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DEPARTMENT OF MINES AND TECHNICAL SURVEYS

OTTAWA

MINES BRANCH INVESTIGATION REPORT IR 63-101

**A RADIOTRACER TEST AT THE
NORANDA SMELTER,
JULY 15-18, 1963**

by

J. L. HORWOOD & H. P. DIBBS

MINERAL SCIENCES DIVISION

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J.L. Horwood and H.P. Dibbs*

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SUMMARY OF RESULTS

A radioactive tracer test was conducted at the smelter of Noranda Mines Ltd., Noranda, Quebec, July 15-18, 1963. Copper-64, a radioactive isotope, was introduced as cuprous oxide into a ladle of converter slag that was returned in the usual manner to a reverberatory furnace. The rather rapid decay of copper-64 to half its activity every 12.8 hours required careful precautions to ensure delivery of the copper source to the test site at the scheduled time; samples from the test were counted at Noranda.

Of principal interest were the relative proportions of the tracer material removed from the furnace in the form of slag and matte, and the residence times in the successive layers in the furnace from which the slag and matte were tapped. Seven per cent of the copper-64 removed from the furnace was found in the slag in measurable amounts during the first fourteen hours after injection--the most active sample in this group was taken at three hours. The balance of the activity was found in the matte; this reached a peak in 12 to 13 hours and was still measurable after 48 hours.

The almost complete recovery of the tracer material from the furnace was verified by calculating and adding the amount removed in each ladle during these operations; this total activity corresponded with the activity of the source measured at the time of injection. Additional experiments were subsequently conducted in Ottawa with known activities of copper-64 to determine the efficiency of the sample geometry and the counting efficiency of the scintillation counters.

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INTRODUCTION

In this country, the use of radiotracer methods is now well-established in a research laboratory where provision can be made for handling highly active materials under carefully controlled conditions. Trained personnel employ such procedures without hesitation in a tracer laboratory where equipment includes protective shielding, remote manipulating devices, filtered ventilation, health monitoring apparatus and sensitive, well-shielded counting equipment. Facilities are provided also for the storage and disposal of the radioactive material as required.

It should also be realized more widely that such techniques may very often be utilized in the field under normal operating conditions in large-scale plants. With suitable precautions, moderately active sources can be employed safely to provide information about the dynamics of a process in regard to factors such as residence times and flow patterns.

It has been stated that the copper smelting process in use today at Noranda has reached a high degree of efficiency and has not been changed greatly in the past forty years. However, more detailed knowledge concerning some factors in the process could result in a small, but economically significant improvement. At present the losses of copper in the slag are estimated to exceed \$2,000,000 annually.

At the request of the Noranda Research Centre, Montreal (1), who are making a long-term study of copper smelting, a radioactive tracer test was conducted at Noranda Mines Ltd., Noranda, Quebec, between July 15-18, 1963. The object of the test was to investigate the fate of copper, in the copper-containing converter slag, when the converter slag is returned to the reverberatory furnace. Radioactive copper was added to a ladle of converter slag and the progress and distribution of the active copper in the reverberatory furnace was followed by sampling the matte and slag at the regular tapping times of the furnace. A tracer test had been conducted previously at Noranda on these lines (2) but had been unsuccessful because of insufficient copper activity.

Note: The information in this report is presented in greater detail than is customary in the expectation that it will serve as reference material for future tests of a similar nature.

COPPER-64 ACTIVITY

Copper is present in the converter furnace slag primarily as cuprous oxide. It is convenient to irradiate copper in this form because oxygen does not become radioactive in the reactor and thus will not give rise to undesirable radioactive products. Allowing for appropriate dilution factors in the reverberatory furnace, calculation had indicated that a copper source of 250 millicuries in the complete furnace charge, should give adequate sensitivity for scintillation counting in a 4500 g sample of slag or matte.

Only one radioactive isotope of copper, copper-64 (half-life, 12.8 hours), was suitable for this test. It is produced by thermal neutron irradiation of copper, the cross section of copper-63 for activation being 4.5 barns. This relatively large cross section allows many curies of copper-64/g of copper to be produced in a high-flux reactor. A potential health hazard, however, exists in adding a few grams of highly radioactive Cu_2O powder to the converter ladle, in that accidental spillage could cause widespread contamination. For this reason it was decided to irradiate the cuprous oxide in sealed, lead tubes that could be added directly to the converter ladle. Lead was chosen as it undergoes negligible activation in a reactor and because it is chemically inert. Aluminum containers would also have been suitable from an induced activity viewpoint, but were rejected because of the possibility of aluminum reducing the cuprous oxide to copper at the temperature of the converter slag and hence invalidating the test as an investigation of the behaviour of cuprous oxide.

Cylinders of spectroscopically pure lead were prepared (approximate dimensions, O.D. = 7 mm, I.D. = 3.5 mm, L = 30 mm), filled with cuprous oxide powder, the open end of each crimped shut and then sealed further by melting with a soldering iron. The cuprous oxide was the best grade readily available (Fisher Scientific Reagent Grade) and was checked spectrographically to ensure that it contained no impurities that would become significantly activated during irradiation.

The short half-life of copper-64 is useful in a tracer test because it ensures that, after a few days decay, no possible health hazard can exist. However, the short half-life did impose transport problems to Noranda. In discussion with Commercial Products Division, A.E.C.L., the shipping schedule arranged, called for the source to be delivered by road from Chalk River to Ottawa, arriving 8:00 a.m. July 16, then to be transferred to Uplands to catch TCA flight #261 to Noranda at 9:40 a.m. As a precaution, in the event of transport failure, a second, more active source (Back-up sample) was to be brought to Ottawa at the same time for delivery to Noranda the next day, its activity being such that it would have 250 mc at noon, July 17.

The irradiations were planned for the N.R.X. reactor, but a few days before the test was due the reactor was shut-down for an indefinite period for emergency repairs. Through the co-operation of A.E.C.L., arrangements were made to perform the irradiations in the Research Physics pneumatic facility in N.R.U. The times of irradiation of the samples in N.R.U. are given below (see Appendix 1).

| | <u>Wt, Cu₂O (g)</u> | <u>Irradiation Time</u> | <u>Time of Test</u> |
|-------------------|--------------------------------|-------------------------|---------------------|
| Go-sample | 0.8 | 18 min | Noon, July 16 |
| Back-up sample | 2.0 | 28 min | Noon, July 17 |

The transport schedule remained the same as originally arranged.

COPPER-64 INJECTION

The easiest method of adding the copper-64 source to the converter slag would be to drop the lead capsule into the ladle from the converter platform. However, the slag itself is a poor conductor of heat and preliminary tests had shown that it was possible, with this method of addition, that the lead capsule could sink to the bottom of the ladle and be lost in the skull before satisfactory melting and dispersion of the activity had occurred.

The active copper was therefore added using the injection rod shown in Figure 1. This consisted of a short section of 2-inch standard iron pipe welded on to a long iron rod. The open ends of the pipe were covered with aluminum foil and the source placed in the pipe through a half-inch diameter hole. Immersion of the rod below the slag surface led to almost immediate melting of the thin aluminum foil. The rod was held so that the iron pipe was horizontal in the ladle and thus had ample time to reach temperature equilibrium. In the actual addition of the source, the ladle was approximately half-full of slag (by volume) and, after a few seconds immersion, the rod was vigorously shaken for about half a minute. Further slag was then added to fill the ladle and give mechanical mixing, the rod being held in such a way that it was "rinsed" by the stream of fresh slag.

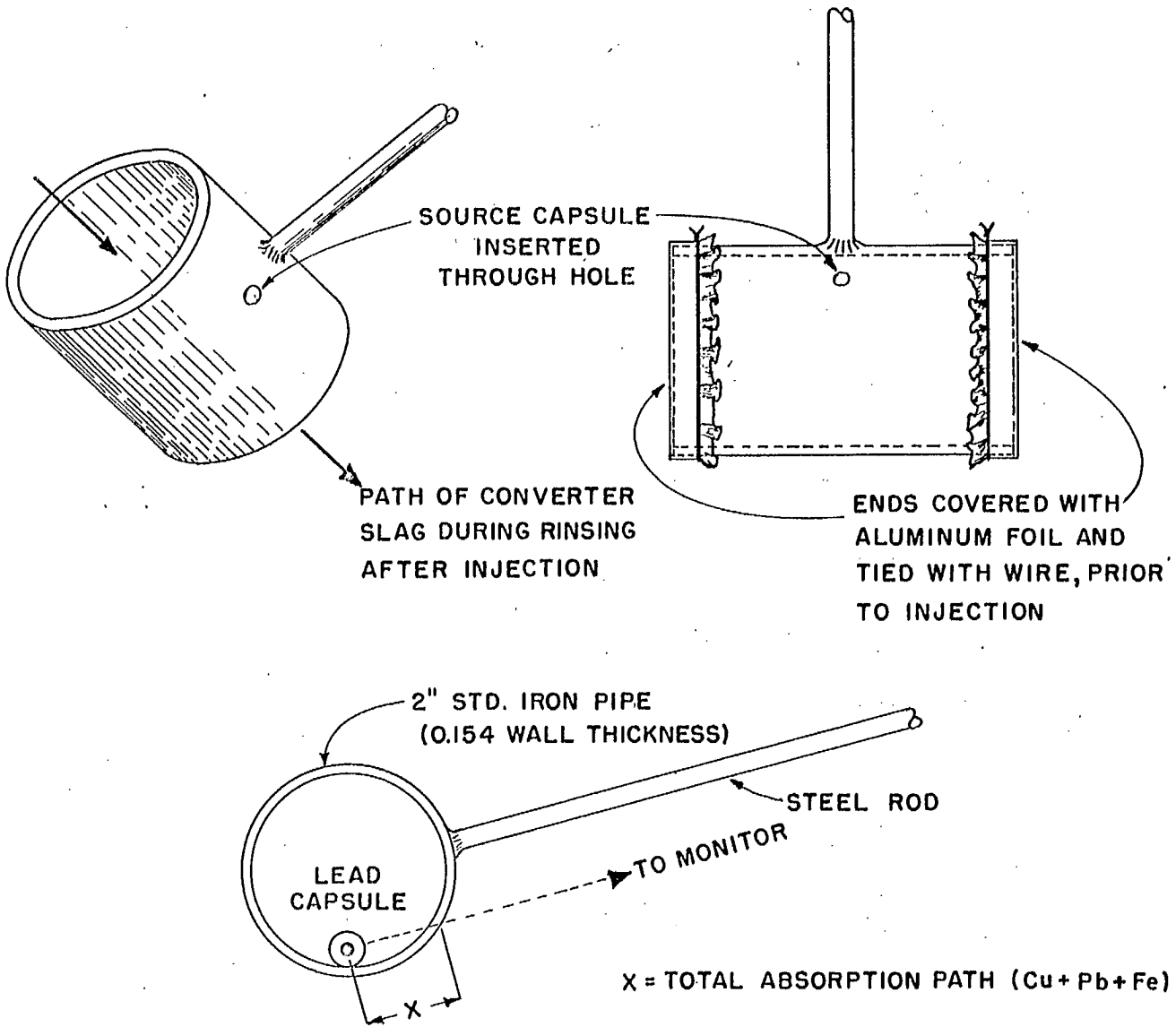
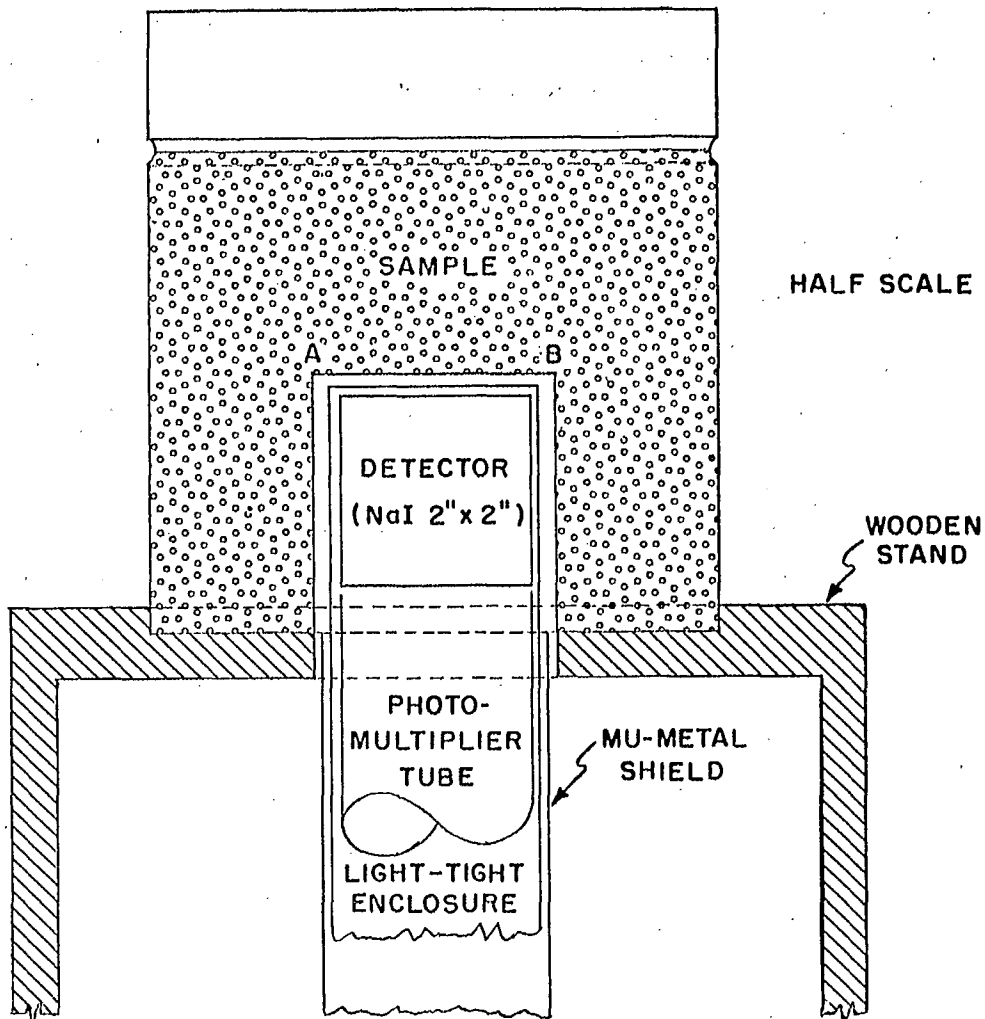


