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ABSTRACT

A new x-ray emission spectrograph is described in which characteristic x-rays excited by radiations of ^{241}Am are detected by a lithium-drifted silicon semiconductor detector. Recent improvements in circuitry result in superior energy discrimination. The procedure for the calibration of the spectrograph is discussed and its usefulness as a quantitative analytical instruments are explored in two general cases (1) analysis of thick samples for an element in the 1-10% weight concentration range and (2) analysis of thin samples of milligram size.

I. INTRODUCTION

In 1961 Kohler (1) pointed out that a semiconductor detector would be a superior x-ray detector for use in fluorescent x-ray analysis if certain improvements could be made in the detectors and the associated electronic amplifiers to achieve better energy discrimination. In a recent publication from our laboratory Bowman et al. (2) describe an x-ray emission spectrometer incorporating a semiconductor detector system with greatly improved capability for the resolution of x-ray spectra.

The component parts of the proposed spectrometer are shown in Figure 1. Radiations from a γ -ray source such as ^{241}Am or ^{57}Co impinge on an analytical specimen and excite characteristic x-rays. A fraction of these reach the semiconductor detector (lithium-drifted silicon or germanium crystals) and produce electrical charge which is converted to electrical pulses. These pulses are amplified and fed to a multichannel analyzer which determines their amplitude. The use of a pre-amplifier circuit developed by Elad and Nakamura (3) made it possible to construct a system with higher energy resolving capability over the whole range of x ray energies than had ever been achieved before with semiconductor detectors. The energy resolving power of the silicon detector used in this system was approximately 1.1 k.e.v. to 1.5 k.e.v. expressed as full width at half maximum, for monoenergetic x-rays in the energy range 4 to 40 k.e.v. For the germanium detector a value of 0.7 k.e.v. was achieved for platinum x-rays (3).

The proposed spectrometer does not differ in principle from the several forms of a radioisotope x-ray emission spectrometer suggested in the past in which a radioactive source replaces the x ray tube of the conventional spectrograph and

a proportional counter or scintillation counter replaces the Bragg scattering crystal for the analysis of the characteristic x radiation. Good discussions of past work on a radioisotope system are given by Cook, Mellish, and Payne (4) (1958) and by Cameron and Rhodes (5,6) (1961, 1963). A few representative applications can be cited. Zemany (7) discussed the use of ^{55}Fe to excite titanium x rays as a means of measurement of titanium coating thicknesses. Forburg and de Ruvo (8) discuss the analysis of uranium solutions by scintillation detection of x-ray fluorescence excited by ^{57}Co x rays. Karttunen et al. (9) describe a portable fluorescent x-ray instrument utilizing radioactive isotopes. Rhodes, Ahier, and Boyce (10) discuss the determination of tin in tin ores by radioisotope x-ray fluorescence.

The semiconductor form of the radioisotope x-ray emission spectrograph could be substituted in these applications and in many instances would be a superior instrument. It is the purpose of the present paper to show how such a system would perform as a general purpose instrument for quantitative analysis. This paper may help the analytical or control chemist to decide whether this new analytical tool offers any advantage over present techniques for his specific purposes.

We show the procedure for calibrating the energy scale, for checking the linearity of the pulse height versus x-ray energy relationship and for computing the energy resolution. Then we turn to two specific analytical problems chosen to represent two very general classes; namely: (1) analysis of a constituent in a 1-10% concentration range in a thick sample and (2) analysis of a trace component subjected to preliminary chemical separation and afterwards presented to the x-ray

spectrograph as a thin sample. We have limited our study to silicon detectors and to ^{241}Am as the exciting source.

APPARATUS

A lithium-drifted silicon diode 1.4 cm. in diameter with a 2.5 mm. deep sensitive layer was mounted in an aluminum housing behind a thin beryllium window. See Figure 2. A preamplifier incorporating a field effect transistor (FET), designated 2N3823, was located immediately behind the silicon detector. The FET served as the first stage of a charge sensitive preamplifier (3). The detector and the FET were maintained at liquid nitrogen temperature. The housing containing the semiconductor diode and the FET was evacuated. Details of the FET preamplifier assembly are given by Elad (3). The resolution capability of the spectrometer is chiefly dependent upon this preamplifier design. Standard commercial amplifiers were used.

