



**AUSTRALIAN ATOMIC ENERGY COMMISSION
RESEARCH ESTABLISHMENT
LUCAS HEIGHTS**

**EFFECT OF VARIATIONS OF ENTRAINED AIR IN MINERAL SLURRIES
ON THE PRECISION OF ON-STREAM ANALYSIS USING
RADIOISOTOPE X-RAY TECHNIQUES**

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ABSTRACT

Variations in entrained air in mineral slurries cause errors in on-stream X-ray analysis for element concentration in the slurry solids. Complete elimination of entrained air may not always be practicable, particularly for radioisotope X-ray techniques where probes are immersed directly into existing plant slurry vessels through which the whole process stream flows.

Calculations of the effect of entrained air on precision of analysis show that X-ray preferential absorption techniques are usually much more affected than X-ray fluorescence techniques. Calculations agree reasonably well with results of plant trials of on-stream analysis.

Errors in analysis using radioisotope immersion probes should in most cases be within limits acceptable for plant control.

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ABSORPTION; ACCURACY; AIR; ENTRAINMENT; ERRORS; FLUORESCENCE;
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Table 1 On-Stream Analysis of Plant Mineral Slurries, Including Experimental and Predicted Errors in Element Concentration Caused by Variations in Entrained Air

Figure 1 Radioisotope probes immersed into slurry in an existing plant vessel

Figure 2 Errors in weight fraction (W_s) of solids in slurry caused by changes in voidage in the slurry

Figure 3 Determination of copper in copper concentrate slurries

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1. INTRODUCTION

On-stream analysis installations based on radioisotope X-ray techniques are being introduced into mineral processing plants (Fookes et al. 1971). Three types of radioisotope probes are used, depending on the techniques of γ -ray absorption, X-ray preferential absorption (XRA) and X-ray fluorescence (XRF) (Ellis et al. 1969). The concentrations of elements in the slurry solids and the density of the slurry are computed from one or more of the simultaneous measurements of signals from the probes.

The probes can be immersed directly into existing slurry vessels in the plant (Figure 1). This major advance (Hinckfuss and Stump 1971) eliminates the need for sample by-lines and hence greatly simplifies the plant analysis system. However, the essentially complete elimination of air entrained in the slurry, which is possible with sample by-lines, may not always be practicable with the larger flows in existing vessels.

This paper reports an investigation of the effect of variations in entrained air on precision of on-stream analysis.

2. EFFECTS OF ENTRAINED AIR

Several radioisotope probes are immersed in the same slurry vessel, and a continuous stream of slurry containing entrained air is seen by each probe. The air normally occurs as very fine bubbles dispersed throughout the slurry. Variations in entrained air change the voidage and hence the mean density of the slurry.

In what follows it is assumed that variations in entrained air occur slowly compared with the limited time (tens of seconds) required for measurement of probe signals, and that each probe sees essentially similar samples of slurry during this limited time. Entrained air and hence mean density of slurry is thus assumed constant throughout the vessel during the measurement time. These assumptions have been shown to be valid for vessels used in tests at the plant of New Broken Hill Consolidated Ltd. (N. W. Stump, private communication) where the density of the aerated slurry was quickly scanned throughout the whole vessel using a γ -ray absorption probe.

γ -ray absorption probes measure mean density of the slurry (i.e. of the slurry-air mixture) and hence are directly affected by variations in entrained air. XRA and XRF probes are also affected by changes in mean density of the slurry, and measurements corresponding to slurries containing no air can be obtained from the signals measured with aerated slurries compensated by the measurement of the γ -ray absorption probe. The compensation required for XRF signals is often small or negligible because, with the backscatter geometry used, an infinite thickness of slurry is seen. Thus the only effect that variations in density of the slurry have on the intensity of X-rays detected is related to geometrical factors which depend on the path length of X-rays in the slurry. However, a larger compensation is required for XRA signals because these depend directly on the weight per unit area of slurry between source and detector.