FIGURE I. THE INJECTION ROD

The copper-64 source arrived on schedule at Noranda and was brought to the platform of Number 6 converter. A temporary shielding wall of anode copper bars had been set-up on the platform for source manipulation. On opening the shipping flask two copper-64 sources were found. No explanation of the two sources was available at the time (see Appendix 1) as the Go-source should have been one only. However, the sources were monitored quickly and the one with the higher activity chosen for the test (the ratio of activities was very roughly 2:1). This source was placed in the injection rod and the field at one metre measured with an ionization chamber (calculations of the activity are shown in Appendix 2). The injection rod was then placed in the slag ladle as described. When the ladle had been filled with slag the rod was removed for monitoring and an air-cooled stainless steel probe immersed vertically in the ladle. This probe collected a sample of slag from the ladle and acted as a check on the activity distribution within the ladle. It was withdrawn after about one minute and placed on a sheet of galvanized iron and allowed to cool. The converter ladle was then taken and added to Number 3 reverberatory furnace at 12:50 p.m. (T₀).

During the addition of the copper-64, only personnel directly concerned with the test were allowed on the platform and all were issued with film badges. Also, an air filter sample was taken during the addition. This was counted immediately afterwards and showed no activity. The slag collected along the probe sample was monitored with a portable scintillation counter and showed a uniform count of about 10,000 cpm along its length (4 ft). The slag was then broken off and taken for crushing and scintillation counting. Monitoring of the injection probe showed a few thousand counts per minute around the iron pipe. As a safeguard, this was sawn off and thrown into the converter furnace.

The second copper-64 source was suitably labelled, then placed in a locked room until returned to A.E.C.L. for disposal.



NOTE: SMALLER PROBE SAMPLES
PLACED IN BEAKERS ON AB
(AND USING 1.5" x 1.5" CRYSTAL)

FIGURE 2. LARGE SAMPLE CONTAINER AND SCINTILLATION DETECTOR

SAMPLING AND COUNTING

Calculation of the possible division of radioactive copper between slag and matte indicated that relatively large samples of several thousand grams should be used in order to determine the activity of the samples during a counting period of a few minutes. The size of a sample is limited, however, by internal absorption of the radiation being measured and the rapidly decreasing probability that radiation from outer regions of the sample will intercept the detector as the solid angle is reduced.

The effective thickness of a sample can be increased greatly by using a dip counter whereby provision is made for the detector to operate within the sample rather than merely beside it. The form of dip counter adopted is shown in Figure 2. Ten sample containers having uniform dimensions were readily constructed using standard mass-produced tins. Tests indicated that the counting efficiency, and hence the sensitivity of the measurements, was increased by a factor of three over that obtained using the same large (105 oz) container without the insert configuration for the detector. (More detailed information concerning counting efficiency will be found in Appendix 3.)

A sample of four- to six thousand grams could be counted in these containers although this much was not always available. Two or three spoon samples were taken as each pot of slag or matte was poured. The material was spread out to cool in metal trays--a water spray helped to accelerate this. The samples were crushed and pulverized to an expected -20 mesh, although the use of new pulverizer plates resulted in a much more finely ground product. Separate machines were used for slag and matte and, as a further precaution, inert material was passed through after every sample.

Residual heat in some of the samples was somewhat of a hindrance. The gain of the photomultiplier tube used in the detector is affected by a temperature change of a few degrees; a hot sample, therefore, must not be placed around the detector (a minor disadvantage of the dip counter). Air cooling at an open window helped to reduce the delay; however, if the work is repeated, it is recommended that a rack be assembled with fans to blow air up beneath a number of samples simultaneously.

The sample containers were marked numerically as an additional means of sample identification. Although ten containers were used, twice that number would have been very useful; several were needed for samples temporarily stored for cooling, for weighing, etc.

The measured count-rate from a sample could be increased considerably by packing--with a very loosely packed sample by as much as 50% in subsequent tests. The several stages of handling are believed to have promoted uniform consolidation. Although this could possibly be a source of error in the comparison of a few samples this effect averaged out over the whole test.

Depth samples were frozen on to probes lowered into the furnace through selected holes in the roof lining. It had been hoped to scan these probes along their length with a small portable scintillation counter. Insufficient activity was available for this counter to give a useful measurement; samples were therefore removed from separate zones of each probe and crushed for counting in the analytical room. As only a limited amount of material was available in these samples, (100 to 500 grams) they were counted in 250 ml beakers.

The effect of variation in mass was subsequently determined and corrections applied for the slag, matte and probe samples.

Two complete laboratory scintillation counting systems were transported to Noranda along with voltage stabilizers, test equipment, spare parts, scintillation and ionization-chamber type portable monitors, air-sampling equipment and tools for manipulating the radioactive source. These are listed in Appendix 4. The response of the counters to the required gamma-ray energies was adjusted using barium-133 (292 keV, 360 keV) and caesium-137 (662 keV) as reference standards. A weak radium source ($\sim 0.1 \mu\text{c}$) suitably mounted as a sensitivity standard would have been a useful addition to check for indications of drift.

A Nuclear-Chicago model 132B, single-channel pulse-height analyser was used to measure the activity of the matte and slag samples. It was operated at maximum channel width to count between 460 and 560 keV, thereby encompassing the principal emission from copper-64 at 510 keV; scattered radiation and background radiation outside these energy limits were rejected. A two-inch by two-inch cylindrical sodium iodide crystal

was used in the associated detector. The count from a sample was recorded* along with the time of sampling, the weight, time of counting and the duration of the counting period. All results were subsequently corrected for radioactive decay to T_0 , 12:50 p.m. July 16, the time at which the source was injected.

A Beckman/Berkeley** model 2101 scaler was used in the second scintillation counting system to count the probe samples. A somewhat smaller crystal (1.5 in. diameter by 1.5 in. thick) was available for this detector. The scaler operated in an integral mode by totalling the counts above the selected threshold energy, near 350 keV. The samples, contained in 250 ml beakers, were placed on the upper face of the insert tin of one of the standard, large-sample containers. The relative counts from the same sample counted in each system were very nearly in the ratio of 4 to 1 (dip-counter with 2 in. x 2 in. crystal to second system with 250 ml beakers).

Each detector was enclosed in a lead shield four inches thick on the sides and top; a hinged door was provided in the front. The shielding at the bottom consisted of two $1\frac{1}{4}$ in. copper anodes mounted beneath the bench top; the latter was covered with $\frac{1}{4}$ in. steel plate. The background radiation originating externally was reduced by the shields to a low level. Normal (unspiked) slag and matte samples were crushed for background determinations. If they had been completely inert, they would have increased the shielding and lowered the previous (instrumental) background level. Instead, they were found to produce appreciable natural radiation, later identified as potassium-40 and daughter elements of uranium, particularly in the case of the large slag samples. This portion of the background continued to increase for several days after the sample had been collected; in the molten state radon, a decay product in the uranium series, was thoroughly de-emanated and the short-lived decay products of radon were removed. Radon, which has a half-life of 3.8 days, built up in the sample after cooling, and the decay products such as bismuth-214, that are responsible for most of the gamma-ray activity, increased towards an equilibrium value.

* The scaling system of the model 132B analyser, based on powers of 2, caused an appreciable delay in readout time.

** The two counting systems are later referred to as the N/C and B/B systems.

Typical background readings from samples containing no active copper are shown in Table 1. They show a marked difference between the slag and matte. Freshly prepared samples were later examined in the Mines Branch gamma-ray spectrometer. The spectra are shown in Figure 3. Reverberatory slag and converter slag both produced spectra; however, the activity of the matte was too low to give a significant spectrum. Potassium-40 produces a definite peak in the spectra at 1460 keV; its contribution is approximately the same in each type of slag and remains constant although the peak appears to grow somewhat from an increasing contribution from bismuth-214. The latter was identified by several peaks; the peak centred at 609 keV was responsible for most of the growth in background activity. It is evident that the background count applied to a sample whose active copper content is very low, must be carefully selected; it must originate from similar material containing no copper-64 but of similar age since solidification. Under the conditions of this test, a significant change in the background count from a sample containing none of the radioactive tracer must not be taken as evidence that the sensitivity of the counter has changed.