A source of radioactive ^{241}Am was prepared by depositing approximately 10^9 alpha disintegrations per minute (about 1/2 millicurie) in a hole in a lead cylinder of 4 mm. diameter. This source was cemented to the front surface of the beryllium window in front of and below the detector. Samples to be analyzed were mounted a few millimeters from the opening in front of the ^{241}Am shield.

Americium-241 is an alpha emitter with a half-life of 470 years. The alpha particles are totally absorbed in the cover over the source. In addition ^{241}Am emits the x-rays and γ rays shown in Figure 3. These radiations cause the fluorescence of characteristic x rays in the sample. A certain fraction of the secondary x radiation reaches the semiconductor diode, where it is absorbed by the photoelectric process, giving rise to an electrical pulse proportional in energy to the absorbed x ray.

The output pulses from the amplifier were fed to a 400 channel analyzer. In most experiments data were collected in 200 channel blocks of the memory. At the end of the data collection period, which was typically 1 to 10 minutes, the stored information was printed out on paper tape and/or plotted out with a Moseley XY plotter (12). The gain and bias controls of the amplifier and pulse height analyzer were varied to select the x-ray region of interest.

This equipment is suitable for the K x-ray analysis of elements from about calcium (element 20) to erbium (element 68). For analysis of K x rays of higher elements a combination of ^{57}Co exciting source and a germanium semiconductor detector is suitable. In this report we do not discuss this second combination.

PROCEDURE FOR ENERGY AND RESOLUTION CALIBRATION

Before using the spectrometer for analysis of an unknown sample it is useful to excite characteristic x rays in a series of pure metallic foils or chemical compounds in order to determine proper gain settings for the amplifiers, to calibrate the x-ray energy range of interest, and to determine the resolution characteristics over this energy range. These preliminary calibration steps are most necessary when a mixture of elements is to be determined.

In preparation for the barium analyses to be discussed below, calibration spectra were taken on more than 10 elements between vanadium and neodymium. Some of these are shown in Figures 4 and 5. From these spectra the calibration curve of Figure 6 was obtained. X-ray energies of the calibrating elements were taken from published tables and plotted at the corresponding peak channel. The derived curve is linear with a slope of 0.233 k.e.v. per channel.

The channel width at half the peak height was determined for the peaks in the x-ray spectra and converted to an energy resolution value referred to as full width at half maximum (FWHM). For our particular spectrometer under the particular operating conditions we used, the FWHM had the variation shown in Figure 7. It is useful to compare this curve with another plotted in the same figure showing separation of K_{α} x rays of neighboring elements as a function of atomic number. We call attention to two features. First we note that the slope of the FWHM curve is considerably less than that of the curve for the separation of neighboring elements. This means that x rays of neighboring elements of heavier elements are much more easily distinguished. Since a noticeable separation of two x-ray peaks present in roughly equal intensity should be observed when their energy separation is about equal to the resolution of the spectrometer, we can conclude that we can expect to separate x-ray contributions from neighboring elements in the rare earth region but not in the region of iron, cobalt, and nickel. In the latter case elements differing by two in atomic number can be distinguished. These conclusions are illustrated in Figures 8 and 9. In Figure 8 there are shown spectra taken on 100 milligram samples of the neighboring rare earth elements lanthanum and cerium, and on a 3 component mixture of 50 mg. each of lanthanum, cerium, and praseodymium. In Figure 9 x-rays of the neighboring elements iron, cobalt, and nickel are presented.

