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THE DETERMINATION OF SILICA IN IRON ORE CONCENTRATES BY FAST NEUTRON ACTIVATION ANALYSIS

by

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BY FAST NEUTRON ACTIVATION ANALYSIS

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H.P. Dibbs* and C. McMahon**

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SUMMARY OF RESULTS

A method has been developed, using fast neutron activation analysis, for the rapid determination of silica in iron ore concentrates. The results obtained show very good agreement with those obtained by conventional chemical analysis.

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INTRODUCTION

During a recent visit by certain Mines Branch personnel* to Hilton Mines Limited, Bristol, Quebec, the question was raised of the possible application of neutron activation analysis to the rapid analysis of silica in high-grade iron ore concentrate. Four samples of concentrate, that had been analysed previously by Hilton Mines for silica content, were brought to Ottawa for trial irradiation using a neutron generator. The quoted silica content of the samples ranged from 1% to 3.72%.

The neutron generator offers a choice of fast (14 MeV) neutron or thermal (0.025 eV) neutron irradiation. Silicon and iron give a number of radioactive products on neutron irradiation, depending on the incident neutron energy (Table 1).

Fast neutron irradiation was chosen as the more suitable irradiation method because of the higher isotopic abundances of the target nuclides, the shorter half-lives of the radioactive isotopes produced, and the greater percentage of gamma emission associated with the decay of the product radioisotopes. These three factors all tend to give enhanced detection efficiency.

EXPERIMENTAL

Figure 1 shows the gamma-ray spectra resulting from separate fast neutron irradiations of silica and iron. Only two radioisotopes are produced in significant amounts; namely, aluminum-28 and manganese-56. In order to measure aluminum-28 in the presence of manganese-56, it is convenient to count the gamma radiation in the region of 1.8 MeV. A single-channel gamma-ray spectrometer was used for this purpose. A forty-second fast neutron irradiation was made on one of the concentrate samples, and the decay of the 1.8 MeV peak followed over a period of three hours. The decay curve (Figure 2) consists of only two components, aluminum-28 and manganese-56. The initial portion of the decay curve was resolved (Figure 3) by subtracting the long-lived manganese-56 component, to give the aluminum-28 component. A comparison of the aluminum-28 activity from the four samples would be expected to give a direct measure of their silicon content.

