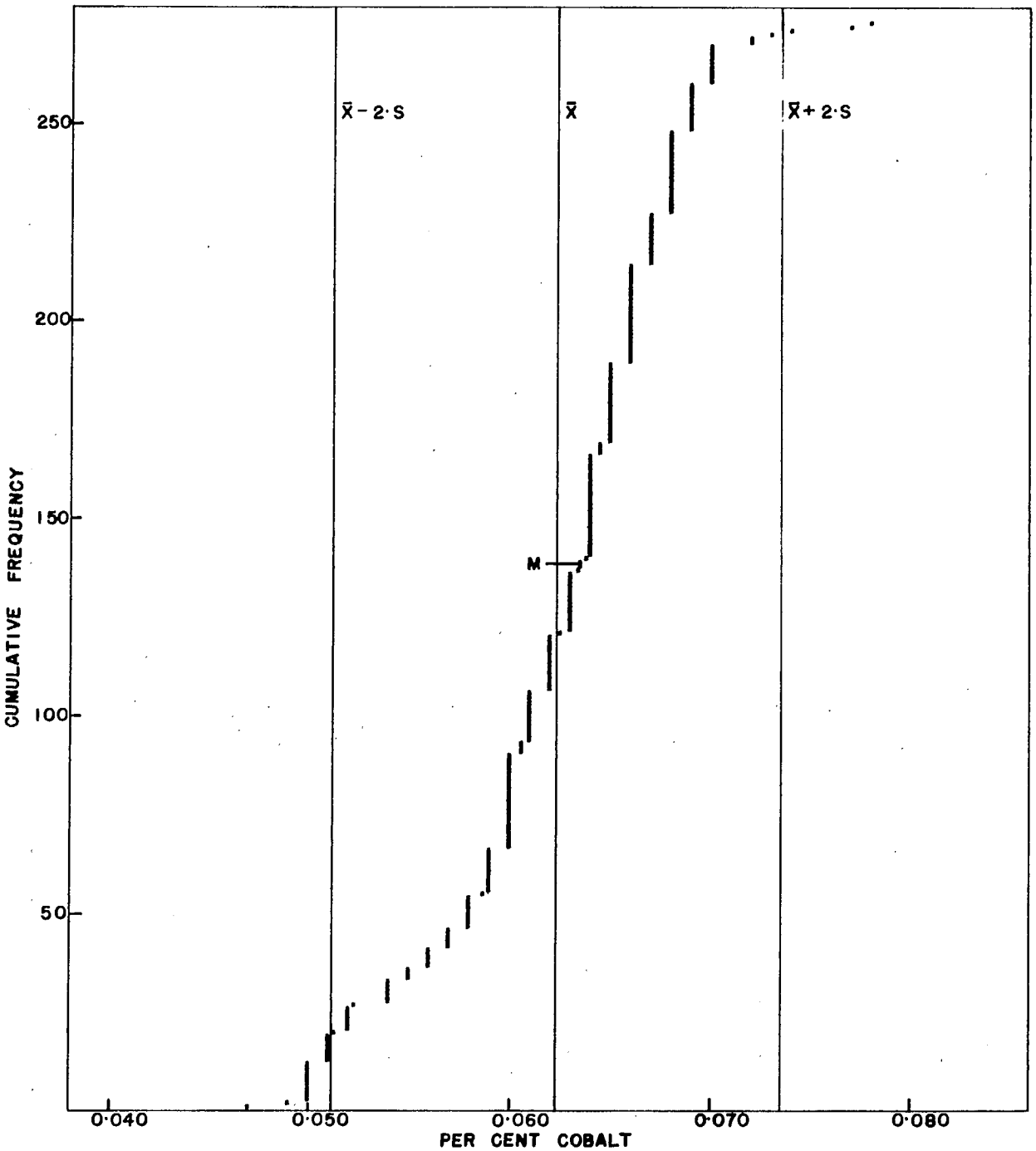


Figure 3. Cumulative Distribution for Cobalt Results.

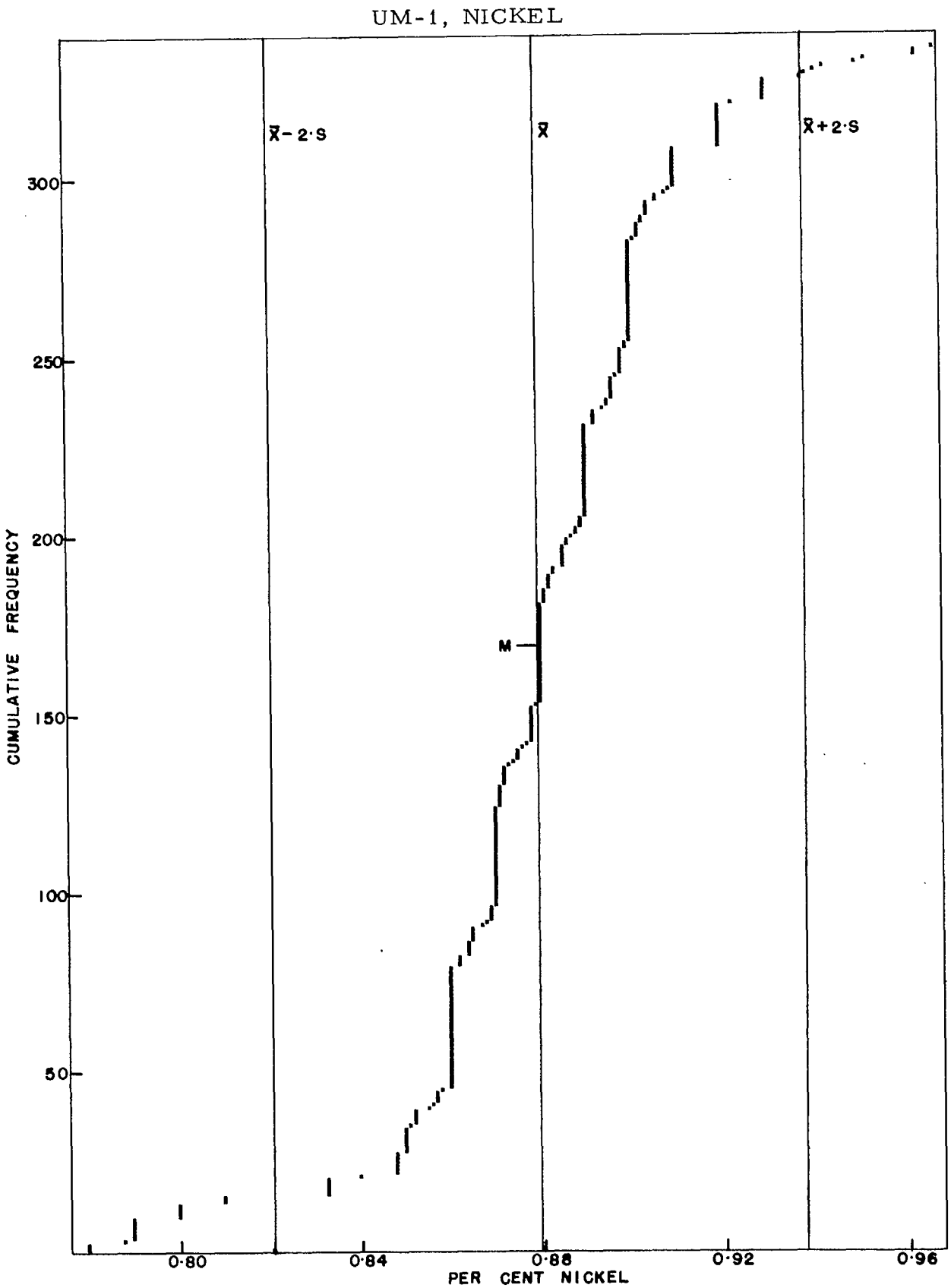


Figure 4. Cumulative Distribution for Nickel Results.