Concentration of the wanted element per unit volume can be precisely determined by XRA techniques independently of voidage because the analysis does not depend on knowledge of solids weight fraction in the slurry. It follows that mass flow of the element can also be determined independently of voidage if (a) the velocity of flow of the slurry in the inlet or outlet pipe to the slurry vessel can be measured and the cross-sectional area of this pipe is known, and (b) the voidage at the measuring point is the same or bears a known relationship to the voidage in the slurry vessel, or the density of the slurry in the pipe is also measured. Knowledge of the mass flow of element would appear to be important for control of flotation processes.

XRF techniques to determine the concentration of the wanted element per unit volume depend slightly on voidage because knowledge of weight fraction of solids in the slurry is necessary and cannot be determined independently of voidage (Section 3).

The more commonly stated requirement of analysis is for concentration of the wanted element per unit weight of the slurry solids (wt/wt) and for this the weight fraction of solids in the slurry must be known. The γ -ray absorption measurement made to determine the weight fraction of solids depends on both mean slurry density and voidage. Hence the precision of analysis is affected by variations in entrained air and calculations of the magnitude of this error are now outlined.

3. SOLIDS DETERMINATION

W_s , the weight fraction of solids in the aqueous slurry, can be expressed in terms of density of the slurry solids ρ_s , mean density of slurry ρ , and voidage V by:

$$W_s = \left(\frac{\rho_s}{\rho_s - 1} \right) \left(1 - \frac{1 - V}{\rho} \right) \quad (1)$$

This is a slight modification of the equation quoted by Carr-Brion (1967). The mean slurry density is determined by a γ -ray absorption measurement:

$$\rho = \frac{1}{\mu x} \log \left(\frac{I_0}{I} \right), \quad (2)$$

where μ is the mass absorption coefficient of γ -rays in the slurry, x the path length traversed by the γ -rays in the slurry, and I and I_0 are respectively the intensities of detected γ -rays with and without slurry.

W_s is determined from equations (1) and (2). Mineral densities are usually much greater than one, and since wide variations in mineral composition usually do not occur (Carr-Brion 1967), $\rho_s / (\rho_s - 1)$ may often be assumed constant for many control purposes. Errors in W_s are caused by uncertainty in voidage and are greatest at low slurry densities. These errors, calculated from equation (1) and expressed as fractions $\Delta W_s / W_s$, are shown in Figure 2. For example, an uncertainty in voidage of 1% by volume of a slurry of density 1.3 g cm⁻³ causes an error of 3.3% of the weight fraction of solids.

4. PREFERENTIAL ABSORPTION

Preferential absorption techniques (Ellis et al. 1969) are often used to determine the concentration of elements of higher atomic number. Absorption measurements are usually made with X-rays of energy just above, and just below, the absorption edge of the wanted element. The higher energy X-rays are more affected by changes in concentration of the wanted element, but are also absorbed by the solids matrix and by water. The measurement with the lower energy X-rays is used to compensate the first for the absorption by the solids matrix and water. The general equations relating the concentration C of the wanted element to probe signals are:

$$(\mu_{el} - \mu_M) C W_s + \mu_M W_s + (1 - W_s) \mu_w = \frac{1}{\rho x} \log \left(\frac{I_0}{I} \right) \quad (3)$$

$$(\mu_{el}^1 - \mu_M^1) C W_s + \mu_M^1 W_s + (1 - W_s) \mu_w^1 = \frac{1}{\rho x} \log \left(\frac{I_0^1}{I^1} \right), \quad (4)$$

where the superscript ¹ refers to the measurement with the lower energy X-rays, μ is the mass absorption coefficient, and the subscripts _{el}, _M, and _w refer respectively to wanted element, solids matrix, and water. In practice $\mu_{el}^1 \ll \mu_{el}$, but $\mu_M \sim \alpha \mu_M^1$ and $\mu_w \sim \alpha \mu_w^1$ where α is a constant of value slightly greater than 1.

The concentration of the wanted element (C) can only be determined approximately by combining equations (1) to (4) because there are four unknown variables (C , μ_M , W_s and V) and only three measurements made. The error in C introduced by uncertainty in voidage can be calculated from the general equation for C obtained by rearranging and combining equations (2) to (4):

$$C = \frac{f(\text{probe signals})}{W_s}, \quad (5)$$

where f is a function of measured probe signals and known constants. The smallest obtainable fractional error $\Delta C / C$ is $\Delta W_s / W_s$ (which can be simply obtained from plant measurements using a density probe, compared with W_s measured by conventional assay of samples taken from the slurry stream).