The separations achieved in the spectra shown in Figures 8 and 9 are limited by our resolution figure of 1.1 to 1.5 k.e.v. But improved resolution can be expected with different semiconductor detectors and pre-amplifier circuits. Elad, for example, reports a resolution figure of 0.7 k.e.v. for x rays of platinum in a germanium detector (3). Bowman and Jared (13)

at 14 k.e.v.

have achieved a resolution of 0.6 k.e.v./with a silicon crystal. In Figure 7 we have drawn lines at the level of 0.7 k.e.v. and 0.4 k.e.v. which are useful for estimating the ability of better detectors to distinguish the x rays of neighboring elements.

THICK SAMPLE ANALYSIS OF BARIUM SAMPLES IN 1-10% RANGE

Introduction. We chose barium-containing mixtures to demonstrate the performance of our spectrometer for a sought element in the 1-10 percent concentration range. We prepared a series of samples containing a known weight percentage of barium by mixing barium chloride and sodium borate powders. The latter was chosen as a representative matrix of light element matter. The matrix effects are quite important but there are no features which pose significantly different problems for the semiconductor detector compared to other X-ray detectors.

The samples prepared for analysis weighed several grams and were infinitely thick compared to the mass absorption coefficient of barium K X-rays. We proceeded in the usual manner to measure a calibration curve of count rate for the X-rays of the sought element versus the content of that element in the sample.

Sample Preparation. Quantities of Reagent grade $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ and sodium tetraborate $\text{Na}_4\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ were ground to powders in separate clean porcelain mortars. Samples of each were carefully weighed, placed in an Erlenmeyer flask and thoroughly mixed by 10-minutes shaking. One hundred gram mixtures of eight different weight percentages of barium were prepared.

Portions of this powder were taken and pressed into plastic bottle caps of 2.6-cm. diameter. The powder was leveled with a spatula and covered with thin Saran sheet which was twisted and held at the back of the cap with Scotch tape. When the bottle cap sample was mounted vertically in front of the ^{241}Am source the fine powder made a uniform surface against the Saran. The total weight of the samples was about 7 grams but was not determined. Replicate samples of each weight concentration were prepared.

Counting Procedure--Checks on Geometric Reproducibility. The bottle-cap samples were placed vertically in a simple cardboard holder in front of the beryllium window of the detector housing. See Figure 2. The front surface of the barium powder sample was 3 mm. away from the front edge of the lead shield on the ^{241}Am and 10 mm. from the Be window. Numerous runs were made on a single sample with slight adjustments of its position forward and backward and upward and downward in order to determine the sensitivity of the count rate in the barium K X-ray peak to the positioning of the sample. The count rate was somewhat sensitive to the positioning of the sample, but within the setting which could easily be reproduced it was constant within two percent. This error could certainly be reduced below 0.5 percent with a carefully designed sample holder and a redesigned ^{241}Am shield. Since our detector was in use mainly for nuclear spectroscopy unrelated to X-ray analytical work we did not have complete freedom to make and test these modifications.

Once the sample was in position and suitable adjustments made on the amplifier gain controls, the pulse-height analyzer was started and data on the X-ray spectrum were collected for a certain period, typically one minute. After

the counting period the contents of the memory were printed out on paper tape and the total count in the K_{α} barium X-rays peak was determined. Figure 10 shows a typical spectrum taken at a barium concentration of 2 percent. A background correction was made by counting a blank sample of sodium borate prepared and mounted in an identical fashion. The background count under the barium K X-ray peak was 400 counts per minute which was negligible compared to the true peak for all concentrations above one percent. The necessary arithmetical operations could be done by hand or greatly simplified by use of the various convenience features built into commercial pulse-height analyzers.

Results. Table 1 summarizes data on replicate samples at all concentrations of barium. The error for each group varies from 0.6 to 3.8. The main contributions to this are positioning error and mixing (sample) error in the preparation of the $BaCl_2$ -borate mixtures. Counting error including background correction is not the major contribution. The calibration curve derived from these data is presented in Figure 11. With the aid of this curve it would be possible to determine the barium content of an unknown sample, in which the matrix was similar to the one used here, to an accuracy of a few percent in a few minutes. By more careful control of sample preparation and mounting, the accuracy could be increased to the point where statistical count errors would be limiting. Our calibration curve runs down to one percent but it could be extended substantially below this concentration even without increasing the ^{241}Am intensity.