TABLE 1

Background Count-Rates from Normal Slag and Matte

| Date July 1963 | Time | Background from Materials Containing no Cu-64 (counts per minute) | | | |
|----------------------|-------|--|-------------|-------|-------------|
| | | Matte | | Slag | |
| | | Total | Sample only | Total | Sample only |
| 16 | 13:50 | 18.4* | -- | 18.4* | -- |
| | | | | 66.8 | 48.4 |
| | 14:10 | 24.6 | 6.2 | | |
| | 15:05 | 25.0 | 6.6 | | |
| | 18:15 | 25.8 | 7.4 | | |
| | 18:30 | | | 67.0 | 48.6 |
| 17 | 20:40 | 25.5 | 7.1 | | |
| | 07:50 | | | 85.1 | 66.7 |
| | 08:50 | 28.5 | 10.1 | | |
| | 14:50 | 27.5 | 9.1 | | |
| 18 | 20:55 | | | 105.0 | 86.6 |
| | 08:15 | | | 101.0 | 82.6 |
| | 11:00 | | | 106.0 | 87.6 |

* No sample = 18.4 counts per minute, obtained using an empty sample container.

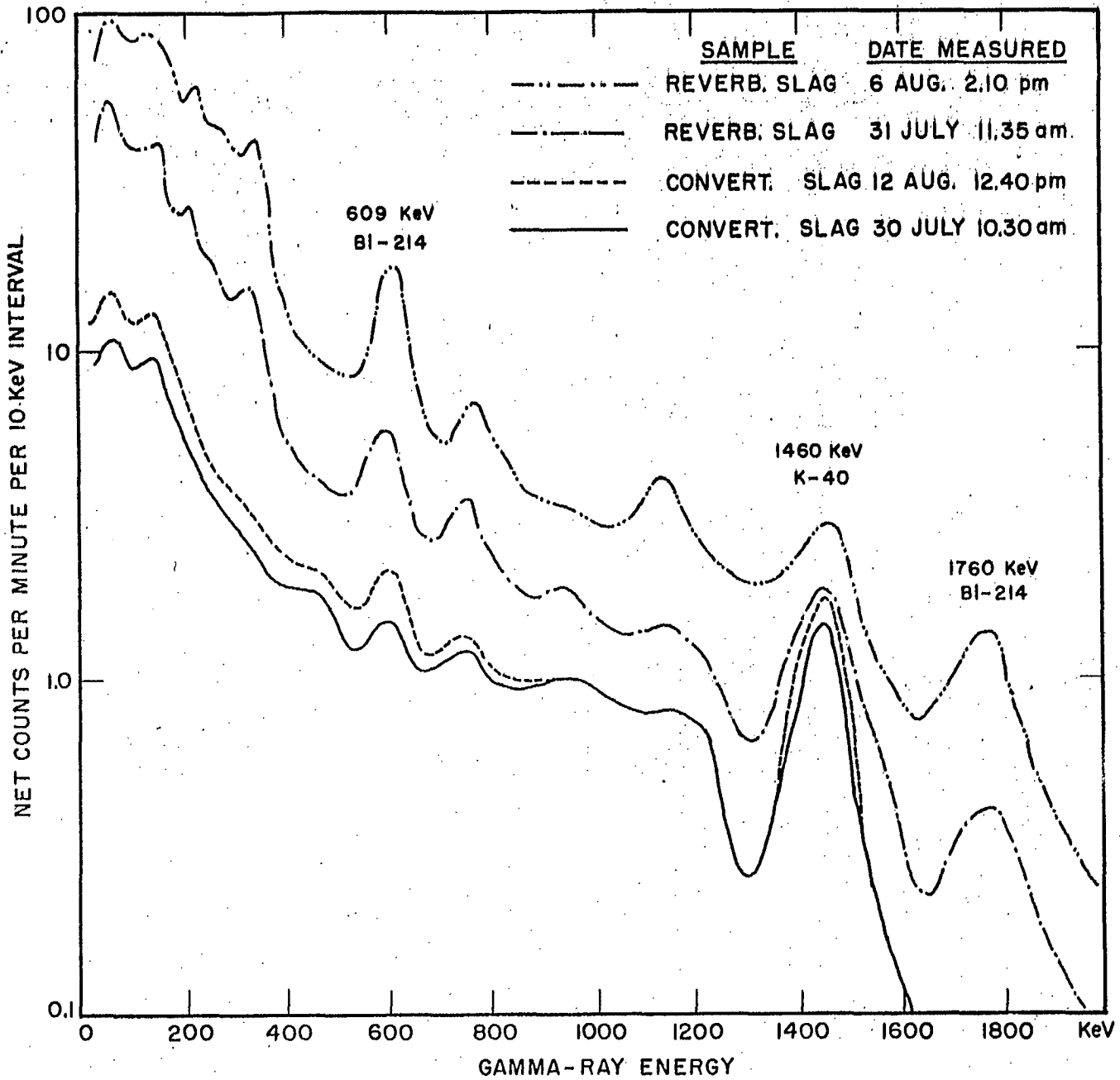


FIGURE 3. GAMMA-RAY SPECTRA OF NATURAL BACKGROUND ACTIVITY IN REVERBERATORY AND CONVERTER SLAGS

RESULTS

Three hundred and five samples were pulverized, weighed and received for counting during the forty-eight hours following injection of the copper-64. The rapid decay of the copper-64 required that all be counted promptly; consequently, the counting period of most samples was restricted to between two and five minutes. All samples were corrected for decay between injection time and the time that each was counted, using the graph in Figure 4.

Weight correction curves were determined by a later series of tests at the Mines Branch using aliquots of both slag and matte that had been spiked with copper-64. Further details concerning the preparation of this material used for calibration and the development of weight correction curves are given in Appendix 5.

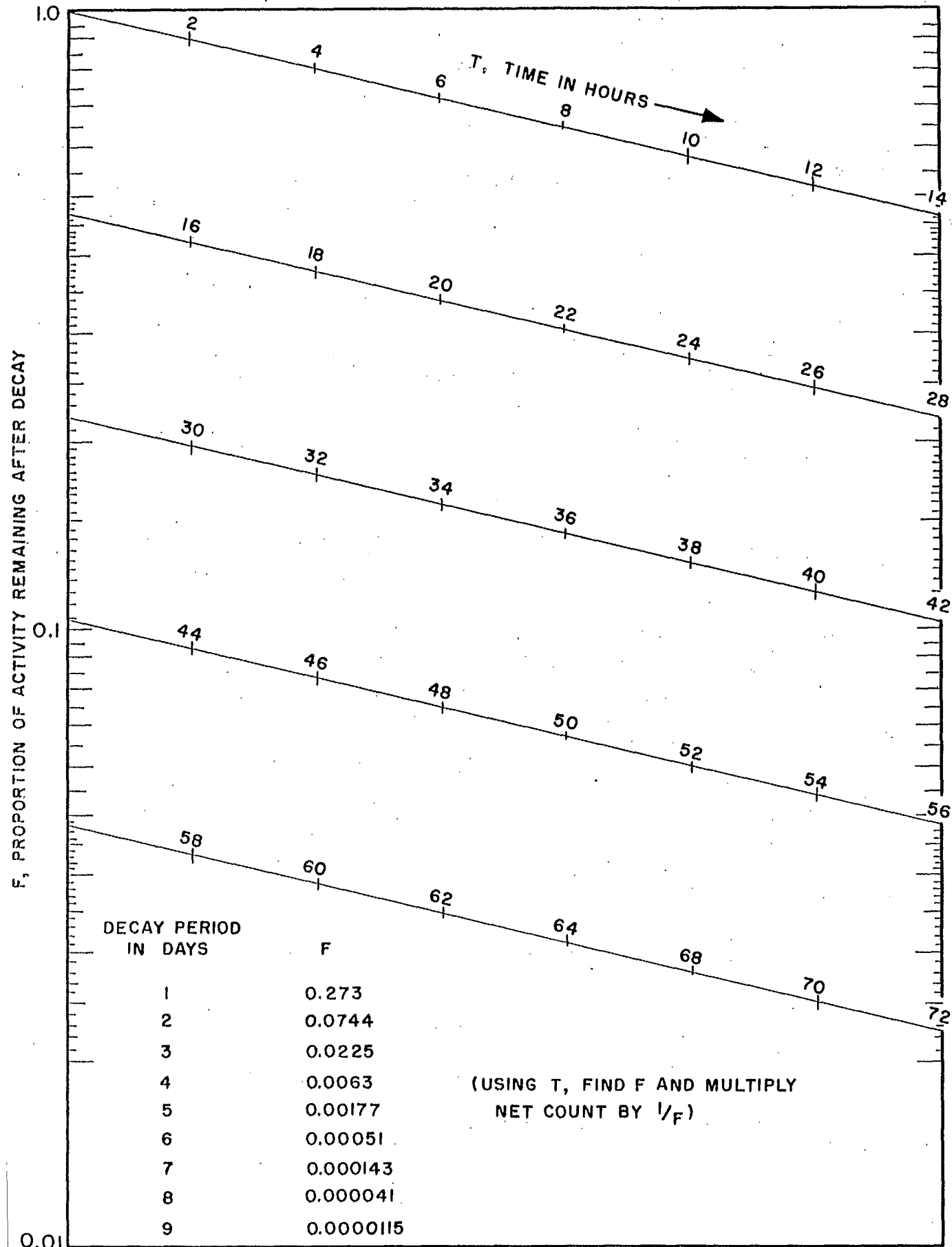
Tests using the spiked material confirmed the sensitivity relationship between the two counting systems established at Noranda where slag sample, S-4, and matte sample, M-32 were counted in each system. Comparative values, corrected for weight, are shown in the following table:

TABLE 2

Ratio of Relative Counting Efficiencies of the Two Systems

| Sample | Date and Location | Count Rate N/C : B/B Counting Systems |
|-------------------|-------------------|--|
| S-4 | 16 July Noranda | 3.84:1 |
| M-32 | 17 July Noranda | 4.09:1 |
| Matte (spiked) | 3 Sept. Ottawa | 4.12:1 |

An average factor of 4:1 is taken to express the relative sensitivity obtained with the two counting systems; that is, counting a standard weight of material in the large dip container and then counting 500 grams of the same sample in a 250 ml beaker using the other (B/B) counting system. The results in Table 2 also indicate that consistent sensitivity was maintained in the counters between Noranda and Ottawa.



Weight correction curves are shown for the tapped samples, slag and matte, in Figure 5 using the N/C counting system. As would be expected, the count-rate from the lower density slag is higher than that from the same volume of matte; equal volumes of the two materials contained equal amounts of copper-64. These curves were used for the most part in calibration procedures to determine absolute sample activities and to calculate the total source intensity. The weight variations in this series of samples were not large, and had little effect on the curves showing residence times; however, the average weight of the matte samples decreased somewhat throughout the test; correction for these variations extended the tail of the matte curve slightly.