UM-1, COPPER

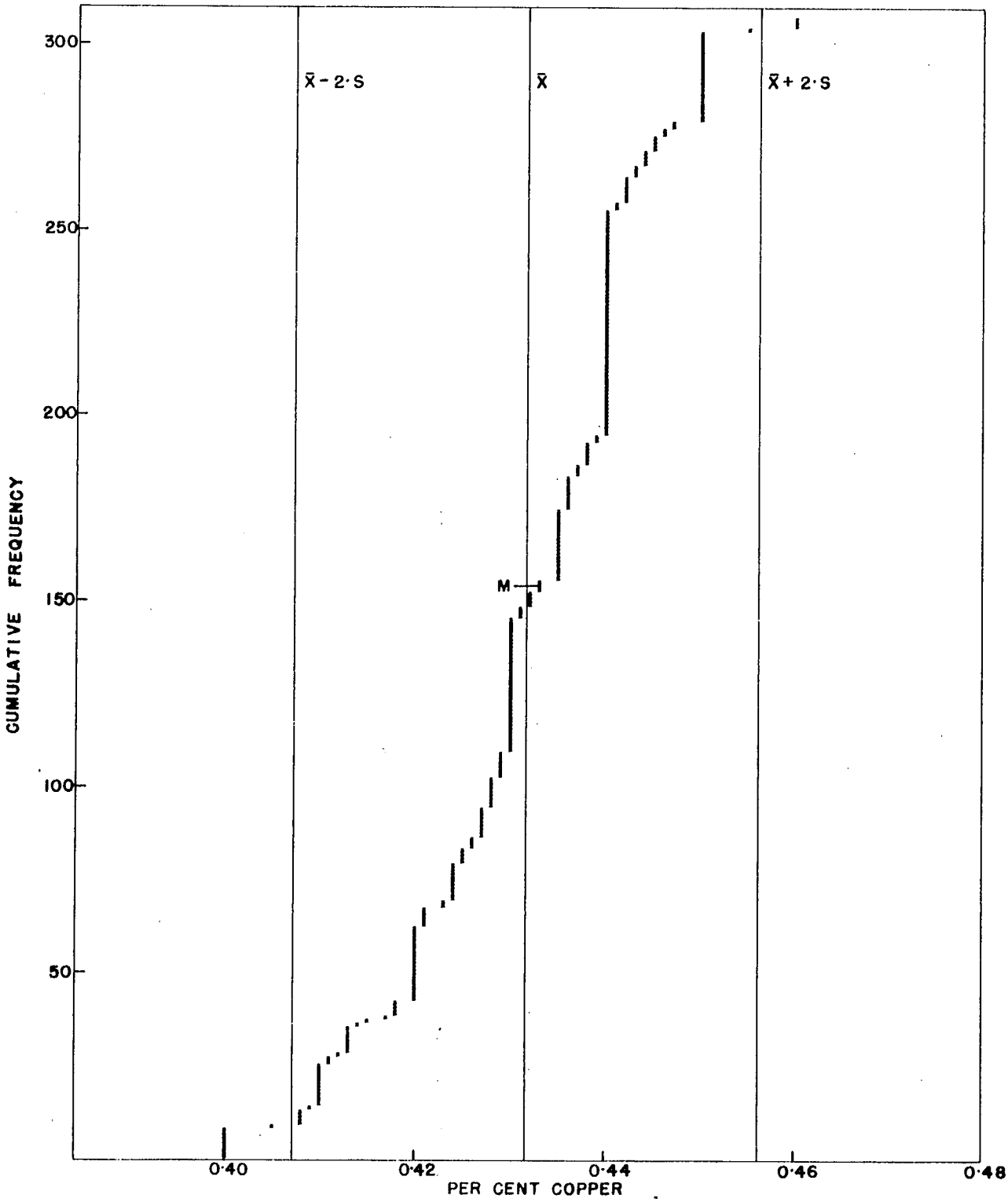


Figure 5. Cumulative Distribution for Copper Results.

UM-1, COBALT

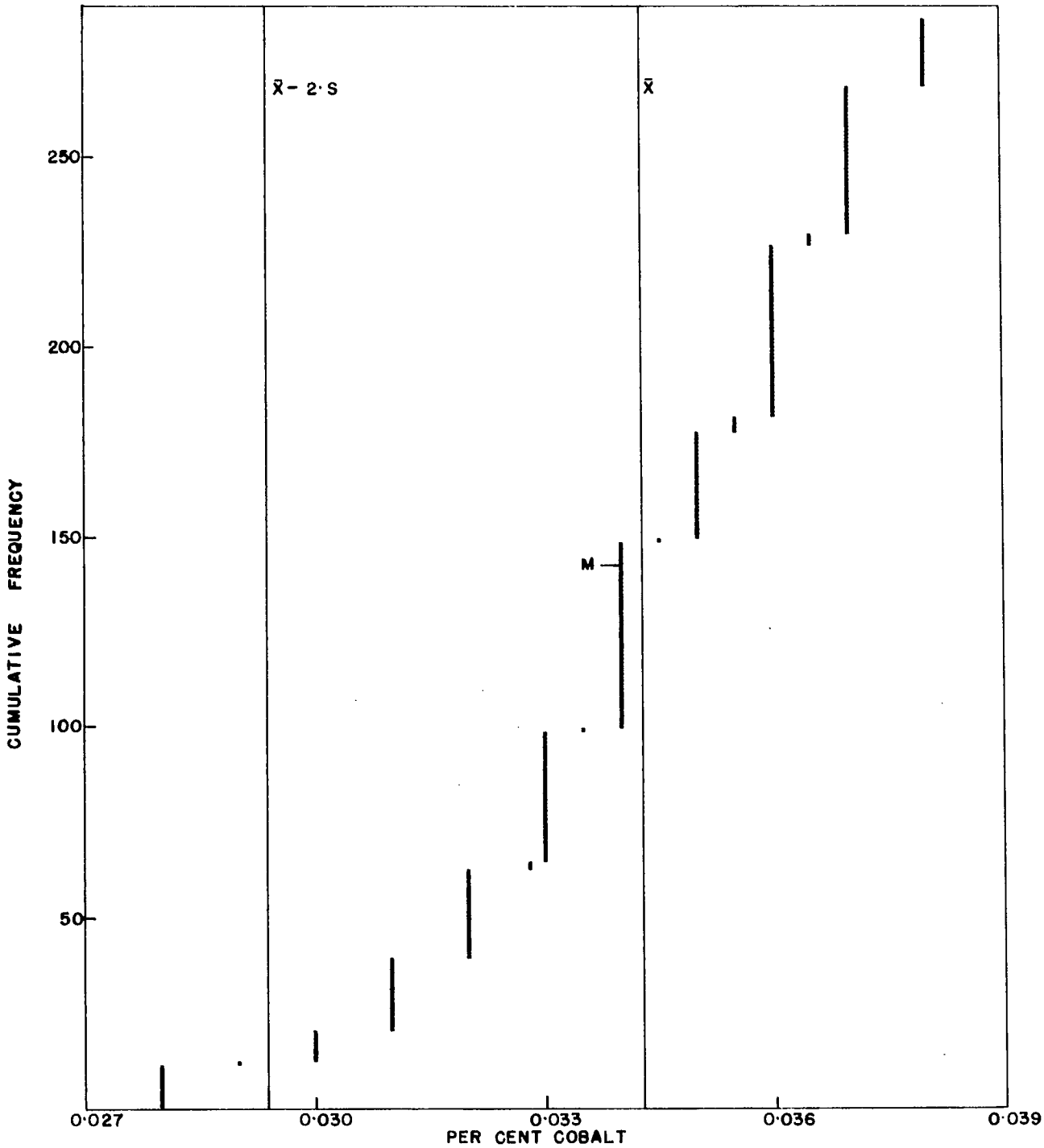


Figure 6. Cumulative Distribution for Cobalt Results.