When no measurement is made using the lower energy X-rays (equation 4), the concentration of the wanted element is determined by combining equations (2) and (3):

$$C = \frac{g(\text{probe signals}) - (\mu_M - \mu_w) W_s}{(\mu_{el} - \mu_M) W_s} \quad (6)$$

where g is a function of measured probe signals and known constants. The fractional error in C in this case is greater than $\Delta W_s / W_s$, and can be calculated simply, knowing the approximate concentrations of the various elements in the solids and the errors in W_s from plant experiments.

These equations have been used to predict the error in determination of lead concentration caused by high and variable amounts of entrained air encountered in two process streams during plant trials. Agreement between the experimental and predicted errors in Table 1 is reasonable. The quoted experimental errors in C due to variations in entrained air during the plant experiments are $\sqrt{R^2 - r^2}$. R is the total r.m.s. error in lead concentration determined from a least squares fit of functions of probe signals compared with lead concentrations assayed by conventional means on samples taken from the streams, and r is the r.m.s. error in lead concentration when the gauge signals are combined with W_s accurately determined from assay of the same samples. This method of determining errors due to entrained air is reasonably accurate when R is much greater than r and when errors in W_s are mainly due to air variations as in the examples selected.

5. X-RAY FLUORESCENCE

The general equation to determine element concentration in the slurry solids by X-ray fluorescence techniques is:

$$C = a N_f \left\{ \left[\sum_i (\mu_i + \mu_{K_i}) C_i - (\mu_w + \mu_{K_w}) \right] + \frac{1}{W_s} (\mu_w + \mu_{K_w}) \right\} \quad (7)$$

where N_f is the intensity of fluorescent X-rays from the wanted element, 'a' is a constant, μ and μ_K are respectively, the mass absorption coefficients of incident and K X-rays of the wanted element, and subscripts 'i' and 'w' respectively refer to the i^{th} element in the solids and to water.

The effect of variations of entrained air is again to introduce an uncertainty in the determination of weight fraction of solids and hence in concentration of the wanted element. Variations in entrained air have no effect on determination of element concentration if

$$\sum_i (\mu_i + \mu_{K_i}) C_i \gg (\mu_w + \mu_{K_w}) .$$

This is the case where X-rays are absorbed essentially only in the slurry solids and hence the water can be considered to be transparent to X-rays at the energies of interest.

The effect of air is most pronounced when in equation (7) the term

$$\left[\sum_i (\mu_i + \mu_{K_i}) C_i - (\mu_w + \mu_{K_w}) \right] \ll (\mu_w + \mu_{K_w}) / W_s$$

and in this case

$$\Delta C / C = \Delta W_s / W_s .$$

In practice this error is approached in slurries which contain little solids and when high energy X-rays are used with slurries containing solids of low atomic number.

XRF measurements are often made in analyses for the lower and medium atomic number elements and the fractional error $\Delta C / C$ is usually much smaller than $\Delta W_s / W_s$. This error can be calculated from plant measurements of $\Delta W_s / W_s$ and approximate values of μ_i and μ_{K_i} from known element concentrations in the solids at the prescribed X-ray energies.

Errors in XRF analysis are more significant when the intensity of fluorescent X-rays (N_f) is not determined from one or more probe signals, but instead from a term containing N_f plus other fluorescent X-rays and/or scattered X-rays from the sample. For example, in the determination of copper in copper concentrates from Cobar Mines Pty. Ltd., N.S.W. (Fookes et al. 1971), the XRF probe signal is appreciable even at zero copper concentration because of detected iron K X-rays (Figure 3). In this case the errors in determination of concentration of the wanted element introduced by variations in entrained air can be found by calculating the effect of the error in W_s (from plant trials) on copper concentration from laboratory measurements (Figure 3). The agreement between predicted errors in copper concentration and those determined experimentally in plant trials is fair (Table 1). This was the only stream encountered in seven plant trials in which entrained air was established to be the main cause of error in XRF analysis, and the air content of the slurry was in practice simply reduced by modifications to sampling technique. This error in analysis could also have been reduced by using a second probe to measure iron K X-rays alone, and hence enabling the intensity of copper fluorescent X-rays to be determined more precisely.