TRACE ANALYSIS OF MILLIGRAM-SIZE SAMPLES

Introduction. When the concentration of a sought element drops below a certain value, analysis by X-ray emission becomes difficult or impossible without preliminary separation of the desired element or elements. In such cases one standard technique is to isolate the element and to analyze it with an X-ray spectrometer in samples so thin that only part of the beam of exciting radiation is absorbed. In thin samples the response is linear, and matrix effects are small or absent. We illustrate the behavior of our spectrometer for such a case by using yttrium as a prototype element.

Preparation of Samples. A pure stock solution of yttrium at a concentration of 10 mg per ml was prepared by weighing a sample of 99.9 percent yttrium chloride, dissolving it in HCl, and diluting to volume in a 100-ml. volumetric flask. Measured aliquots of this solution were pipetted into test tubes and mixed with ammonium hydroxide to precipitate yttrium hydroxide. This compound was filtered onto a 3/4-inch filter paper (Wattmann No. 40) and air dried at 120°C. The dried filter paper was covered with a 0.0025-inch Scotch-brand tape and mounted in the center of a 1-1/4-inch hole punched in a 2-1/2 × 3-1/2 inch piece of aluminum stock. This aluminum holder was slipped into a notched stand which allowed the yttrium hydroxide sample to be positioned reproducibly in front of the beryllium window of the counter housing. The gain controls on the amplifiers were set at appropriate values and the pulse-height analyzer was turned on for a period of several minutes. The collected data were then printed out and the total count in the K_{α} and K_{β} peaks of yttrium was determined. Then the sample was removed and a blank piece of filter paper was mounted in the same sample holder and counted

for the same period of time. The counts in the region of the yttrium K X-rays were totaled and subtracted from the previous integrated total. The appearance of the spectrum and the extent of the background correction are illustrated in Figure 12. The bumps in the background are caused by Compton scattering of Neptunium L X-rays from the filter paper.

Results. Table 2 lists results of replicate samples together with estimate of errors. Figure 13 shows the calibration constructed from these results. The curve is linear up to about 5 mg. yttrium. Above this point there is some self absorption of yttrium X-rays. Errors in sample preparation are approximately 5% and those in positioning the sample about 2%. These could be reduced by a factor of 5 or 10 by more careful technique. Counting error at the one-mg level is 1% for 7-minute counting periods on sample and blank. This error could be reduced by use of a more intense ^{241}Am source or by use of a larger semiconductor detector, or by somewhat different geometrical relationships of ^{241}Am source, specimen, and detector. Background radiation could be reduced by supporting the samples on mylar film or other material instead of filter paper. Even with the techniques and apparatus described here yttrium could have been detected at a considerably lower level.

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- (11) We used a Radiation Industries Development Laboratory Model 34-12 Analyzer. Comparable instruments manufactured by Technical Measurements Corporation, or other manufacturers would have served as well.
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FIGURE CAPTIONS

Fig. 1. Schematic arrangement of apparatus for excitation and measurement of characteristic x radiations of an analytical sample. An encapsulated nuclear γ source (^{241}Am , ^{57}Co or other nuclide) is positioned near the specimen to excite x rays in the specimen. A fraction of these characteristic x rays reaches the lithium-drifted silicon or germanium semiconductor diode and produces an electrical charge. A field effect transistor (FET) serves as the first stage of a charge sensitive preamplifier. The preamplifier output is linearly amplified and the pulse height is measured and recorded in a multichannel pulse-height analyzer. That part of the apparatus outlined in dashed lines is in a vacuum chamber and is maintained at low temperature.

Fig. 2 Experimental arrangement of ^{241}Am source, lithium-drifted silicon detector, and specimen to be analyzed. FET signifies field effect transistor used as first element of charge-sensitive preamplifier.