The weight correction curve for the depth probe samples is shown in Figure 6. This correction is very important for this series of samples which varied in weight by as much as sixfold. All samples were corrected to a standard value of 500 grams. No distinction was made between slag and matte material in this case because the curves did not differ greatly relative to the expected experimental error; the mean value between the slag and matte curves was therefore plotted in this figure.

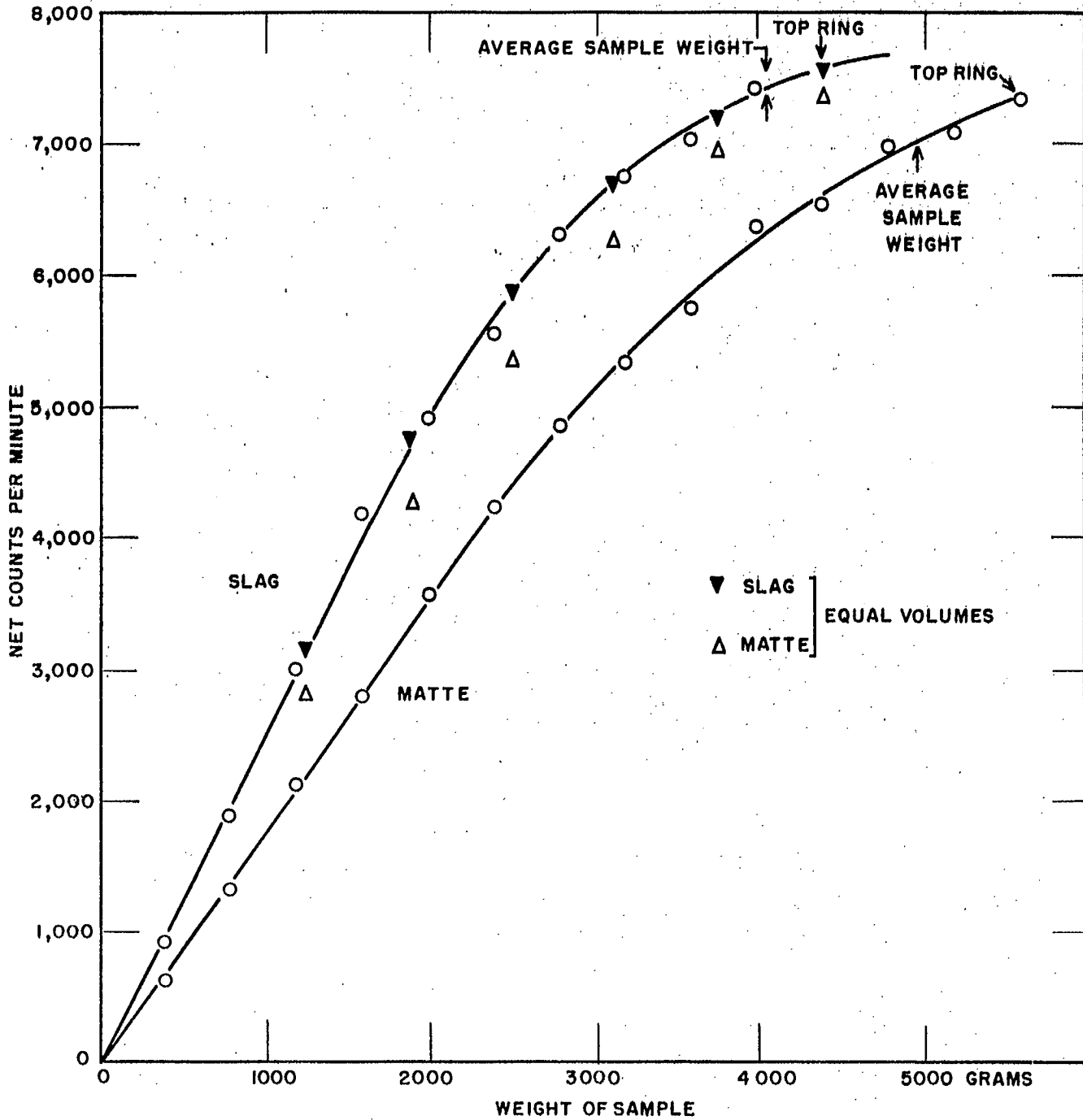


FIGURE 5. VARIATION OF COUNT-RATE OF SPIKED MATTE AND SLAG SAMPLES WITH WEIGHT

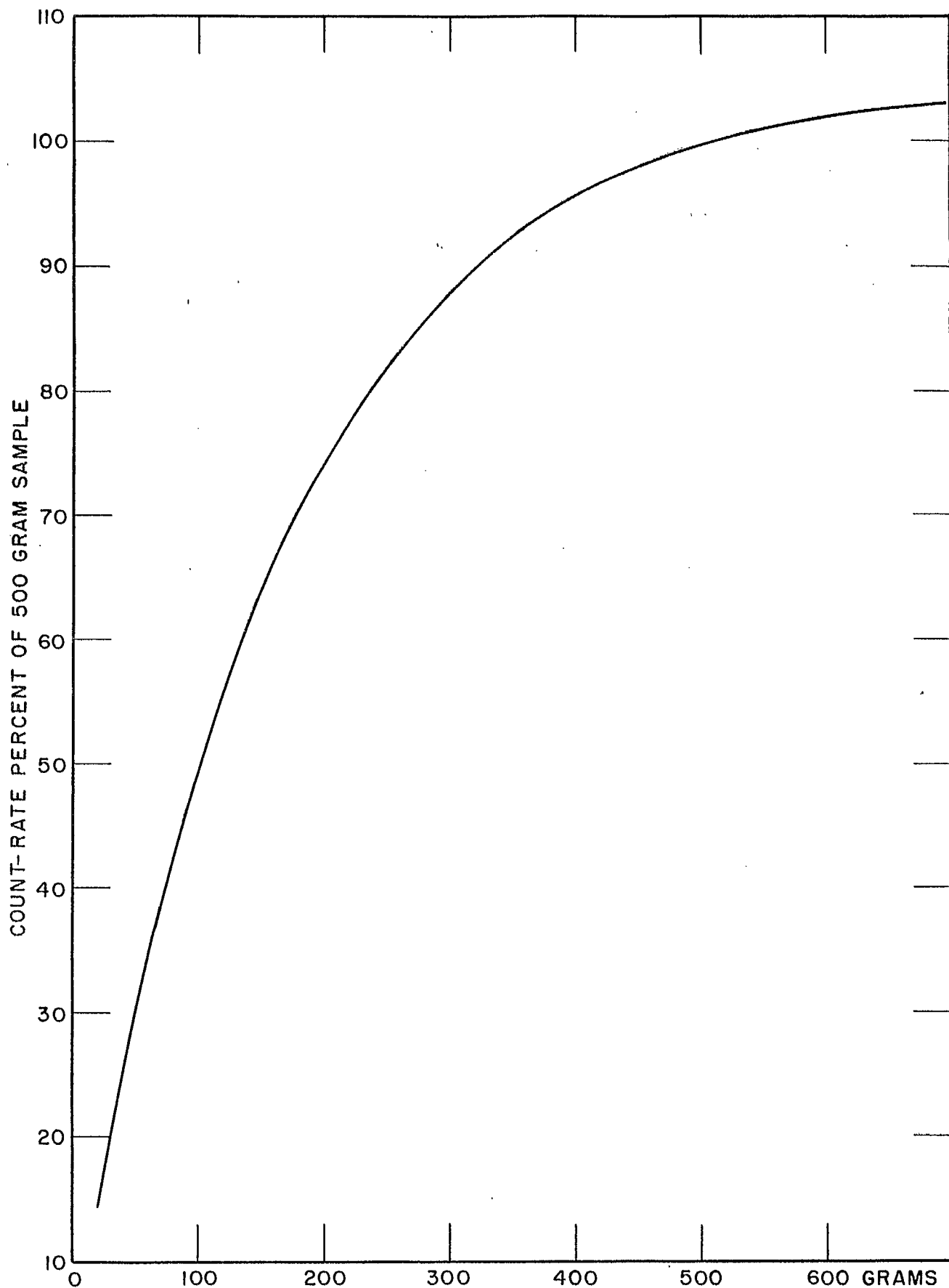


FIGURE 6. WEIGHT CORRECTION CURVE FOR THE DEPTH PROBE SAMPLES
(250 ML BEAKERS WITH B/B COUNTING SYSTEM)

(a) Tapped Samples

Corrected net activities for the slag samples are given in Table 3, and for the matte samples in Table 4. Graphs are shown in Figures 7 and 8 for each in linear and in semi-logarithmic form. The linear form is useful in visualizing comparative activities; the total area under the linear curve represents the total activity, slag or matte. The semi-logarithmic form displays more readily the initial and final readings which are of low activity, and enables the curve showing the activities actually measured without correction for decay to be displayed. The activity shown in the matte during the first two hours is believed not to be real but rather due to contamination of the crusher and pulverizer by the converter slag sample whose activity was 1,500,000 counts per minute. The tail of the curve for the matte samples extended over several days; after the first forty-eight hours, values were obtained by extrapolation assuming an exponential relationship (3).

The method for determining the total activities tapped off in the slag and matte phases is described in Appendix 6. The total activity in the slag was calculated to be 46.9 millicuries and in the matte 615 millicuries; the total, 662 millicuries. The activity in the slag represented 7.1 per cent of the total. The approximate total activity measured with the ionization chamber just prior to injection was 643 millicuries (see Appendix 2).

(b) Depth Probe Samples

The readings obtained from these samples were reduced to net counts per minute by subtracting a value for the background which varied down the probe in accordance with the change from the high natural background due to potassium and traces of radium decay products in the upper slag layers, down to a lower background value for the matte material. Corrections for weight variations and decay after injection time were applied; the resulting activities are shown in Table 5. The expressions 1a, 1b, etc. represent 5-inch sections of the coating on probe No. 1, "a" being the upper section, "b" the next lower, etc.

TABLE 3

Corrected Net Activities of Tapped Slag Samples

| Slag Samples (net activities corrected for decay to T_0 except where noted)* | | | | | |
|--|------------------------------------|-------------------|------------|------------------------------------|-------------------|
| Sample No. | Time of Sampling Hours after T_0 | Counts per Minute | Sample No. | Time of Sampling Hours after T_0 | Counts per Minute |
| S-1 | 0.92 | 97 | S-16 | 12.00 | 122 |
| S-2 | 1.25 | 4700 | S-17 | 12.17 | 130 |
| S-3 | 1.33 | 8100 | S-18 | 13.08 | 140 |
| S-4 | 2.92 | 12,300 | S-19 | 13.33 | 74 |
| S-5 | 3.00 | 12,100 | S-20 | -- | -- |
| S-6 | 4.00 | 10,050 | S-21 | 14.00 | 94** |
| S-7 | 4.17 | 8300 | S-22 | 15.17 | 79 |
| S-8 | 4.92 | 6150 | S-23 | 15.67 | 79 |
| S-9 | 5.00 | 3900 | S-24 | 16.92 | 108 |
| S-10 | 7.25 | 1530 | S-25 | 18.33 | 81 |
| S-11 | 7.33 | 1270 | S-26 | 18.50 | 79 |
| S-12 | 8.08 | 600 | S-27 | 44.92 | 50*** |
| S-13 | 8.17 | 1000 | S-28 | 45.08 | 51 |
| S-14 | 9.67 | 355 | S-29 | -- | -- |
| S-15 | 10.67 | 544 | S-30 | 46.17 | 52 |

* No weight corrections applied to Table 3.