Plant trials in Australia have shown that it is unusual to find a mineral slurry stream with entrained air of sufficiently high and variable content to affect the precision of XRF analysis at the levels required for plant control.

6. DISCUSSION

Errors in element concentration determination due to variations in voidage can be predicted from plant measurements of errors in solids weight fraction combined with simple calculations and/or laboratory measurements on solids taken from plant streams. If these errors in element concentration determination are too great for plant control purposes, minor modifications to plant will often reduce entrained air in the measuring vessel. For example, air entrained in the flotation feed stream at the New Broken Hill Consolidated Ltd. plant has been much reduced by simply changing the speed of the pump and thereby making a more constant flow from the flotation feed sump (N.W. Stump, private communication).

These simple methods for predicting errors in element concentration due to entrained air enable a step-by-step approach to modifications to plant to reduce entrained air. For example, this approach would involve starting with a simple modification, testing by the above method to check for errors in element concentration, and where reduction is insufficient, making an additional modification and then testing again, etc.

Most difficulty with entrained air is likely to occur when preferential absorption techniques are used for the analysis, a high precision of analysis is necessary (e.g. for concentrate streams), the stream has a low slurry density, and the slurry contains a large and variable amount of entrained air (e.g. concentrate streams in plants depending on flotation processes). All these factors apply to the lead concentrate stream at the plant of North Broken Hill Pty. Ltd., Broken Hill, N.S.W. The slurry density is about 1.3 g cm^{-3} , and the required high precision of analysis is $\pm 1\%$ of the mean lead concentration. Plant trials would be required to determine whether the desired precision could be obtained using immersion probes.

In extreme and unusual cases where entrained air may continue to be a problem even after minor modification to plant, it can be overcome by the use of a sample by-line or by a direct measure of water per unit volume using a neutron backscatter probe. X-ray preferential absorption analysis can often be replaced by XRF analysis which is less affected by air.

The precision of on-stream analysis required for plant control purposes, expressed in per cent of the mean element concentration ($\Delta C/C\%$), is usually between one and five per cent for concentrate streams, two to ten per cent for feeds, and five to fifteen per cent for tailings.

Seven plant trials in Australia on a total of 14 mineral slurry streams containing copper, zinc, tin or lead have shown that these required precisions can be realised using radioisotope techniques. Sample by-lines were used in all but the last of these trials, and some effort was made to eliminate air in the by-lines. More plant trials with immersion probes should be made to prove that errors in analysis due to entrained air can be kept within the limits required for plant control purposes.

These plant trials would be part of the feasibility assessment undertaken when a plant installation for on-stream analysis is being considered. Most of this assessment, including choice of radioisotope X-ray technique, effect of variations in mineral composition on precision of analysis, and effect of changes in solids weight fraction, is best carried out in the laboratory. Plant experiments are now required only to determine the vessels into which the probes should be immersed and to test them (using a γ -ray absorption probe) for uniformity of slurry density throughout the vessel and for variations in entrained air with time.

7. CONCLUSION

The effect of variations of entrained air in mineral slurries on precision of on-stream analysis using radioisotope X-ray techniques has been calculated on the assumption that air is uniformly dispersed throughout the slurry. The error in determining the concentration of element in the slurry solids is often significant when X-ray preferential absorption techniques are used, but in most cases is likely to be within limits acceptable for plant control.

Analysis for concentration of element per unit volume is not affected by variations in entrained air. This analysis is required for determination of the mass flow of the element which appears to be of fundamental importance for control of flotation processes.

8. ACKNOWLEDGMENT

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TABLE 1
ON-STREAM ANALYSIS OF PLANT MINERAL SLURRIES, INCLUDING
EXPERIMENTAL AND PREDICTED ERRORS IN ELEMENT CONCENTRATION
CAUSED BY VARIATIONS IN ENTRAINED AIR

Plant Stream	Solid Weight Fraction (W_s) (% by wt.)	$\frac{\Delta W_s}{W_s}$ (%)	Wanted Element	Concentration of Element in Solids (C) (% by wt.)		$\frac{\Delta C}{C}$ (%)		Technique*
				Range	Mean	Expt.	Calculated	
Copper Concentrate	26	9.8	lead	0.7-5.4	1.8	23	24	XRA
						8.5	9.8	
Flotation Feeds	51	5	lead	6-15	9	6.7	5.6	XRA
Copper Concentrate	26	9.8	copper	17-29	24	6	4.0	XRF

* XRA: X-ray preferential absorption

XRF: X-ray fluorescence

+ This is the only case quoted in this Table for which compensation for matrix absorption was made.