SU-1, NICKEL

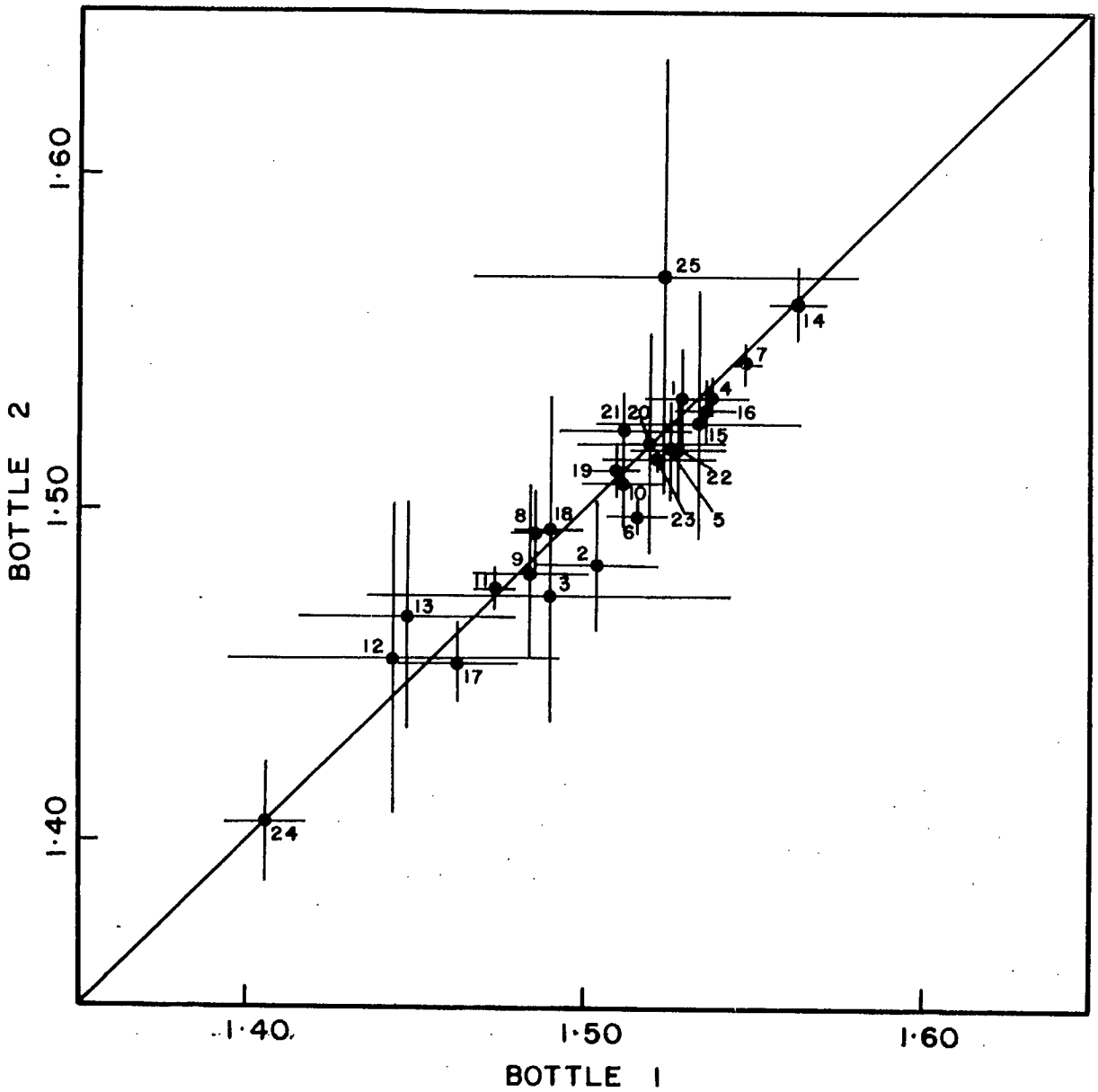


Figure 7. Average Nickel Analyses (%) from Each Participating Laboratory. (The crosses indicate one standard deviation on either side of the average for both bottles analyzed).