Fig. 3. Gamma and x radiation emitted by ^{241}Am . This spectrum was taken by the silicon semiconductor detector with source shining directly into detector. A 2 mm. thick plastic cover absorbed the alpha radiations. Intensities per α disintegration of these radiations are as follows: 59.57 k.e.v. γ -0.36, 26.36 k.e.v. γ -0.025, 13.96 k.e.v. x ray-0.12, 17.76 k.e.v. x ray-0.13, and 20.80 k.e.v. x ray-0.03.

Fig. 4 Calibration spectra taken separately on x-rays of copper, silver, and cerium. Energy width at half maximum of K_{α} peak is shown.

Fig. 5. Calibration spectra taken separately on x-rays of nickel, molybdenum, iodine and neodymium.

Fig. 6. X-ray energy versus channel number of pulse height analyzer as derived from calibration spectra.

Fig. 7. Energy resolution expressed as full width at half maximum (FWHM) versus x-ray energy (and atomic number of element having K_{α} x-rays of that energy) for this spectrometer. Lines are drawn in at 0.7 k.e.v. FWHM to show performance achieved to date with certain semiconductor detectors and at 0.4 k.e.v. Also shown is the energy separation of the K_{α_1} x-ray of neighboring elements and elements differing by 2 in atomic number plotted against the atomic number of the lower member of the pair.

Fig. 8. X-ray spectra excited in lanthanum (element 57), cerium (element 58), and praseodymium (element 59) by ^{241}Am . Curve 1 is for 100 mg La_2O_3 powder in plastic tube placed a few millimeters in front of ^{241}Am source. Counting time about 2 minutes, no background subtracted. Curve 2 is for 100 mg CeO_2 under same conditions. Curve 3 is for 50 mg each of La_2O_3 , CeO_2 , Pr_2O_3 . Amplifier gain settings different from Figures 4 and 5.

Fig. 9. K x-rays excited separately in gram-sized samples of Fe, Co, and Ni. Upper curve shows x-ray spectrum excited in mixed sample of iron and nickel chlorides.

Fig. 10. Typical spectrum excited in standard mixture of BaCl_2 and sodium borate. This curve shows 5 min count on sample with 2% barium content. For this run the ^{241}Am radiations were filtered through .01 inch copper and .03 inch aluminum. Bump at channel 125 reveals unexpected presence of iodine in the BaCl_2 .

Fig. 11. Calibration curve showing count rate of barium K_{α} x-rays versus weight percent barium in thick powder samples.

Fig. 12. Yttrium K x-rays excited in 7.5-mg sample mounted as $Y(OH)_3$ on filter paper in five-minute count. Background shows traces of back scattered peaks from Np L x-rays in ^{241}Am source.

Fig. 13. Calibration curve showing count rate of yttrium K x-rays versus milligrams of yttrium.

Table I. Count Data on Barium K_{α} x-rays

Wt. % Ba	No. of Samples	Count Time	Integrated count over Bkg in Ba K x-ray peak* (counts/min)	Standard (counts/min)	Deviation %	Counting error in- cluding Bkg (counts/min)
56 [#]	2	1 min	137,800	1770	1.3	380
10	3	1 min	111,700	934	0.8	335
9	3	1 min	106,700	1900	1.8	327
6	3	1 min	86,000	1240	1.4	293
5	4	1 min	76,500	1350	1.8	277
4	3	1 min	67,130	2120	3.2	259
3	3	2 min	54,110	1500	2.8	110
2	3	2 min	41,900	230	0.6	145
1	3	4 min	23,000	865	3.8	79