** Samples S-21 to S-30 are gross activities at time of counting. They included a background count which was 68 to 90 c/min for slag material.

*** Samples S-27 to S-30 were tapped after the furnace was blown. Their low activities indicate the mixing of lower (natural) activity material with the slag.

TABLE 4

Corrected Net Activities of Tapped Matte Samples

| Matte Samples (net activities corrected for decay to T_0 , and for weight variation to 5000 g) | | | | | |
|--|------------------------------------|-------------------|------------|------------------------------------|-------------------|
| Sample No. | Time of Sampling Hours after T_0 | Counts per Minute | Sample No. | Time of Sampling Hours after T_0 | Counts per Minute |
| M-1 | 0.58 | 908 | M-41 | 22.83 | 7457 |
| M-2 | 1.00 | 126 | M-42 | 23.33 | 11,705 |
| M-3 | 1.42 | 72 | M-43 | 23.75 | 11,446 |
| M-4 | 1.83 | 49 | M-44 | 24.25 | 12,222 |
| M-5 | 2.25 | 41 | M-45 | 24.75 | 7794 |
| M-6 | 2.67 | 231 | M-46 | 25.17 | 7021 |
| M-7 | 3.00 | 616 | M-47 | 25.67 | 6094 |
| M-8 | 3.67 | 770 | M-48 | 26.17 | 7570 |
| M-9 | 4.17 | 1107 | M-49 | 26.58 | 5819 |
| M-10 | 5.00 | 1396 | M-50 | 27.25 | 5882 |
| M-11 | 5.50 | 1830 | M-51 | 27.83 | 6898 |
| M-12 | 6.00 | 3137 | M-52 | 28.58 | 5098 |
| M-13 | 6.42 | 3279 | M-53 | 29.17 | 6244 |
| M-14 | 6.83 | 4135 | M-54 | 29.92 | 4680 |
| M-15 | 7.25 | 6606 | M-55 | 30.75 | 6535 |
| M-16 | 7.67 | 6244 | M-56 | 31.75 | 5357 |
| M-17 | 8.00 | 6876 | M-57 | 32.83 | 6064 |
| M-18 | 8.50 | 6702 | M-58 | 33.42 | 7560 |
| M-19 | 8.92 | 8181 | M-59 | 34.33 | 5897 |
| M-20 | 9.33 | 10,100 | M-60 | 35.67 | 4391 |
| M-21 | 9.92 | 12,524 | M-61 | 36.75 | 5556 |
| M-22 | 10.33 | 13,827 | M-62 | 37.33 | 5668 |
| M-23 | 10.75 | 15,122 | M-63 | 37.83 | 5556 |
| M-24 | 11.42 | 11,397 | M-64 | 38.17 | 4255 |
| M-25 | 12.50 | 11,811 | M-65 | 38.50 | 5179 |
| M-26 | 13.17 | 12,427 | M-66 | 38.92 | 3313 |
| M-27 | 13.83 | 13,506 | M-67 | 39.33 | 4620 |
| M-28 | 14.17 | 12,066 | M-68 | 39.75 | 5060 |
| M-29 | 14.67 | 11,408 | M-69 | 40.17 | 5178 |
| M-30 | 15.00 | 13,178 | M-70 | 40.83 | 5457 |
| M-31 | 15.50 | 10,828 | M-71 | 41.42 | 4865 |
| M-32 | 15.83 | 11,919 | M-72 | 42.42 | 4700 |
| M-33 | 16.17 | 11,692 | M-73 | 43.58 | 5274 |
| M-34 | 17.00 | 10,136 | M-74 | 44.17 | 4370 |
| M-35 | 17.33 | 12,176 | M-75 | 44.58 | 3715 |
| M-36 | 17.67 | 10,388 | M-76 | 44.92 | 4307 |
| M-37 | 18.00 | 14,521 | M-77 | 45.83 | 2810 |
| M-38 | 18.67 | 14,634 | M-78 | | |
| M-39 | 22.08 | 7,245 | M-79 | 46.83 | 1947 |
| M-40 | 22.50 | 10,200 | | | |

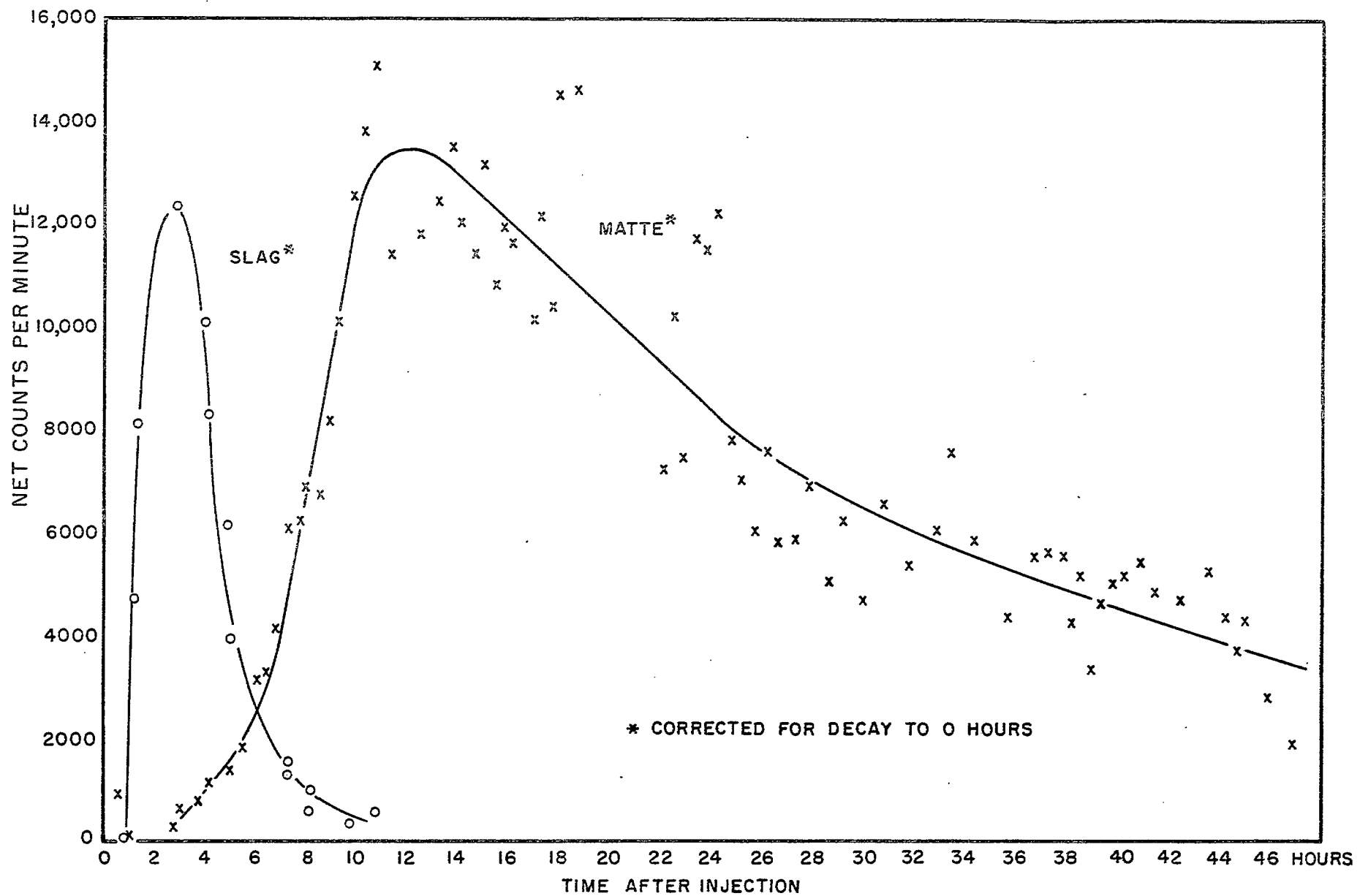


FIGURE 7. ACTIVITIES OF TAPPED SAMPLES

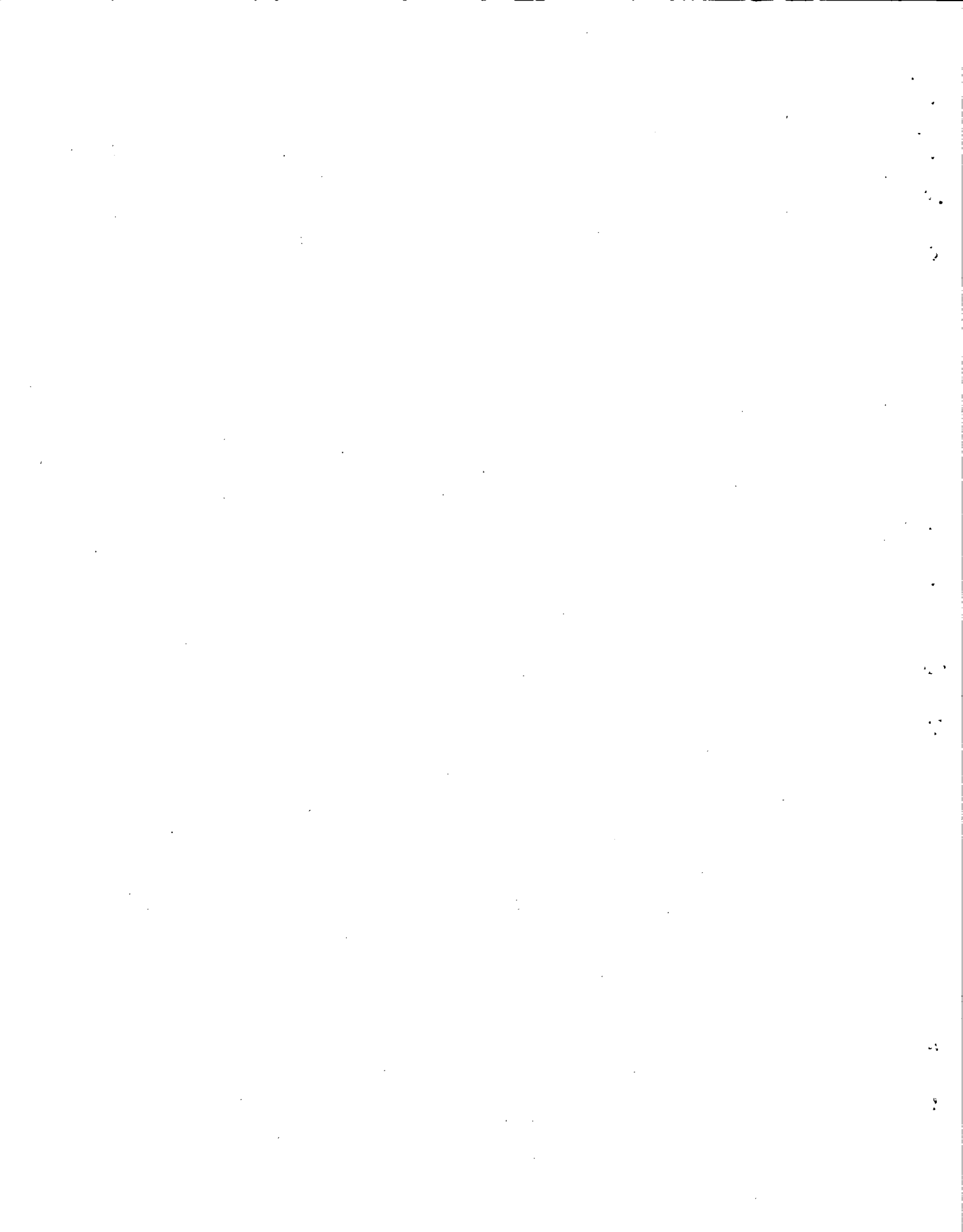


TABLE 5

Depth Probe Samples - Net counts per minute, corrected for decay to T₀ and for weight variation to 500 grams