SU-1, COPPER

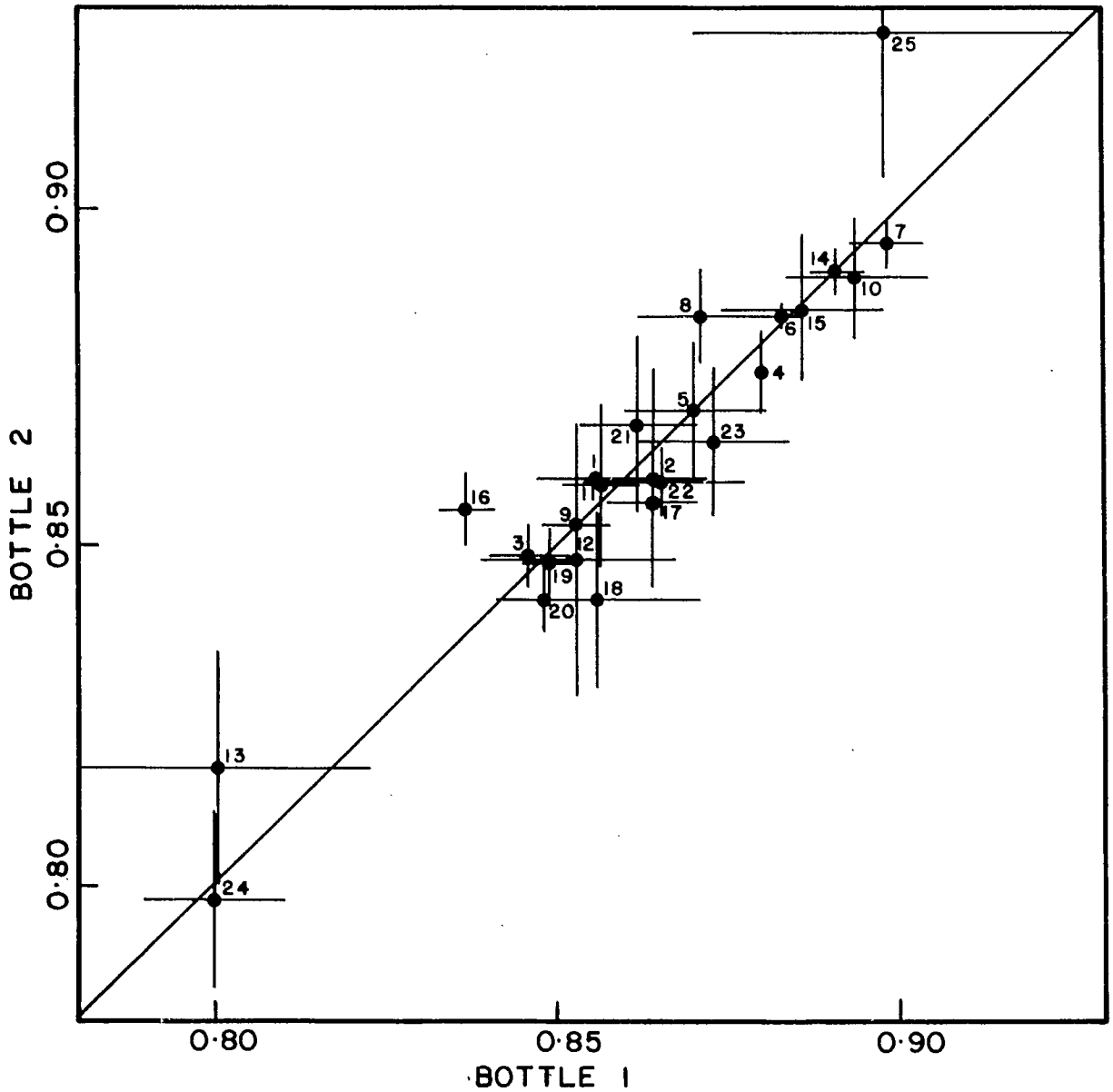


Figure 8. Average Copper Analyses (%) from Each Participating Laboratory. (The crosses indicate one standard deviation on either side of the average for both bottles analyzed).

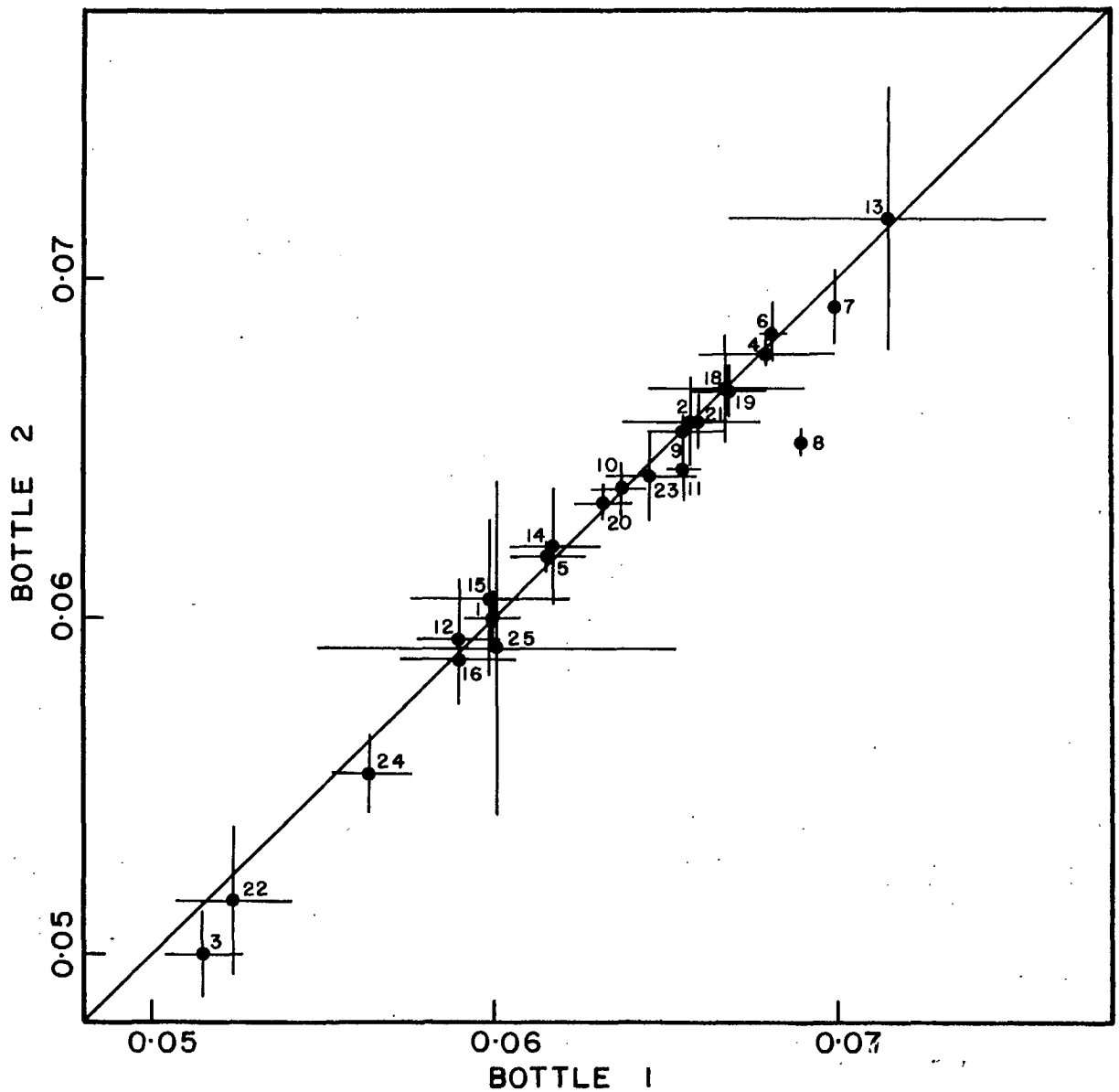


Figure 9. Average Cobalt Analyses (%) from Each Participating Laboratory. (The crosses indicate one standard deviation on either side of the average for both bottles analyzed).