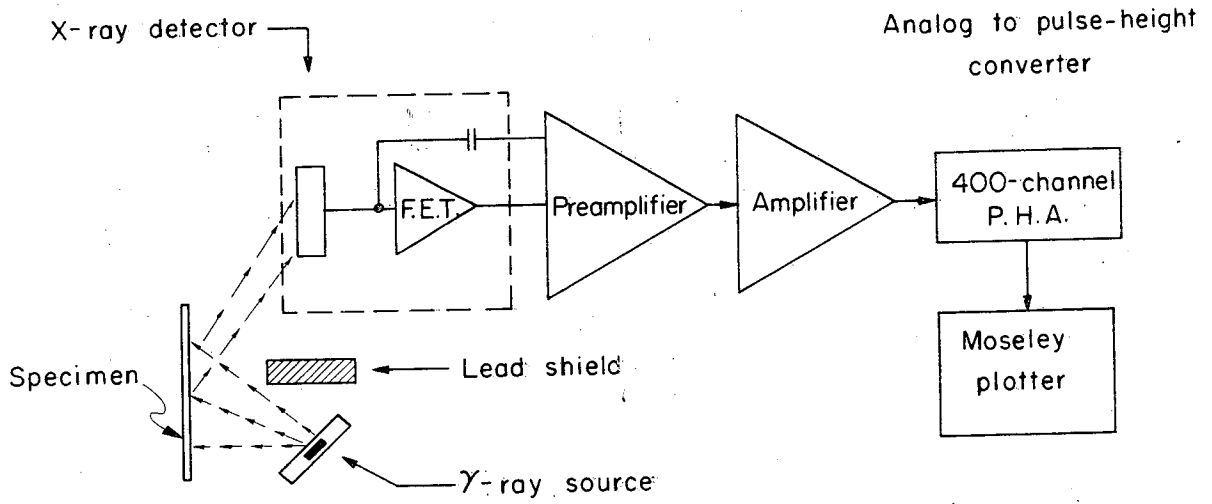
* Background under Ba K_{α} peak was 400 c/min.

[#] These samples were pure BaCl₂ · H₂O.

Table II Count Data on Yttrium K x-rays

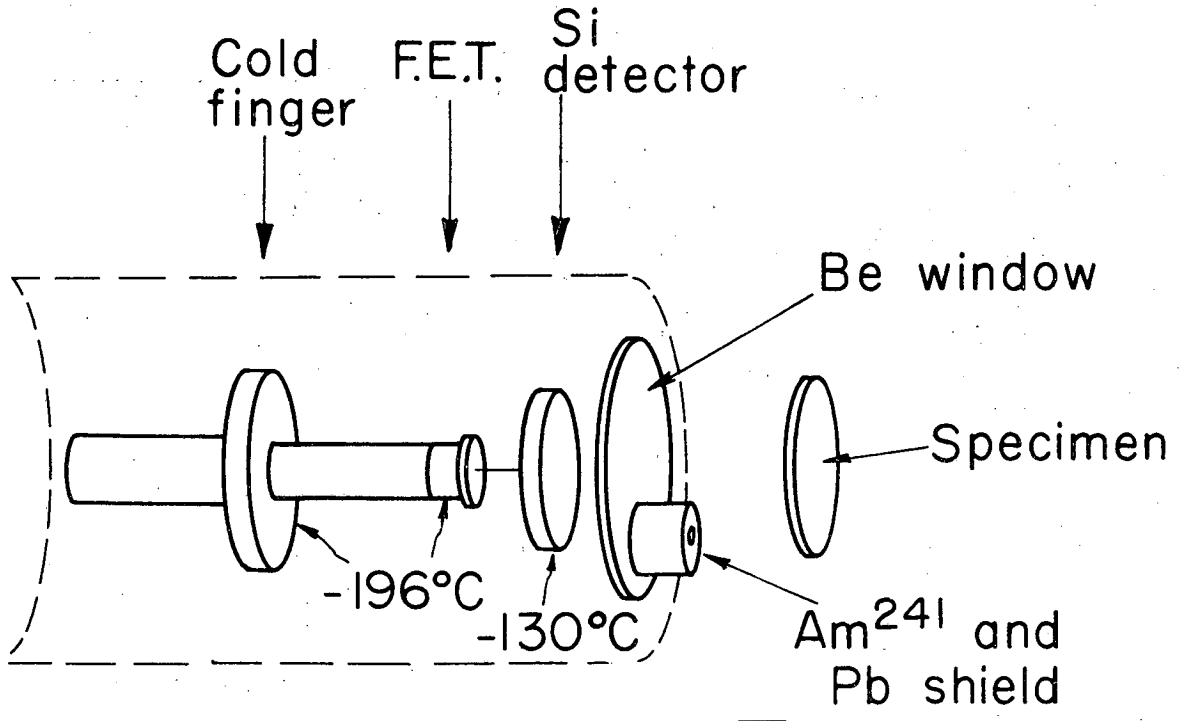
Mg Yttrium in sample	Number of sample	Count time (min)	Average counts/min in Yttrium K x-ray peak above back- ground*	Standard		Statistical count error count/min
				Deviation	%	
10	5	10	18,300	330	1.8	43
7.5	1	10	15,280	---	---	40
5.0	2	10	11,600	30	0.3	35
2.5	2	10	5,610	130	2.4	25
1.0	5	10	2,230	115	5.1	17
0.5	3	20	810	95	11.7	8