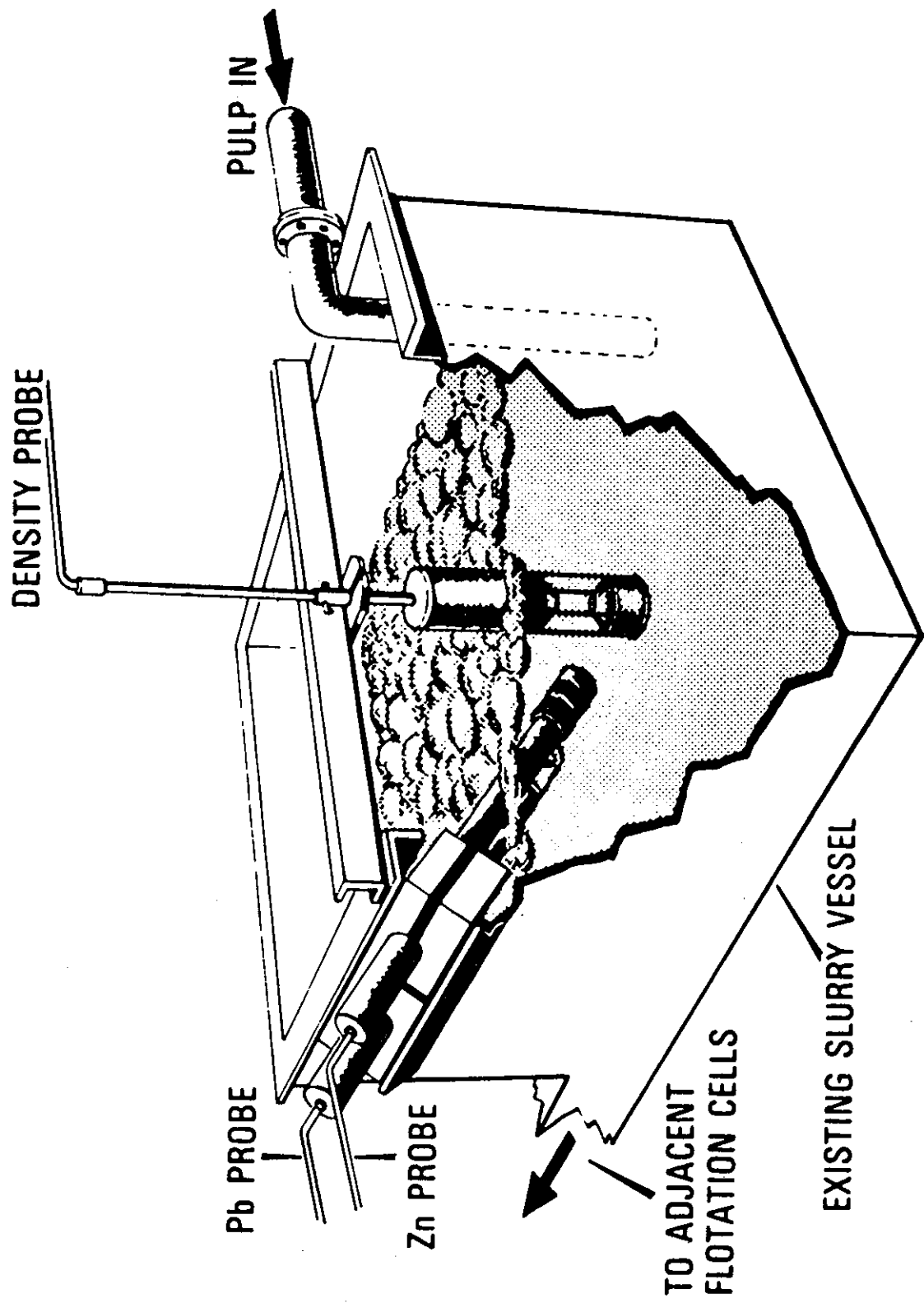


FIGURE 1. RADIOISOTOPE PROBES IMMERSSED INTO SLURRY IN AN EXISTING PLANT VESSEL