UM-1, NICKEL

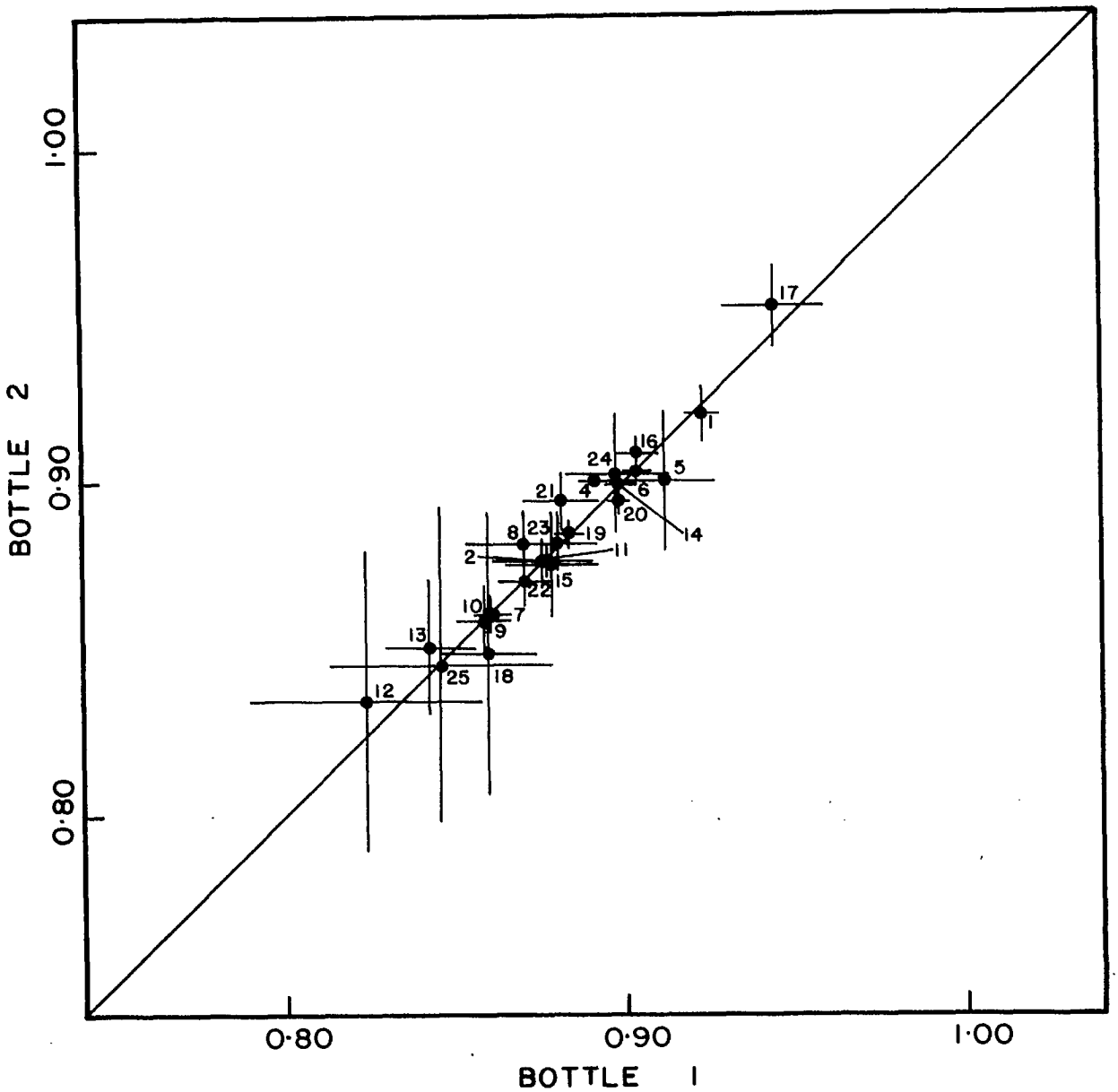


Figure 10. Average Nickel Analyses (%) from Each Participating Laboratory. (The crosses indicate one standard deviation on either side of the average for both bottles analyzed).

UM-1, COPPER

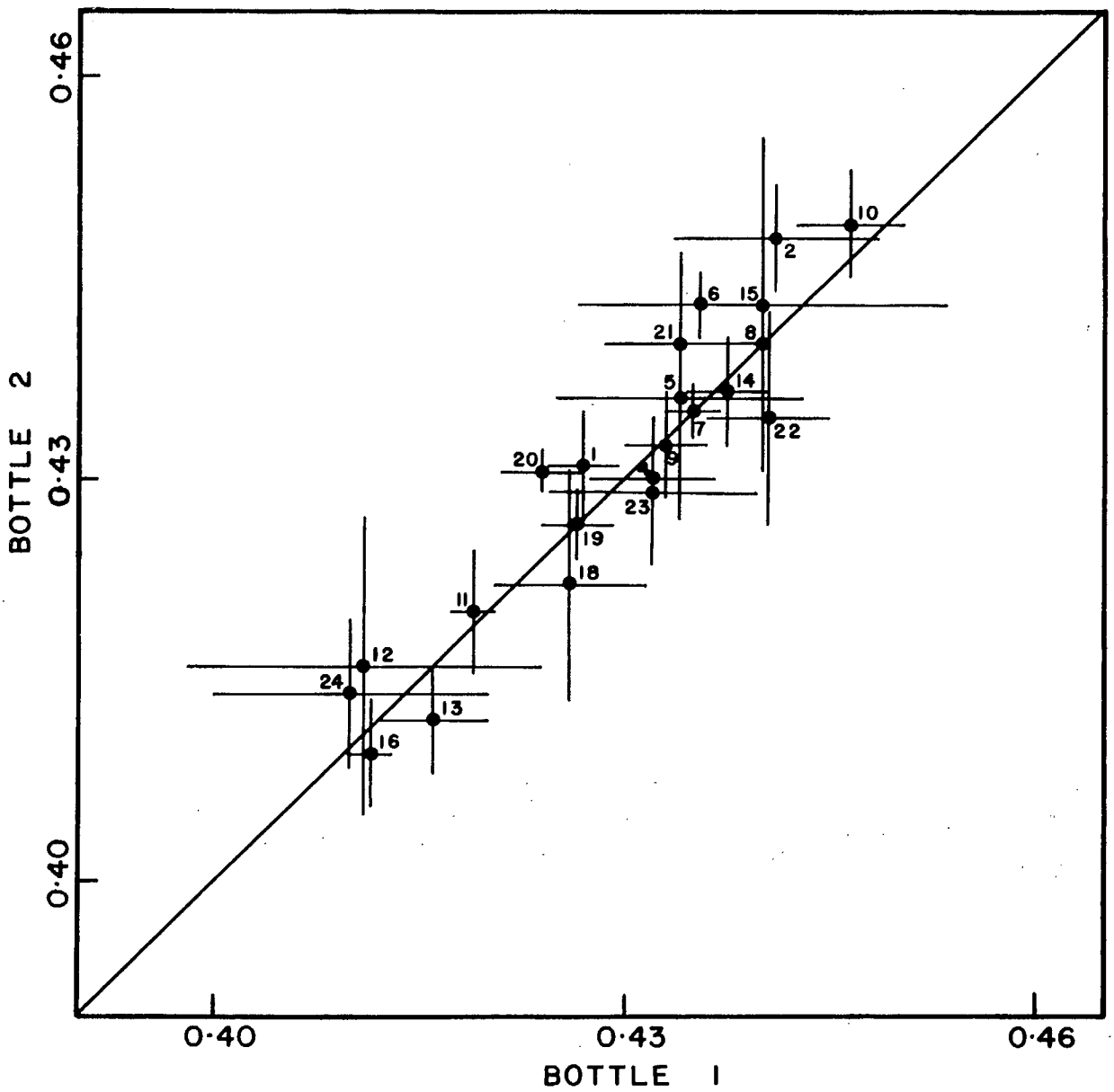


Figure 11. Average Copper Analyses (%) from Each Participating Laboratory. (The crosses indicate one standard deviation on either side of the average for both bottles analyzed).