| | No. 1 | No. 2 | No. 3 | No. 4 | No. 5 | No. 7 | No. 8 | No. 9 | No. 10 |
|---|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| a | 3598 | 3539 | 3027 | }1777 | 4086 | 653 | 1983 | 2160 | 2461 |
| b | 3558 | 4176 | 3238 | | 4249 | 160 | 2127 | 1978 | 2125 |
| c | 3312 | 4310 | 4145 | }1656 | 4407 | | 2363 | 1837 | 2008 |
| d | 3362 | 2368 | 6990 | | 2967 | | 2082 | 1476 | 1723 |
| e | 3256 | 2070 | | }2141 | 2751 | | 1633 | 1477 | 1535 |
| f | 3545 | 3007 | | | 2689 | | 1295 | 1329 | 1561 |
| g | 3493 | 3627? | | }1991 | 2077 | | 1549 | 1323 | 2016 |
| h | 3069 | 3469? | | | 2380 | | 1402 | 1316 | 2035 |
| i | | | | }1610 | | | 1728 | 1382 | 1660 |
| j | | | | | | | 1294 | 1119 | 1608 |
| k | | | | | | | 933 | | 1425 |
| | No. 11 | No. 12 | No. 13 | No. 14 | No. 15 | No. 17 | No. 18 | No. 19 | No. 20 |
| a | 546 | 429 | 425 | 454 | 282 | 770 | 854 | 623 | 584 |
| b | 1709 | 988 | 481 | 452 | 858 | 484 | 504 | 762 | 264 |
| c | 1072 | 397 | 603 | 528 | 638 | 432 | 248 | 573 | 269 |
| d | -- | 133 | 550 | 871 | 567 | 326 | 280 | 331 | 232 |
| e | | | 648 | 1267 | 433 | 291 | 172 | 252 | 163 |
| f | | | 849 | 2036 | 392 | 265 | 225 | 262 | 355 |
| g | | | 754 | 1601 | 383 | 242 | 218 | 180 | 555 |
| h | | | 776 | 989 | 358 | 209 | 346 | 287 | 556 |
| i | | | 1154 | 1142 | 391 | | 397 | 116 | 372 |
| j | | | | | 289 | | | | |
| k | | | | | 170? | | | | |
| | No. 21 | No. 22 | No. 23 | No. 24 | No. 25 | No. 26 | | | |
| a | 419 | 194 | 155 | 177 | 209 | -- | | | |
| b | 211 | 239 | 123 | 138 | 172 | -- | | | |
| c | 141 | 289 | 129 | 32 | 176 | -- | | | |
| d | 202 | 116 | 109 | 71 | 192 | -- | | | |
| e | 185 | 131 | 127 | | 204 | 50? | | | |
| f | 193 | 74 | 132 | | 256 | 118? | | | |
| g | 223 | | 182 | | 221 | 125? | | | |
| h | 190 | | | | 208 | 150? | | | |
| i | 162 | | } 294 | | 173 | 102? | | | |
| j | 144 | | | | | 217 | 113? | | |
| k | | | 256 | | 174 | 121? | | | |
| l | | | | | 165 | 178? | | | |
| m | | | | | 167 | 61? | | | |
| n | | | | | 185 | } 84? | | | |
| o | | | | | 159 | | | | |
| p | | | | | 160 | 110? | | | |

No. 6 and No. 16--no sample obtained.

? result may not be accurate because of the low activity.

Before plotting the depth probe readings, coordinates were assigned to each hole as shown in Table 6. The prefixed letter S, C, or N, indicates whether a hole is in the southern, central, or northern group; the figure following expresses the west-to-east position as an approximate percentage of the total travel through the furnace in that direction. The latter is plotted as the abscissa in Figure 9 with the time interval between injection and the time the probe sample was taken as the ordinate. Attached to each probe position in the figure is a histogram indicating the variation in activity down the probe for each interval of 5 inches. In the right hand column is a further histogram relating the corresponding activities for the tapped matte and slag samples using the previously mentioned factor of four and the same scale of activity in terms of counts per minute per centimetre.

(c) Converter Slag - Depth Probe Sample P-1

This sample was scanned using a portable scintillation counter before removal from the probe and was found to indicate uniform activity along its entire length. The sample was crushed for counting in the large sample container but was too active until two days had elapsed. Results obtained from counting this sample are given in Table 7. The last four readings do not agree well because of magnetic effects, from the sample and its iron container, on the photomultiplier tube in the counter. The problem of magnetic interaction was eliminated in Ottawa by placing a mu-metal shield around the photomultiplier tube. A steel plate below the counter, such as that used as a bench top at Noranda, also eliminated the effect.

TABLE 6

Depth Probes in No. 3 Reverberatory Furnace

| Probe Number | Time p.m. 16 July 1963 | Time After T ₀ (hrs:min) | Hole Designation | Location of Holes, 0 - input 100 - output | | |
|--------------|---------------------------|---|------------------|--|--------|-------|
| | | | | South | Centre | North |
| | 12:50 | 0 | | | | |
| 1 | 1:50 | 1:0 | A | | C7 | |
| 2 | 1:50 | 1:0 | B | | C16 | |
| 3 | 2:20 | 1:30 | C | | C29 | |
| 4 | 2:32 | 1:42 | F | S10 | | |
| 5 | 2:39 | 1:49 | D | | C42 | |
| 6 | 3:03 | | J | | | |
| 7 | 3:10 | 2:20 | Q | | C68 | |
| 8 | 4:11 | 3:21 | K | | | N63 |
| 9 | 4:17 | 3:27 | T | | C90 | |
| 10 | 4:40 | 3:50 | E | | C55 | |
| 11 | 4:44 | 3:54 | W | | C96 | |
| 12 | 5:04 | 4:14 | V | | C94 | |
| 13 | 5:08 | 4:18 | H | S42 | | |
| 14 | 5:34 | 4:44 | X | | | N42 |
| 15 | 5:42 | 4:52 | T | | C90 | |
| 16 | 6:49 | | L | | | |
| 17 | 6:52 | 6:02 | O | | | N84 |
| 18 | 7:04 | 6:14 | N | S74 | | |
| 19 | 7:29 | 6:39 | P | S84 | | |
| 20 | 7:57 | 7:07 | M | | | N74 |
| 21 | 8:16 | 7:26 | Q | | C68 | |
| 22 | 8:41 | 7:51 | V | | C94 | |
| - | 9:23 | | G | | | |
| 23 | 10:02 | 9:12 | G | | | N21 |
| 24 | 10:27 | 9:37 | V | | C94 | |
| 25 | 11:32 | 10:42 | Q | | C68 | |
| 26 | | 27:10 | | | | |