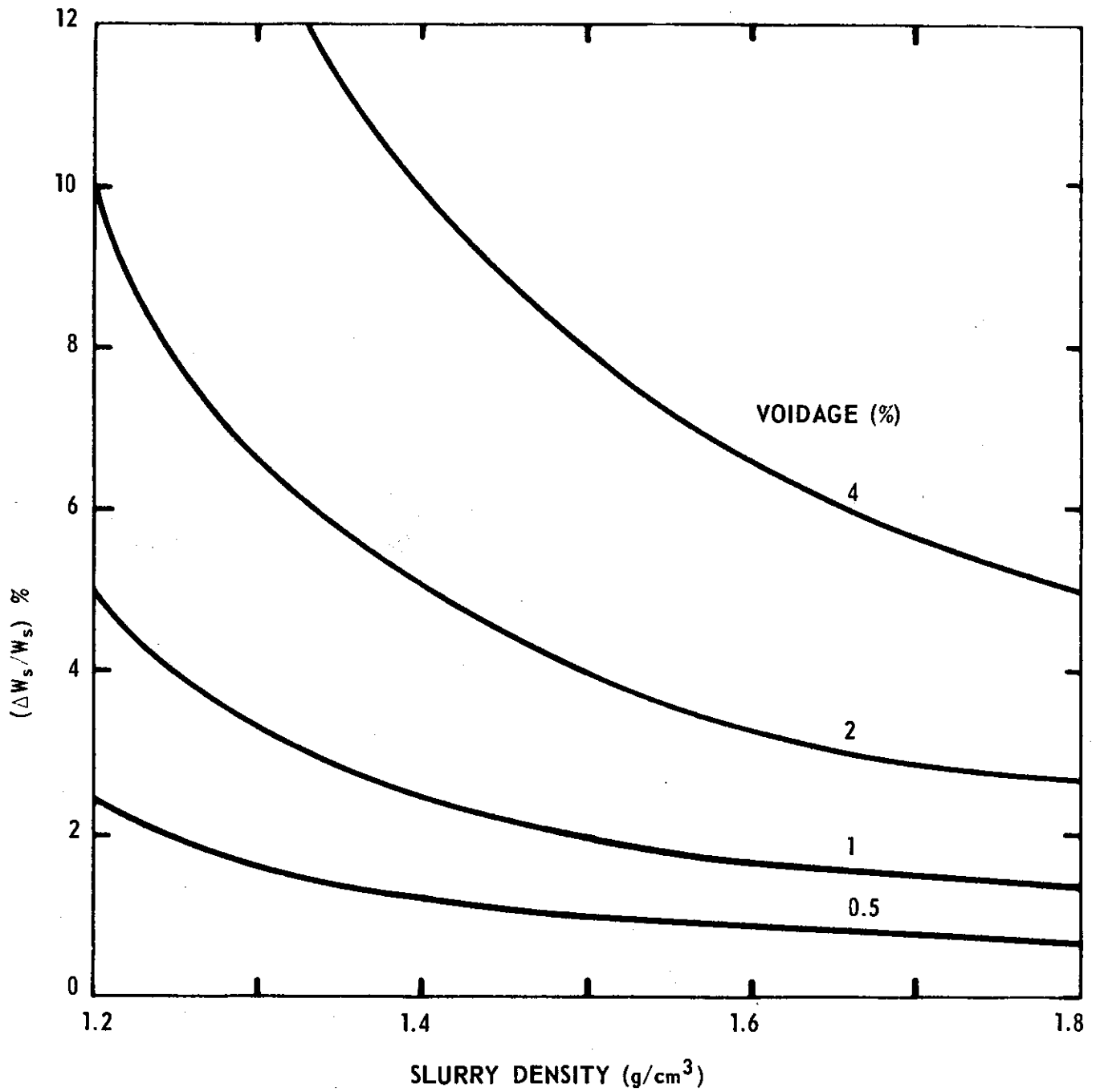


FIGURE 2. ERRORS IN WEIGHT FRACTION (W_s) OF SOLIDS IN SLURRY CAUSED BY CHANGES IN VOIDAGE IN THE SLURRY