UM-1, COBALT

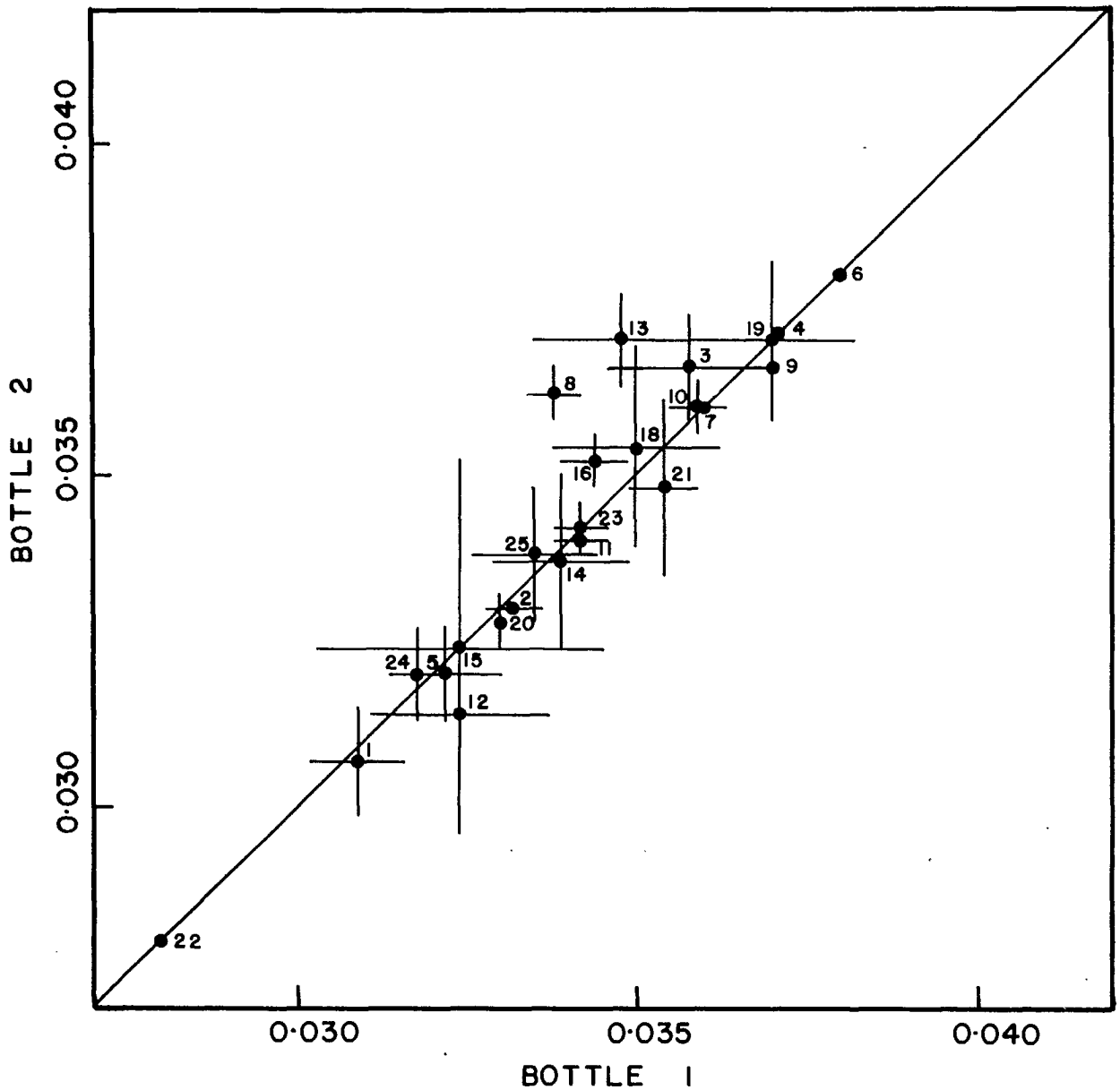


Figure 12. Average Cobalt Analyses (%) from Each Participating Laboratory. (The crosses indicate one standard deviation on either side of the average for both bottles analyzed).

TABLE 7

Statistical Parameters for SU-1 and UM-1, Based on the Assumption that the Analytical Results are all Independent*

| Elements | No. of Observations N | Median M(%) | Mean $\bar{x}$(%) | Standard Deviation s(%) | Coefficient of Variation c.v.(%) | Skewness Factor $\alpha_3$ | Kurtosis Coefficient $\alpha_4$ |
|---|---|---|---|---|---|---|---|
| | | | | SU-1 | | | |
| Nickel | 357 | 1.51 | 1.50 | 0.04 | 2.8 | -0.6 | 4.1 |
| | 333 | 1.51 | 1.51 | 0.03 | 2.1 | -0.1 | 2.5 |
| Copper | 339 | 0.86 | 0.86 | 0.03 | 2.9 | -0.4 | 4.5 |
| | 319 | 0.86 | 0.87 | 0.02 | 2.2 | 0.1 | 2.3 |
| Cobalt | 276 | 0.064 | 0.062 | 0.006 | 9.0 | -0.6 | 3.1 |
| | 254 | 0.064 | 0.063 | 0.004 | 7.1 | -0.5 | 2.9 |
| | | | | UM-1 | | | |
| Nickel | 336 | 0.88 | 0.88 | 0.03 | 3.3 | -0.6 | 5.2 |
| | 313 | 0.88 | 0.88 | 0.02 | 2.3 | 0.2 | 2.7 |
| Copper | 327 | 0.44 | 0.44 | 0.02 | 4.6 | 2.8 | 18.7 |
| | 312 | 0.43 | 0.43 | 0.01 | 2.9 | -0.5 | 3.0 |
| Cobalt | 286 | 0.034 | 0.034 | 0.002 | 7.1 | -0.6 | 2.9 |
| | 274 | 0.035 | 0.035 | 0.002 | 6.1 | -0.2 | 2.2 |

*The first set of parameters for each element was computed from all results; the second set was computed from results that deviate from the over-all means by no more than twice the standard deviation.

TABLE 8

Laboratory Means and Coefficient of Variation for SU-1

| Lab. No. | Nickel | | | Copper | | | Cobalt | | |
|---|---|---|---|---|---|---|---|---|---|
| | $n_i$ | $\bar{x}_i$ | $c.v._i$ (%) | $n_i$ | $\bar{x}_i$ | $c.v._i$ (%) | $n_i$ | $\bar{x}_i$ | $c.v._i$ (%) |
| 1 | 14 | 1.53 | 0.8 | 10 | 0.86 | 0.7 | 14 | 0.060 | 1.3 |
| 2 | 20 | 1.49 | 1.5 | 20 | 0.86 | 1.4 | 10 | 0.066 | 2.5 |
| 3 | 22 | 1.48 | 3.2 | 22 | 0.85 | 0.6 | 20 | 0.051 | 2.8 |
| 4 | 10 | 1.54 | 0.5 | 10 | 0.88 | 0.5 | 10 | 0.068 | 2.0 |
| 5 | 10 | 1.52 | 0.7 | 10 | 0.87 | 1.1 | 10 | 0.062 | 1.3 |
| 6 | 10 | 1.51 | 0.8 | 10 | 0.88 | 0.3 | 10 | 0.068 | 1.0 |
| 7 | 10 | 1.55 | 0.3 | 10 | 0.90 | 0.5 | 10 | 0.070 | 1.2 |
| 8 | 20 | 1.49 | 0.7 | 20 | 0.88 | 1.2 | 10 | 0.067 | 3.0 |
| 9 | 20 | 1.48 | 1.5 | 10 | 0.85 | 0.5 | 10 | 0.066 | 1.3 |
| 10 | 12 | 1.51 | 0.8 | 12 | 0.89 | 1.1 | 12 | 0.064 | 1.2 |
| 11 | 10 | 1.48 | 0.4 | 10 | 0.86 | 1.1 | 10 | 0.065 | 1.5 |
| 12 | 20 | 1.45 | 3.3 | 20 | 0.85 | 2.0 | 10 | 0.059 | 2.5 |
| 13 | 10 | 1.46 | 2.3 | 10 | 0.81 | 2.6 | 10 | 0.072 | 5.5 |
| 14 | 20 | 1.56 | 0.6 | 20 | 0.89 | 0.4 | 20 | 0.062 | 2.4 |
| 15 | 20 | 1.53 | 2.1 | 20 | 0.89 | 1.2 | 20 | 0.060 | 3.8 |
| 16 | 10 | 1.53 | 0.6 | 10 | 0.85 | 1.3 | 10 | 0.059 | 2.5 |
| 17 | 10 | 1.46 | 1.0 | 10 | 0.86 | 0.8 | - | - | - |
| 18 | 10 | 1.49 | 1.8 | 10 | 0.85 | 1.8 | 10 | 0.067 | 2.8 |
| 19 | 10 | 1.51 | 0.5 | 10 | 0.85 | 0.5 | 10 | 0.067 | 1.4 |
| 20 | 20 | 1.52 | 1.8 | 15 | 0.85 | 0.8 | 10 | 0.063 | 1.1 |
| 21 | 10 | 1.52 | 1.1 | 10 | 0.87 | 1.2 | 10 | 0.066 | 1.1 |
| 22 | 20 | 1.52 | 1.0 | 20 | 0.86 | 1.1 | 10 | 0.052 | 3.6 |
| 23 | 19 | 1.52 | 0.8 | 20 | 0.87 | 1.3 | 10 | 0.064 | 2.0 |
| 24 | 10 | 1.41 | 1.0 | 10 | 0.80 | 1.4 | 10 | 0.056 | 2.1 |
| 25 | 10 | 1.55 | 4.1 | 10 | 0.91 | 3.0 | 10 | 0.060 | 8.0 |
| Total | 357 | 1.50 | 2.8 | 339 | 0.86 | 2.9 | 276 | 0.062 | 9.0 |