TABLE 1

Radioisotopes Formed by Thermal Neutron

and Fast Neutron Irradiation of Silicon and Iron

| Target Nuclide	Isotopic Abundance (%)	Thermal Neutron Irradiation			Fast Neutron Irradiation		
		Reaction	$T_{\frac{1}{2}}$ Product	E_γ(MeV)	Reaction	$T_{\frac{1}{2}}$ Product	E_γ(MeV)
Si28	92.27	Si28(n, γ)Si29	Stable	-	Si28(n, p)Al28	2.3 min	1.78 (100%)
Si29	4.68	Si29(n, γ)Si30	Stable	-	Si29(n, p)Al29	6.6 min	2.43(15%) 1.28(85%)
Si30	3.05	Si30(n, γ)Si31	2.62 hr	1.26(0.07%)	Si30(n,∝)Mg 27	9.45 min	1.02(30%) 0.84(70%)
Fe54	5.84	Fe54(n, γ)Fe55	2.6 yr	None	Fe54(n, p)Mn 54	291 d	0.84 (100%)
Fe56	91.68	Fe56(n, γ)Fe57	Stable	-	Fe56(n, p)Mn 56	2.58 hr	0.85(99%)1.81(25%) 2.12 (15%)
Fe57	2.17	Fe57(n, γ)Fe58	Stable	-			
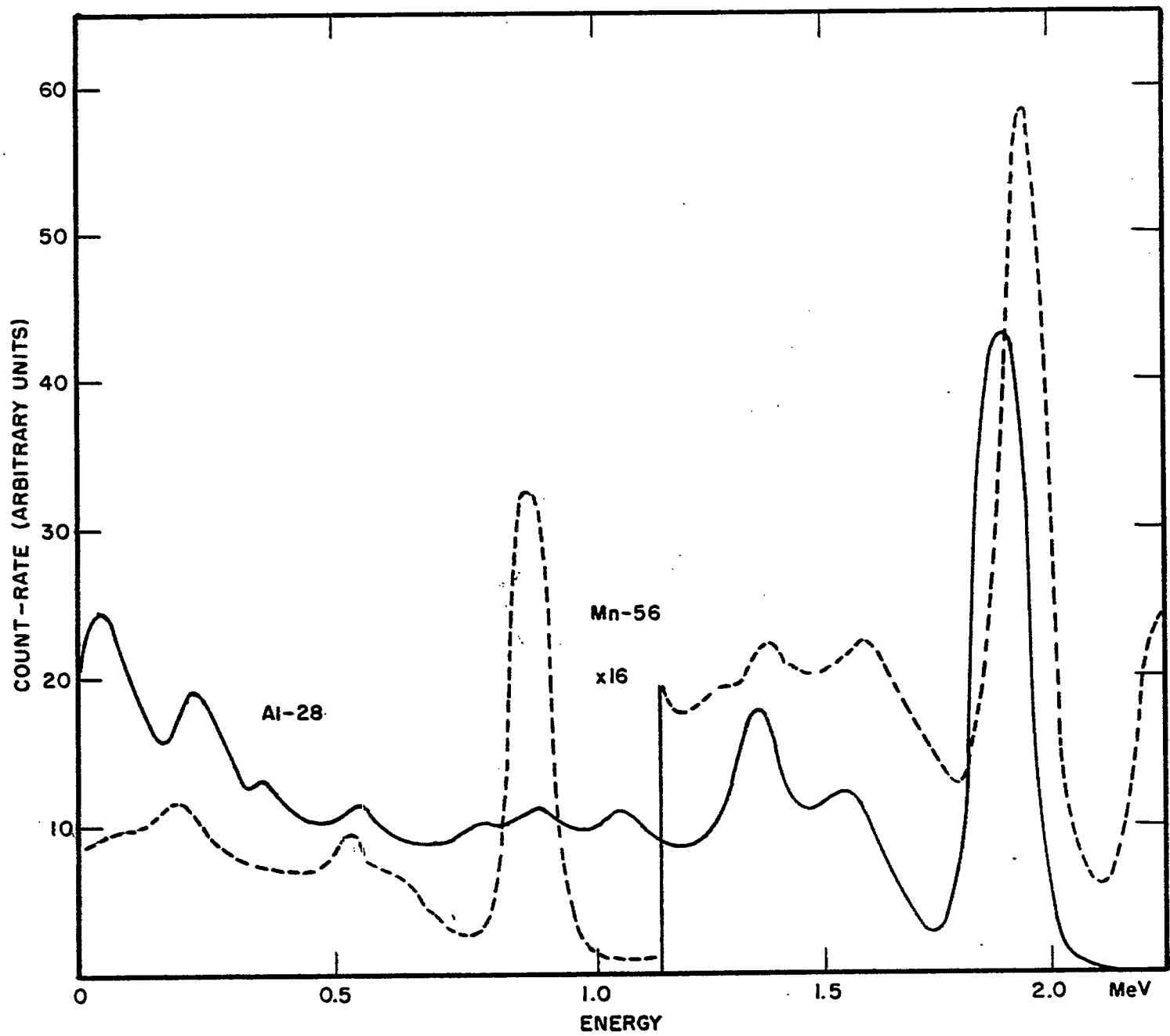
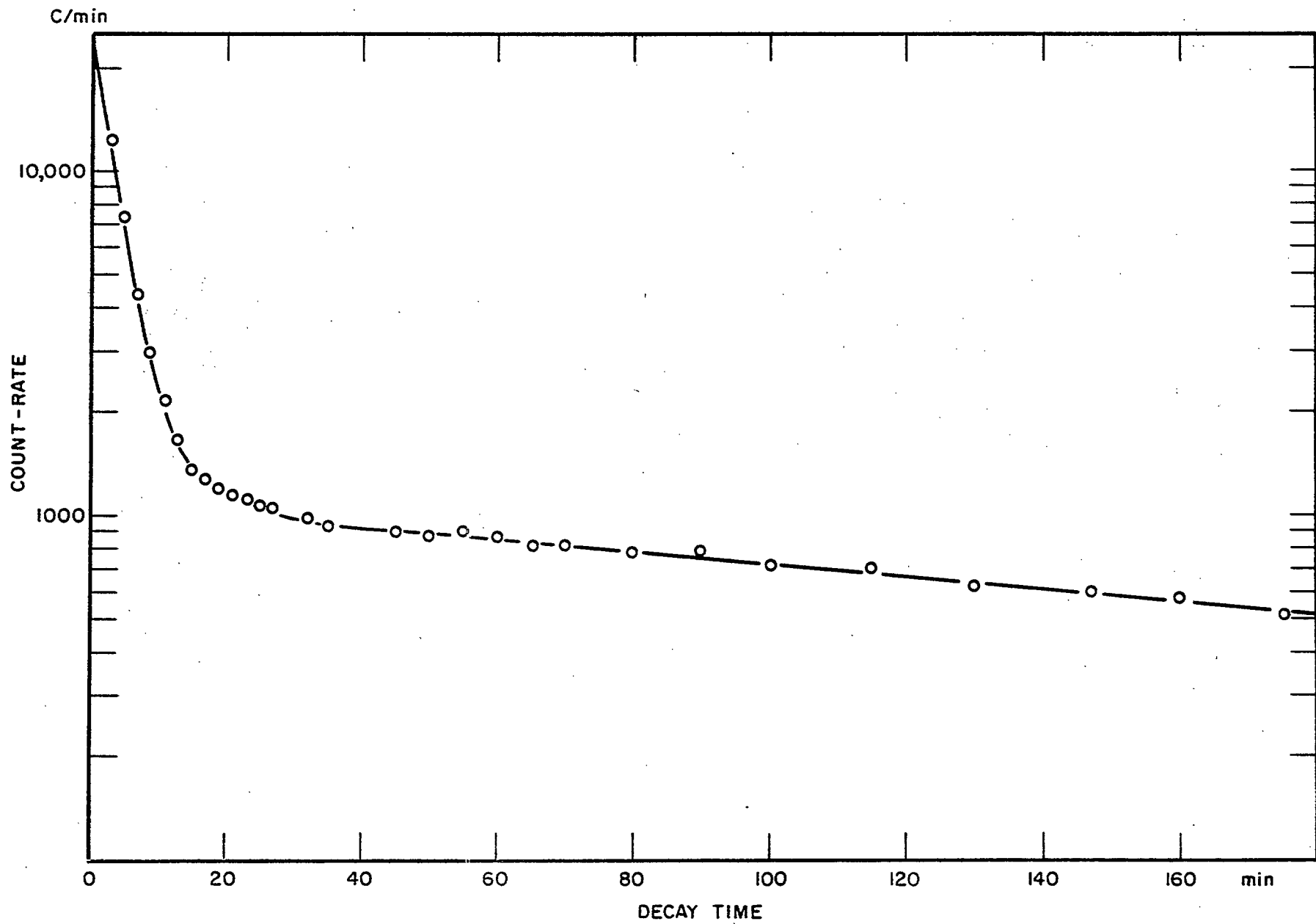FIGURE I   GAMMA-RAY SPECTRUM OF Al-28 AND Mn-56.