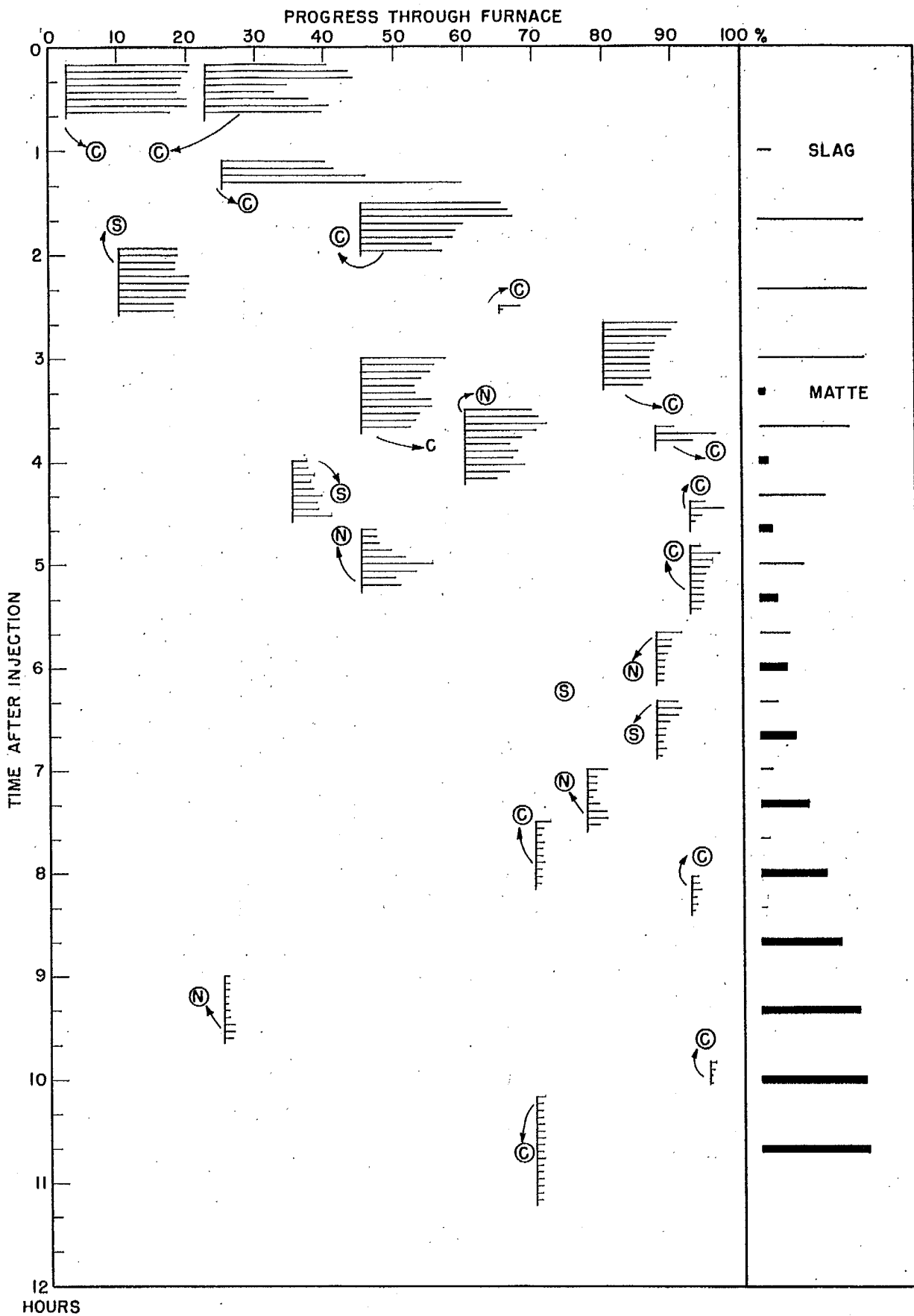


FIGURE 9. DEPTH PROBE SAMPLES, NO. 3 REVERBERATORY FURNACE

TABLE 7

Measured Activities of Converter Probe Slag Sample

| Date (July) and Hour | Hours After T _o | Decay Factor | Measured Counts per Minute | Counts per min at T _o | Activity μc at T _o | Remarks |
|----------------------|----------------------------|--------------|----------------------------|----------------------------------|-------------------------------|--|
| 16th 2:40 pm | | | -- | | | Counter blocked |
| 17th 9:45 pm | 33 | 0.167 | 272,000 | 1.62 x 10 ⁶ | 450 | Count rate very high |
| 18th 12:30 pm | 47.7 | 0.075 | 102,000 | 1.36 x 10 ⁶ | 378 | |
| 20th 8:25 pm | 103.6 | 0.00366 | 8,900 | 2.43 x 10 ⁶ | 338 | B/B counting system approx. 2X counting sensitivity of N/C system (see note to Table 9, Appendix 3) Possible decrease in sensitivity due to magnetic effects (encountered at first in Ottawa) |
| 20th 8:45 pm | 103.9 | 0.00360 | 8,800 | 2.44 x 10 ⁶ | 339 | |
| 21st 10:45 am | 117.9 | 0.00169 | 4,200 | 2.49 x 10 ⁶ | 346 | |
| 21st 2:30 pm | 121.7 | 0.00137 | 1,563 | 1.14 x 10 ⁶ | 317 | |
| 22nd 10:40 am | 141.8 | 0.000463 | 326 | 0.70 x 10 ⁶ | 195 | |
| 22nd 9:30 pm | 152.7 | 0.000255 | 245 | 0.96 x 10 ⁶ | 267 | |
| 23rd 8:00 am | 163.3 | 0.000144 | 178 | 1.23 x 10 ⁶ | 342 | |

- (1) Average value of activity at T_o taken as 350 μc per 5100 grams.
 Total activity measured at injection with ion chamber 643 mc ± 20%
 Total activity calculated from tapped samples 662 mc ± 15%

- (2) Average taken as 655 millicuries.

Assuming specific activity in (1) and total activity in (2),

$$\text{Mass would be } \frac{655,000}{350} \times \frac{5100}{454 \times 2000} = 10.5 \text{ tons.}$$

Mass of converter slag in ladle 14 tons.

DISCUSSION

This radiotracer experiment has shown that a very small amount of copper, having a source strength of a few hundred millicuries, is adequate to study the flow of material through a reverberatory furnace containing about 1600 tons of slag and matte. The use of a relatively efficient counting arrangement in which a large sample almost enclosed the gamma-ray detector within a thick lead shield, permitted the copper-64 content of the tapped samples to be measured during more than two days. It is, perhaps, surprising that only a brief counting period (5 minutes or less) was required at the end of this time after the source had decayed by almost a factor of sixteen and very great dilution had taken place through the addition to, and removal from the furnace of another 1500 tons of material. The ladle poured at the time the last matte sample was taken, contained somewhat less than 2.5 milligrams of the original copper source in 19 tons.

The subsequent tests conducted to calibrate the two sizes of sample containers showed consolidation, that is the degree of packing, to be a possible source of error in determining the activity of an individual sample. Fine structure in the matte curve, therefore, may not represent entirely the short term variations in activity at the matte tap hole. In future tests uniform weight and sample volume should both be maintained wherever possible and a record kept of each.

The 50% efficiency factor used for the reactor irradiation of copper would appear to be pessimistic. Assuming the neutron flux to be exactly as stated (2.5×10^{14} n/cm²/sec); an irradiation efficiency of close to 75% was achieved. Theoretical calculations would indicate that the efficiency should be greater than 90%. However, in view of uncertainties in the reactor flux, it is suggested that a figure of 75% should be used for preliminary calculations.

Further work, possibly chemical analysis, will be required to explain the discrepancy between the depth probe results obtained near the slag and matte tap holes, and the results from the related tap samples. Both of these groups were plotted to scale in Figure 9. The activities of the depth probe samples are apparently too low; however, they do show the spreading out of the radioactive material in the furnace at lower levels. Further investigations of this discrepancy should be designed to show whether the probes picked up along their length, a layer of relatively inactive material from the surface layers of the charge in the furnace upon insertion, withdrawal, or both.

In the counting procedures, desirable improvements should include: the use of all-decimal, moderately fast (200 kc +) counters; large, ruled forms prepared for recording data; a larger supply of sample containers, some of which would be small dip containers for the smaller probe samples. With the services of at least one technical assistant it should then be possible to reduce the results as the measurements proceed. Rapid fluctuations in line voltage readily visible on a voltmeter were a cause for concern; however, the regulating capabilities of the counters, and the automatic line regulators seem to have been adequate.

Insofar as smelter metallurgy is concerned, the test has shown that the recovery of copper from cuprous oxide in converter slag returned to the reverberatory furnace is high. It would be interesting to find out whether the recovery of copper from cuprous sulphide in converter slag shows similar behaviour.

The methods used to handle a copper-64 source of this strength were quite adequate from the standpoint of health safety. No detectable radioactivity, as collected by the air sampler, was imparted to the atmosphere around the converter floor; no measurable activity was found on any of the film badges. The radiation dose at the surface of the shipping container was so low, < 5 milliroentgens/hour, that it would be preferable in future tests to ship a stronger source for delivery the day before the test, and to commence the test at the beginning of the working day. This would make it unnecessary to carry on sample preparation, and counting until 1:00 a.m. after a full working day from 8:00 a.m.

In the matter of liaison, several discussions were held prior to the test with personnel from the Noranda Research Centre to plan aspects of the test, in particular the injection of the source. This was to ensure that the source in its capsule did not become entrapped in the skull of the ladle, also that it did not remain on the surface to be splashed, even partly, out of the ladle as the latter was being filled. On the day before the test, further discussions were held including, also, staff of the Noranda Smelter. The design of the injection rod finally adopted combined the suggestions of all three groups.

At the final meeting mentioned above, areas of responsibility were carefully assigned. The authors found this to be very helpful when several unexpected problems arose in the course of the test and during sample preparation.

ACKNOWLEDGEMENTS

The authors wish to acknowledge the effective contribution in the planning and the conducting of the radiotracer test by the staff of the Noranda Co., including Dr. N.J. Themelis and his group from the Noranda Research Centre, Montreal, and Mr. L.A. Mills, Smelter Metallurgist, and his staff at Noranda, Que.

Mr. B. Ullyett, Atomic Energy of Canada Ltd., despite last minute reactor problems, provided for the irradiation, and delivery on schedule of the radioactive source.

The assistance of other Mines Branch personnel in the construction and testing of equipment, and in the subsequent standardization procedure is acknowledged with thanks, in particular Messrs. C. McMahon, A.F. Seeley and D.A. Fisher (Summer Student Assistant).

Dr. G.G. Eichholz, former Head of the Physics and Radiotracer Subdivision initiated this work.

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2. G.G. Eichholz, "Report on a Field Trip to Noranda, Que. and Test Work at Noranda Mines Ltd., May 12-15, 1958", Mines Branch Internal Report No. Ra-280/58.
3. J.C. Turgeon, "A Study of Contact Times in Pilot Plant Agitators, Using Radioactive Tracers", The Can. Mining and Metallurgical Bulletin, Jan. 1956.

APPENDIX 1

Calculation of Copper-64 Activity for Reactor Irradiation

The equation for the formation of a radioactive isotope by neutron irradiation in a reactor is

$$\left(\frac{dN}{dt}\right)_0 = \frac{w}{A.Wt} \times f \times N \times \sigma \times \frac{F}{E} \cdot \left(1 - e^{-\frac{0.693 \times T}{T_{\frac{1}{2}}}}\right) \quad (\text{Eq 1})$$

where $\left(\frac{dN}{dt}\right)_0$ = the number of disintegrations per second of the induced activity at the end of the irradiation,

w = weight of the target element (g),

A.Wt = atomic weight of target element (g),

f = fraction of target isotope in element (abundance),

N = Avogadro's Number,

σ = cross section of target isotope in cm^2 (1 barn = 10^{-24}cm^2),

F = neutron flux in $\text{n/cm}^2/\text{sec}$,

E = irradiation efficiency,

T = irradiation time,

$T_{\frac{1}{2}}$ = half-life of radioisotope (same units as T).

The activity remaining at any time "t", after the irradiation, is given by

$$\left(\frac{dN}{dt}\right)_t = \left(\frac{dN}{dt}\right)_0 e^{-\frac{0.693 \times t}{T_{\frac{1}{2}}}} \quad (\text{Eq 2})$$

Copper-64 is produced by thermal neutron irradiation of copper-63 (abundance 69.09%), which has a cross section, of 4.5 barns. In view of this relatively high cross section it was suggested by Commercial Products Division of A.E.C.L. that a 50% irradiation efficiency be assumed. For the Noranda test, the irradiation was performed in the pneumatic facility of the N.R.U. reactor at a flux of $2.5 \times 10^{14} \text{n/cm}^2/\text{sec}$.

Two separate irradiations were performed, the Go-sample consisting of 0.8 g Cu_2O and the Back-up sample consisting of 2g Cu_2O in two separate lead cylinders, containing 1.2 g and 0.8 g Cu_2O respectively. The calculated irradiation times were based on the irradiations ending at 8:00 a.m. July 15, for use at noon on July 16 for the Go-sample and at noon on July 17 for the Back-up sample. Thus for the Go-sample there

would be a decay time of 28 hours and for the Back-up sample a decay time of 52 hours.

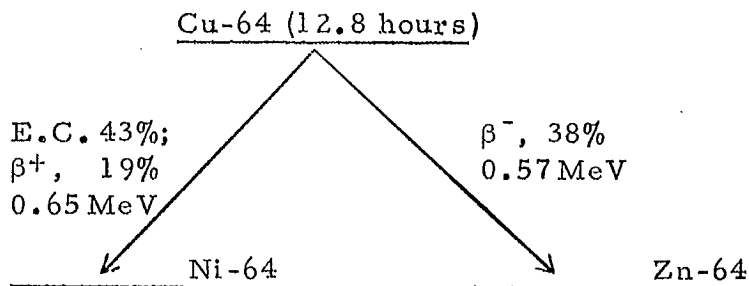
Using the above data in equations (1) and (2) the irradiation times were calculated as 18 minutes and 28 minutes for the Go and Back-up samples respectively.