TABLE 9

Laboratory Means and Coefficient of Variation for UM-1

| Lab. No. | Nickel | | | Copper | | | Cobalt | | |
|---|---|---|---|---|---|---|---|---|---|
| | $n_i$ | $\overline{x}_i$ | $c.v._i$ (%) | $n_i$ | $\overline{x}_i$ | $c.v._i$ (%) | $n_i$ | $\overline{x}_i$ | $c.v._i$ (%) |
| 1 | 14 | 0.92 | 0.7 | 10 | 0.43 | 0.9 | 14 | 0.031 | 2.3 |
| 2 | 20 | 0.88 | 1.3 | 20 | 0.44 | 1.5 | 10 | 0.033 | 1.0 |
| 3 | 10 | 0.88 | 0.4 | 10 | 0.44 | 0.8 | 30 | 0.036 | 2.8 |
| 4 | 10 | 0.90 | 0.6 | 10 | 0.43 | 0.7 | 10 | 0.037 | 0.0 |
| 5 | 10 | 0.91 | 2.0 | 10 | 0.44 | 2.0 | 10 | 0.032 | 2.3 |
| 6 | 10 | 0.90 | 0.4 | 10 | 0.44 | 0.9 | 10 | 0.038 | 0.0 |
| 7 | 10 | 0.86 | 0.4 | 10 | 0.43 | 0.4 | 10 | 0.036 | 0.0 |
| 8 | 20 | 0.88 | 1.6 | 20 | 0.44 | 0.0 | 10 | 0.035 | 3.8 |
| 9 | 20 | 0.86 | 1.1 | 10 | 0.43 | 0.8 | 10 | 0.037 | 1.1 |
| 10 | 12 | 0.86 | 0.6 | 12 | 0.45 | 0.9 | 12 | 0.036 | 1.1 |
| 11 | 10 | 0.88 | 0.5 | 10 | 0.42 | 0.7 | 10 | 0.034 | 0.9 |
| 12 | 20 | 0.83 | 4.7 | 20 | 0.41 | 2.9 | 10 | 0.032 | 3.5 |
| 13 | 10 | 0.85 | 1.9 | 10 | 0.41 | 1.0 | 10 | 0.036 | 4.2 |
| 14 | 20 | 0.90 | 0.4 | 20 | 0.44 | 0.8 | 20 | 0.034 | 3.3 |
| 15 | 20 | 0.88 | 1.7 | 20 | 0.44 | 2.9 | 20 | 0.032 | 7.4 |
| 16 | 10 | 0.91 | 0.6 | 10 | 0.41 | 0.7 | 10 | 0.035 | 1.8 |
| 17 | 10 | 0.95 | 1.5 | 10 | 0.48 | 1.2 | - | - | - |
| 18 | 10 | 0.85 | 3.5 | 10 | 0.42 | 1.6 | 10 | 0.035 | 3.7 |
| 19 | 10 | 0.88 | 0.5 | 10 | 0.43 | 0.5 | 10 | 0.037 | 3.1 |
| 20 | 10 | 0.90 | 0.5 | 15 | 0.43 | 0.9 | 10 | 0.033 | 1.0 |
| 21 | 10 | 0.89 | 1.3 | 10 | 0.44 | 1.5 | 10 | 0.035 | 2.8 |
| 22 | 20 | 0.87 | 0.9 | 20 | 0.44 | 1.6 | 10 | 0.028 | 0.0 |
| 23 | 20 | 0.88 | 1.1 | 20 | 0.43 | 1.3 | 10 | 0.034 | 1.2 |
| 24 | 10 | 0.90 | 1.7 | 10 | 0.41 | 1.9 | 10 | 0.032 | 1.8 |
| 25 | 10 | 0.85 | 4.5 | 10 | 0.49 | 12.0 | 10 | 0.034 | 2.7 |
| Total | 336 | 0.88 | 3.3 | 327 | 0.44 | 4.6 | 286 | 0.034 | 7.1 |

TABLE 10

Statistical Parameters for SU-1 and UM-1

| | Nickel | | | Copper | | | Cobalt | | |
|---|---|---|---|---|---|---|---|---|---|
| | A | B | C | A | B | C | A | B | C |
| **SU-1** | | | | | | | | | |
| No. of Participating Laboratories | 24 | 24 | 24 | 24 | 23 | 23 | 24 | 23 | 23 |
| No. of Observations | 333 | 337 | 337 | 319 | 319 | 319 | 254 | 256 | 256 |
| Median, % | 1.51 | 1.51 | 1.51 | 0.86 | 0.86 | 0.86 | 0.064 | 0.064 | 0.064 |
| Mean, % | 1.51 | 1.51 | 1.51 | 0.87 | 0.87 | 0.87 | 0.063 | 0.063 | 0.064 |
| 95% Confidence Interval of the Mean, % | | | | | | | | | |
| Low | 1.51 | 1.50 | 1.50 | 0.86 | 0.86 | 0.86 | 0.063 | 0.061 | 0.062 |
| High | 1.51 | 1.52 | 1.52 | 0.87 | 0.88 | 0.88 | 0.064 | 0.065 | 0.065 |
| **UM-1** | | | | | | | | | |
| No. of Participating Laboratories | 25 | 24 | 24 | 24 | 23 | 23 | 23 | 23 | 23 |
| No. of Observations | 313 | 316 | 316 | 312 | 307 | 307 | 274 | 276 | 276 |
| Median, % | 0.88 | 0.88 | 0.88 | 0.43 | 0.43 | 0.43 | 0.035 | 0.034 | 0.034 |
| Mean, % | 0.88 | 0.88 | 0.88 | 0.43 | 0.43 | 0.43 | 0.035 | 0.035 | 0.035 |
| 95% Confidence Interval of the Mean, % | | | | | | | | | |
| Low | 0.88 | 0.87 | 0.87 | 0.43 | 0.43 | 0.43 | 0.034 | 0.034 | 0.034 |
| High | 0.88 | 0.89 | 0.89 | 0.43 | 0.44 | 0.44 | 0.035 | 0.035 | 0.035 |