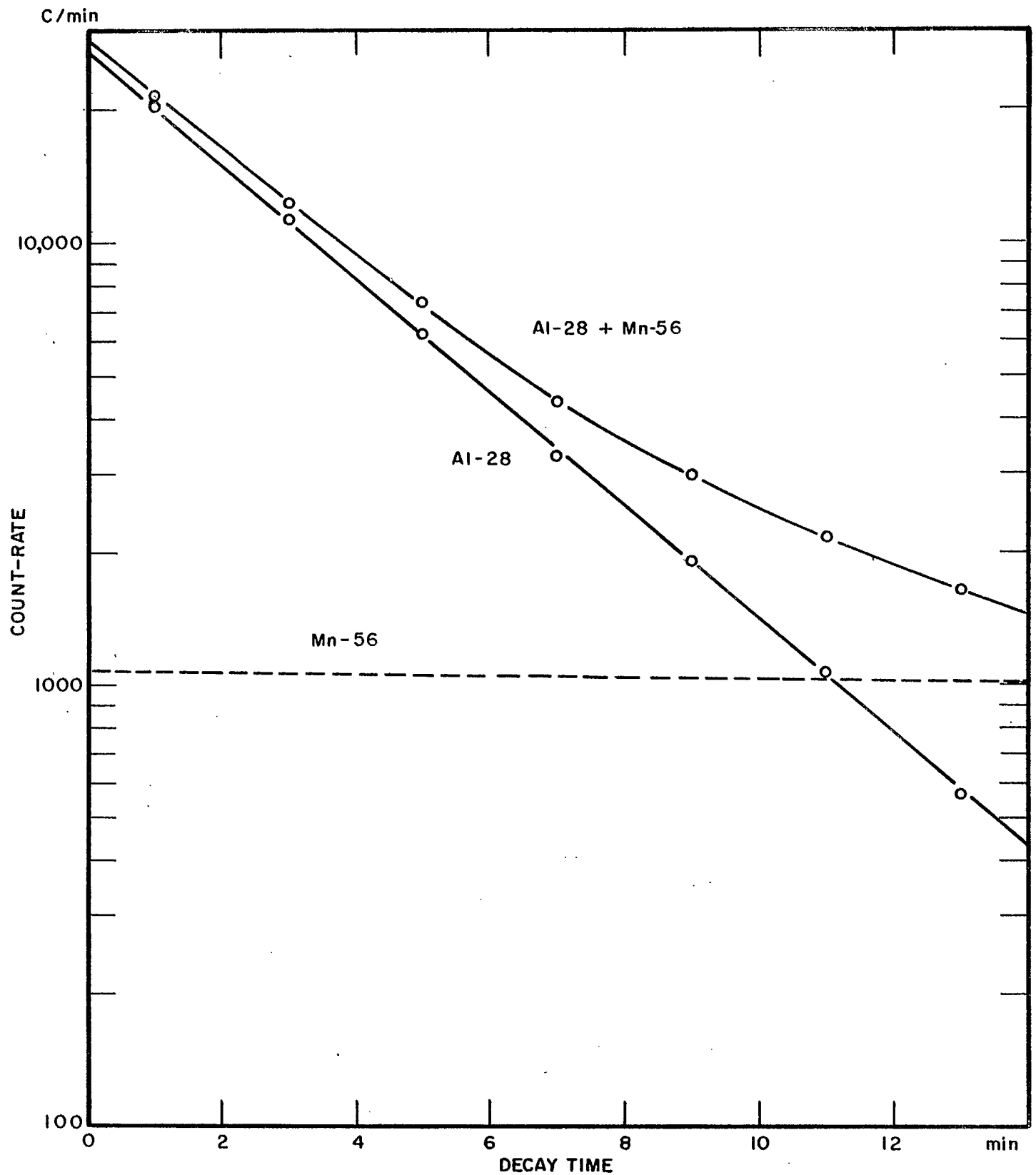FIGURE 2 DECAY CURVE OF IRON CONCENTRATE FOLLOWING FAST NEUTRON ACTIVATION.