In practice, when the Go-sample was opened in the hot cell at Chalk River for re-encapsulation following irradiation, it was found that the lead capsule had melted. Irradiation of the Back-up samples was immediately stopped at 9:40 a.m. (July 15) after 20.5 minutes in the reactor. Fortunately, in this case, no melt-down had occurred and both sources were shipped to Noranda for the test. The induced activity expected in the two separate Back-up samples at the time of injection into the furnace (12:50 p.m., July 16) may be calculated as; 0.8 g Cu_2O , 300 mc; 1.2 g Cu_2O , 450 mc, assuming as before, an irradiation efficiency of 50%.

APPENDIX 2

Total Activity from Ionization Chamber Measurements

The decay scheme of copper-64 is given below:



Two 0.51 MeV gamma photons are produced when a positron (β^+) is stopped, so, for dose-rate measurements, copper-64 may be regarded as decaying by 38% of 0.51 MeV gamma emission. The dose-rate response of an ionization chamber is dependent on the energy of the incident gamma flux and is related to the activity of the gamma-emitting source by the "K" factor for the particular gamma energy. The "K" factor for one curie of 0.51 MeV gamma radiation is 320 mr/hr* at one metre. A dose-rate of approximately 35 mr/hr at one metre was measured from the copper-64 source in the injection rod. This reading is lower than the true dose-rate reading because of the attenuation of the radiation by the lead irradiation cylinder, the iron injection pipe, and the source itself. Correction for this attenuation is made from

$$I = I_0 e^{-\mu d}$$

where I and I_0 are the transmitted and source intensities, μ the linear absorption coefficient and d the thickness of the absorber.

* Siegbahn, K., Beta- and Gamma-Ray Spectroscopy, p. 851, 1955, North-Holland Publishing Co., Amsterdam (Chapter XXVI - Measurement of Disintegration Rate by J.L. Putman).

Benson, P.A. and Gleit, C.E., Nomograph for Determination of Neutron-Induced Activities, Nucleonics, Vol. 21, No. 8, 148-150 (1963).

TABLE 8

Data for Absorption Calculations

| Material | d | μ | $\sqrt{I_0}$ |
|----------|---------|-------|--------------|
| Pb | 1.75 mm | 1.72 | 0.74 |
| Fe | 6.6 mm | 0.66 | 0.65 |
| Cu | 1.36 mm | 0.502 | 0.93 |

(d for the iron is somewhat greater than the pipe wall thickness since, in the dose-rate measurement position, a greater thickness of iron was traversed).

The combined reduction in source intensity by the iron and lead is thus $\sim 55.3\%$. Therefore, the total source strength is

$$\frac{35}{320} \times \frac{1}{0.447} \times \frac{1}{0.38} = 0.643 \text{ curies} \\ \text{or } 643 \text{ mc}$$

APPENDIX 3

Counting Efficiency

The usual definition of counting efficiency relates the number of counts, N, recorded to the number of radioactive disintegrations, D, actually occurring in a sample during the counting interval; that is, $E = N/D \times 100\%$. In copper-64 only 38% of the disintegrations result in gamma rays; beta particle production is 57%. However, owing to the limited penetration of beta particles and the consequently small sampling volume, attempts to measure beta particles with an end-window Geiger-Müller tube resulted in a very small number of recorded counts relative to those obtained with a gamma counter from the same sample.

Maximum efficiency in the case of a small sample, less than say 20 grams, may be realized by placing it within the gamma-ray detector, that is, by using a well-counter. The efficiency will then approach the upper limit attainable. (Because of the simultaneous emission of the two gamma rays, for practical reasons this efficiency will not exceed 19%). However, D will be small in this case, and therefore $N = (D \times E)$ will also be small. If a large amount of sample material is available, D may be increased greatly until N reaches a limiting value because E becomes very small for layers of the material remote from the detector. This latter condition results from attenuation of radiation due to internal self-absorption and inverse square law effects. These limitations are minimized by placing the sample around the detector to form a dip counter.

The investigation by Perkins* of various sample/detector configurations is perhaps the closest parallel case to that of the copper-64 in slag and matte. He used solutions containing chromium-51 as a gamma emitter. The combination of the softer gamma radiation, 320 keV, and less dense material mostly water, produces similar absorption effects to those for copper-64 in smelter material. Dip counter results by Perkins for samples similar in size to those in the copper test are shown in Table 8, along with results using spiked matte. (A smaller dip counter would have been useful for the probe samples had it been known that they would be counted as pulverized specimens).

*Perkins, R. W., The Responses of Various Detector Sample Arrangements for Gamma-Ray Spectrometer Measurements, Hanford Atomic Products Operation, Richland, Washington, U.S.A. Report No. HW 6852 (Office of Tech. Serv., Dept. of Commerce, Washington 25, D.C.).

TABLE 9

Dip Counter Measurements

| Sample | Volume or Mass | Counts/Minute | Ratio | Crystal Size (inches) | Remarks |
|--|----------------|---------------|-------|-----------------------|---|
| 1. Cr-51 in H ₂ O | 500 ml | 232 | 1 | 3 x 3 | 500 ml polythene bottle. |
| 2. " | 3100 ml | 1490 | 6.4 | " | Dip counter, 2 in. at the sides and top of the crystal. |
| 3. " | 6500 ml | 2070 | 8.9 | " | Dip counter, 3 in. on sides and top. |
| 4. Cu-64 in matte | 500 g | 2225 | 1 | 1.5 x 1.5 | 250 ml beaker B/B counting system. |
| 5. " | 5360 g | 20,640 | 9.28 | " | Dip counter B/B counting system. |
| 6. Cu-64 in matte | 500 g | 640 | 1 | 2 x 2 | 250 ml beaker N/C counting system. |
| 7. " | 5360 g | 9260 | 14.4 | " | Dip counter N/C counting system. |
| Ratio of N/C to B/B systems (as used at Noranda) | | | 4.16 | Comparing 4 and 7. | |

Note: B/B - integral system, therefore higher apparent count rate than N/C system which was a window system (comparing 4 and 6, also 5 and 7).

The counting efficiency, E, of the N/C counting system was estimated to be about 2%. This value is consistent with those obtained by Wagner and Guinn*.

* Wagner, C.D. and Guinn, V.P., Nucleonics, 13 (10), 56 (1955).

APPENDIX 4

List of Equipment

(1) Ionization Chamber:

Radiation Survey Meter, Model SUIH,
Tracerlab, Boston, Mass., U.S.A.

(2) Portable Scintillation Counter:

Universal Radiation Meter, Model 1700 fitted
with 3/4 in. x 3/4 in. sodium iodide detector.
Nuclear Enterprises Ltd.,
Winnipeg 21, Canada.

(3) N/C Counting System:

2 in. x 2 in. sodium iodide detector with photo-
multiplier and pre-amplifier.
Nuclear-Chicago Single-channel Analyser,
Model 132B.
Sola Constant Voltage transformer, 500 VA capacity.

(4) B/B Counting System:

1.5 in. x 1.5 in. sodium iodide detector with
photomultiplier and pre-amplifier.
Beckman/Berkeley Scaler, Model 2101.
Sorensen Voltage Regulator, Model 250S,
250 VA watts.

APPENDIX 5

Counting Sensitivity for Reverberatory Slag and Matte

In order to determine accurately the distribution of copper-64 between the slag and matte, it is necessary to know the relative count-rates obtained from the same copper-64 activity in equal volumes of both phases. Since the matte is approximately 1.27 times denser than the slag, it is obvious that internal self-shielding in the matte will reduce its measured count-rate relative to the slag. The extent of this reduction was measured at the Mines Branch, in a separate test, after returning from Noranda and the opportunity was also taken to obtain a measure of the total activity added to the reverberatory furnace.

Using the Mines Branch neutron generator as a neutron source, 34.4 g of cuprous oxide were irradiated in a thermal flux of about 5×10^7 n/cm²/sec for 40 minutes. After the irradiation, the powder was thoroughly mixed to avoid any flux non-uniformity during irradiation, and a 0.26 g sample sent to Dr. A. Baerg of the National Research Council for absolute counting. The remainder of the cuprous oxide was then dissolved in a minimum amount of 40% nitric acid and divided into two equal aliquots. These aliquots were added to 5600 g matte and 4400 g slag, contained in shallow trays, that had been pre-heated to 150°C. After addition of the activated copper solution, the slag and matte samples were maintained at 150°C for a further hour, then allowed to cool to room temperature, crushed and thoroughly mixed.

Successive 400 g samples of the matte were added to the counting vessel, tamped down firmly and counted with the single-channel analyser. In this manner the relationships between weight of sample and measured count were found (Figure 5). The same procedure was followed for the slag.

The N.R.C. results indicated that, for the 5600 g matte sample, 7400 cpm corresponded to an activity of 2.1 μ c; the comparable result for the slag was that in 4400 g, 2.1 μ c gave 7550 cpm.

APPENDIX 6

Calculation of Activity Distribution Between Reverberatory
Matte and Slag

(i) Matte Samples

In order to measure the total activity in the matte phase, it is necessary to know the total number of counts obtained from the matte. This total comprises:

- (a) the 75 samples counted at Noranda that showed definite activity above background (M 5-M 79),
- (b) correction for the four taps made between M 38-M 39 from which no samples were taken,
- (c) extrapolation of Figure 8 to negligible activity in the matte phase.

These separate counts are:

| | | |
|--|---|---------------|
| Counts from 74 matte samples | = | 526,772 |
| Interpolation from Figure 8 for four samples between M 38-M 39 | = | 44,800 |
| Extrapolation of Figure 8 to 121 hrs, assuming tapping every 0.6 hours (124 samples) | = | <u>98,400</u> |
| | | 669,972 |

This total count is from 202 samples, giving an average count-rate of 3317 cpm/sample. The average weight of the samples taken was 4970 g. From Figure 5 it may be shown that 1.864 μ c of copper-64 in 4970 g of matte would give 7050 cpm. Therefore, for the Noranda samples that gave an average of 3317 cpm, there should be

$$\frac{3317}{7050} \times 1.864 = 0.877 \mu\text{c/sample.}$$

Assuming that the activity in the sample taken from the ladle is accurately representative of total activity in the ladle, then the total

matte activity for 202 ladles (each of 19 tons) is

$$\frac{202 \times 19 \times 2000 \times 453.6 \times 0.877}{4970 \times 10^3} \text{ mc}$$
$$= 615 \text{ mc}$$

(ii) Slag Samples

The total activity in the slag phase may be calculated in a similar manner. Samples S1-S19 gave an average count-rate of 3130 cpm/sample of average weight 4064 g. As before, from Figure 5, 1.94 μc of copper-64 in 4064 g of slag would give 7400 cpm. Therefore, 3130 cpm corresponds to

$$\frac{3130}{7400} \times 1.94 = 0.82 \mu\text{c of Cu-64/sample.}$$

In 19 ladles of slag, weighing 13.5 tons each, there would be

$$\frac{19 \times 13.5 \times 2000 \times 0.82 \times 453.6}{10^3 \times 4064} \text{ mc}$$
$$= 46.9 \text{ mc}$$

Thus, the total activity for both phases is 662 mc and the distribution of activity is 92.9% in the matte and 7.1% in the slag.

JLH:HPD/DV