TABLE 11

Recommended Values and Their Confidence Intervals for Standard Reference Materials SU-1 and UM-1

| SU-1 | | | |
|---|---|---|---|
| | % Nickel | % Copper | % Cobalt |
| Recommended Value | 1.51 | 0.87 | 0.063 |
| 95% Confidence Intervals | | | |
| Low | 1.50 | 0.86 | 0.061 |
| High | 1.52 | 0.88 | 0.065 |
| **UM-1** | | | |
| | % Nickel | % Copper | % Cobalt |
| Recommended Value | 0.88 | 0.43 | 0.035 |
| 95% Confidence Intervals | | | |
| Low | 0.87 | 0.43 | 0.034 |
| High | 0.89 | 0.44 | 0.035 |

TABLE 12

Comparison of Analytical Methods for SU-1

| Analytical Method | Nickel | | | | Copper | | | | Cobalt | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. of Labs | N | $\bar{x}$ | c.v.(%) | No. of Labs | N | $\bar{x}$ | c.v.(%) | No. of Labs | N | $\bar{x}$ | c.v.(%) |
| Atomic absorption | 21 | 210 | 1.50 | 3.0 | 22 | 222 | 0.86 | 3.0 | 21 | 212 | 0.063 | 8.7 |
| Volumetric | 2 | 20 | 1.51 | 1.4 | 6 | 60 | 0.87 | 1.8 | -- | - | - | - |
| Gravimetric | 10 | 93 | 1.51 | 2.0 | -- | - | - | - | -- | - | - | - |
| Spectrophotometric (colorimetric) | 1 | 4 | 1.54 | 1.2 | 2 | 15 | 0.87 | 1.6 | 6 | 54 | 0.60 | 8.8 |
| Polarographic | 1 | 10 | 1.49 | 0.7 | 2 | 12 | 0.87 | 1.1 | -- | - | - | - |
| Electrolytic | -- | - | - | - | 1 | 10 | 0.89 | 0.5 | -- | - | - | - |
| X-ray Fluorescence | 1 | 10 | 1.46 | 1.0 | 1 | 10 | 0.86 | 0.8 | -- | - | - | - |
| Emission Spectrographic | 1 | 10 | 1.55 | 4.1 | 1 | 10 | 0.91 | 3.0 | 1 | 10 | 0.060 | 8.0 |
| Total, non-a.a. | 16 | 147 | 1.51 | 2.3 | 13 | 117 | 0.87 | 2.4 | 7 | 64 | 0.060 | 8.6 |
| All results | 37* | 357 | 1.50 | 2.8 | 35* | 339 | 0.86 | 2.9 | 28* | 276 | 0.062 | 9.0 |

*Many of the 25 participating laboratories reported results by more than one method.

TABLE 13

Comparison of Analytical Methods for UM-1

| Analytical Method | Nickel | | | | Copper | | | | Cobalt | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | No. of Labs | N | $\bar{x}$ | c.v.(%) | No. of Labs | N | $\bar{x}$ | c.v.(5) | No. of Labs | N | $\bar{x}$ | c.v.(%) |
| Atomic absorption | 21 | 203 | 0.87 | 3.3 | 22 | 215 | 0.43 | 2.9 | 21 | 217 | 0.034 | 7.0 |
| Volumetric | 2 | 20 | 0.89 | 2.9 | 6 | 55 | 0.44 | 2.6 | -- | - | - | - |
| Gravimetric | 9 | 79 | 0.89 | 1.7 | -- | - | - | - | -- | - | - | - |
| Spectrophotometric (colorimetric) | 1 | 4 | 0.92 | 0.6 | 2 | 15 | 0.43 | 1.5 | 6 | 59 | 0.034 | 8.1 |
| Polarographic | 1 | 10 | 0.87 | 0.6 | 2 | 12 | 0.44 | 1.8 | -- | - | - | - |
| Electrolytic | -- | - | - | - | 1 | 10 | 0.44 | 1.0 | - | - | - | - |
| X-ray Fluorescence | 1 | 10 | 0.95 | 1.5 | 1 | 10 | 0.48 | 1.2 | -- | - | - | - |
| Emission Spectrographic | 1 | 10 | 0.85 | 4.5 | 1 | 10 | 0.49 | 12.0 | 1 | 10 | 0.034 | 2.7 |
| Total, non-a.a. | 15 | 133 | 0.89 | 3.2 | 13 | 112 | 0.45 | 6.1 | 7 | 69 | 0.034 | 7.6 |
| All results | 36* | 336 | 0.88 | 3.3 | 35* | 327 | 0.44 | 4.6 | 28* | 286 | 0.034 | 7.1 |

*Many of the 25 participating laboratories reported results by more than one method.

## DISCUSSION

Many laboratories voluntarily determined nickel, copper and cobalt in SU-1 and UM-1 by more than one analytical method. As expected, results obtained by atomic-absorption methods are preponderant. A comparison of the means of the pooled results for each class of method (Tables 12 and 13) shows that there are small differences between the results obtained by atomic-absorption methods and those by non-atomic-absorption methods. For both SU-1 and UM-1, the non-atomic-absorption results for nickel and copper are higher than the atomic-absorption results. However, there is no additional evidence that these differences reflect a real bias of the atomic-absorption technique to give low results for the two ores. Therefore, the authors suggest that the most useful values for copper and nickel are the average results which are the recommended values given in Table 11.

Previously, it was noted that many laboratories used hydrofluoric acid in their sample decomposition procedures. However, a substantial fraction of the participating laboratories did not use this acid (Table 5); therefore, a comparison of the atomic-absorption results obtained after the two methods of decomposition was made for both SU-1 and UM-1. Table 14 shows that there is a definite bias toward higher results for nickel, copper and cobalt from laboratories that used hydrofluoric acid in their decomposition of SU-1. Table 14 also shows that this bias is less evident for the analytical results for UM-1. The implication of this correlation, especially for SU-1, is that a small fraction of siliceous matter remained undecomposed by acid mixtures not containing hydrofluoric acid and that, in the larger grains, some base-metal minerals remained entrapped. This argument is supported by the data in Table 1, which show that 15% by weight of SU-1 is plus 100-mesh, whereas UM-1 is entirely minus 100-mesh.

It is to be noted that, because most results for SU-1 and UM-1 were obtained after a decomposition that involved hydrofluoric acid, the higher values for nickel, copper, and cobalt given in Table 14 correspond closely with the recommended values for these elements (Table 11).

TABLE 14

Correlation of Atomic-Absorption Results for SU-1 and UM-1 with Nature of Acid Attack

| | Nickel | | Copper | | Cobalt | |
|---|---|---|---|---|---|---|
| | N | $\bar{x}$ (wt %) | N | $\bar{x}$ (wt %) | N | $\bar{x}$ (wt %) |
| **SU-1** | | | | | | |
| HF used | 142 | 1.510 | 152 | 0.865 | 152 | 0.065 |
| HF not used | 68 | 1.482 | 70 | 0.848 | 60 | 0.060 |
| Total | 210 | 1.501 | 222 | 0.860 | 212 | 0.063 |
| **UM-1** | | | | | | |
| HF used | 142 | 0.877 | 152 | 0.432 | 152 | 0.035 |
| HF not used | 61 | 0.869 | 63 | 0.425 | 65 | 0.034 |
| Total | 203 | 0.875 | 215 | 0.430 | 217 | 0.034 |

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