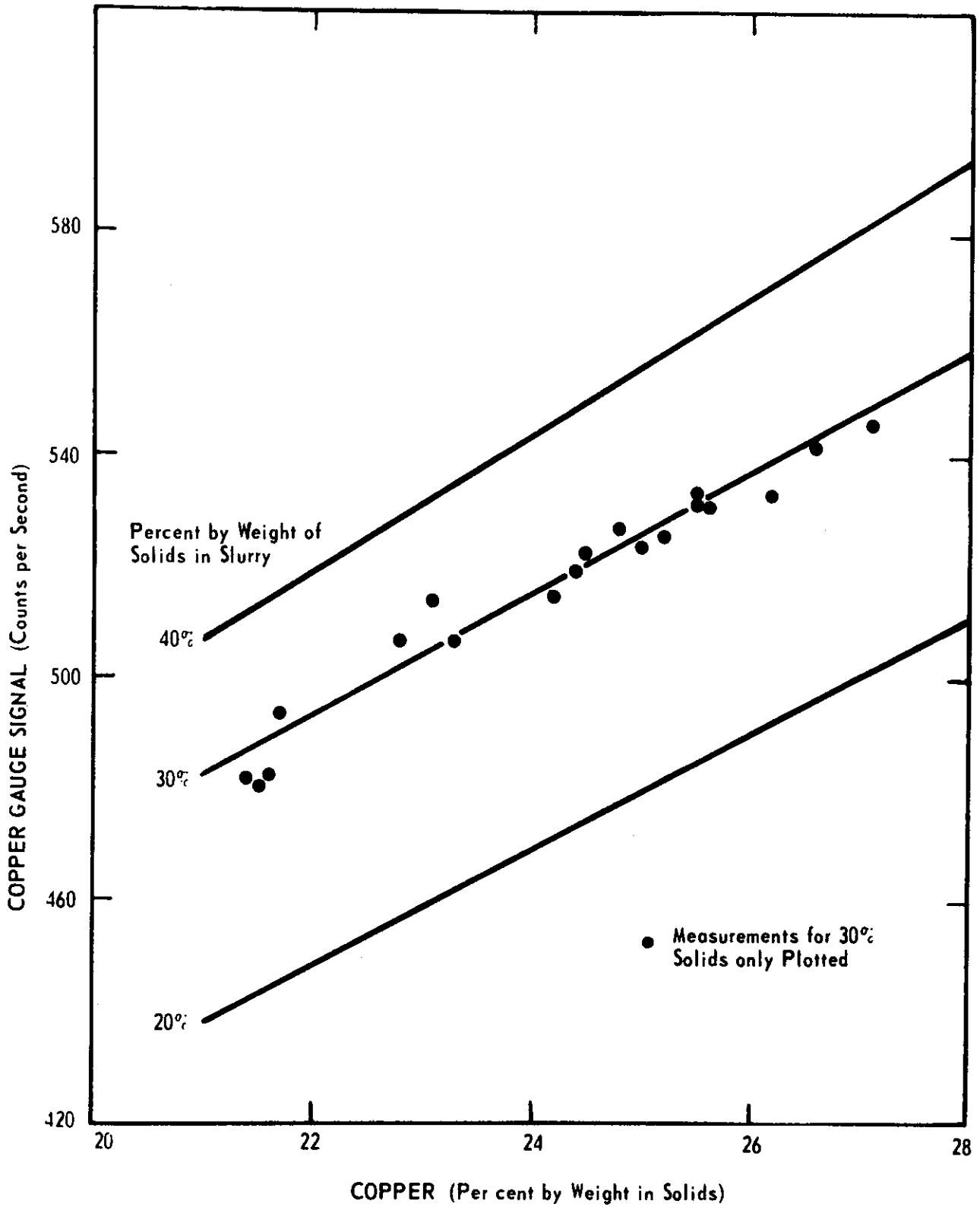


FIGURE 3. DETERMINATION OF COPPER IN COPPER CONCENTRATE SLURRIES