FIGURE 3   RESOLVED DECAY CURVE OF AI-28 AND Mn-56.

For irradiation, the samples were sealed in 5-ml capacity polythene vials, each vial holding about 21 g of the sample. Each sample was then irradiated for forty seconds. After two minutes decay, a one minute count was taken for aluminum-28 and manganese-56. Forty minutes after the end of the irradiation, during which time all the aluminum-28 had decayed (see Figure 2), a further four minute count was taken for manganese-56. After appropriate correction for the thirty-eight minutes difference in measurement time, the aluminum-28 activity was obtained by the difference in the counts per minute readings. A comparison standard, containing 8.415 g of high-purity silica rod, was also run with the concentrates. A comparison of the counts per minute from the standard, following two minutes decay, with the corrected aluminum-28 count from the concentrate, provides a direct measure of the silicon content of the concentrate. The comparison, however, does not give a quantitative measure of the silica concentration in the concentrate because of the difference in density between the samples and standard. This density difference causes a different amount of self-absorption of the 1.8 MeV gamma radiation within the sample, as compared with the standard. This factor, however, should be constant for the four samples, with all the results correspondingly low as compared to the chemical values. Four separate silica determinations were made for each of the concentrate samples and the results compared to the silica standard. The activation results were in good internal agreement, ($\pm$ 4% or better), but the ratios of the activation results to the quoted chemical assays were poor (Table 2). In view of this discrepancy a second chemical analysis was made. The chemical results obtained for the silica content from this analysis differed from the Hilton values but were in very good ratio agreement to the activation values (Table 3) and indicated that a factor of 11% was required to correct for gamma-ray absorption within the sample.

TABLE 2

The Silica Content of Iron Oxide Samples as

Determined by Fast Neutron Activation Analysis

Sample Number	Net Weight of Sample (g)	Average Value of SiO_2 Content by Activation (%)	Hilton Value for SiO_2 Content (%)	Ratio of Activation, to Hilton Values
1	21.09	3.34 $\pm$ 0.06	3.62	0.92
2	21.07	1.49 $\pm$ 0.06	1.46	1.02
3	20.73	3.89 $\pm$ 0.10	3.72	1.05
4	20.90	1.24 $\pm$ 0.02	1.0	1.24

TABLE 3

Comparison of Activation and Chemical

Silica Content for Iron Oxide Samples

Sample Number	Silica Content, Mines Branch Chemical Analysis (%)	Silica Content by Activation (%)	Ratio of Activation, to Mines Branch Chemical
1	3.77	3.34	0.89
2	1.69	1.49	0.88
3	4.37	3.89	0.89
4	1.36	1.24	0.91

INTERFERENCE

Serious interference in the determination of silicon by the Si-28 (n, p) Al-28 reaction, will occur in the presence of phosphorus from the (n, α) reaction:

$$P\text{-}31 + n = Al\text{-}28 + \alpha$$

A separate experiment indicated that a silica-free sample containing 100 mg of phosphorus would give an apparent silica content of 93 mg. As the iron oxide concentrate has a very low phosphorus content (1), this source of error is not significant in the present case.

CONCLUSIONS

Fast neutron activation analysis can provide a non-destructive technique for the determination of silica in iron oxide. The method is consistent and has been shown to give good agreement with conventional chemical methods. For iron oxide containing only a few percent of silica the iron concentration in different samples will be essentially constant. Thus, if a previously determined value is used for the manganese-56 contribution to the count taken after two minutes decay, the assay could be completed in less than five minutes with little loss in accuracy.

ACKNOWLEDGEMENTS

The chemical analyses were made by Mr. R. Craig, Mineral Sciences Division.

REFERENCES

(1) G. Viens, private communication.

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