* Background from blank run under Y x-ray peak = 270 c/min.



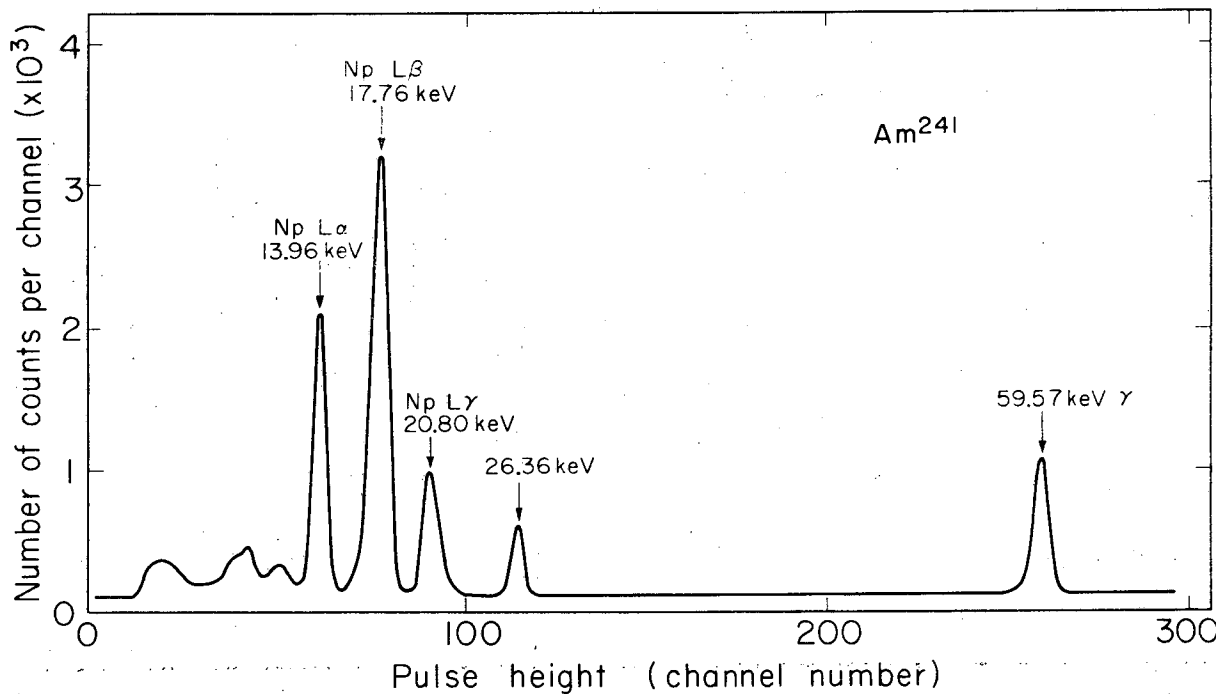
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Fig. 1



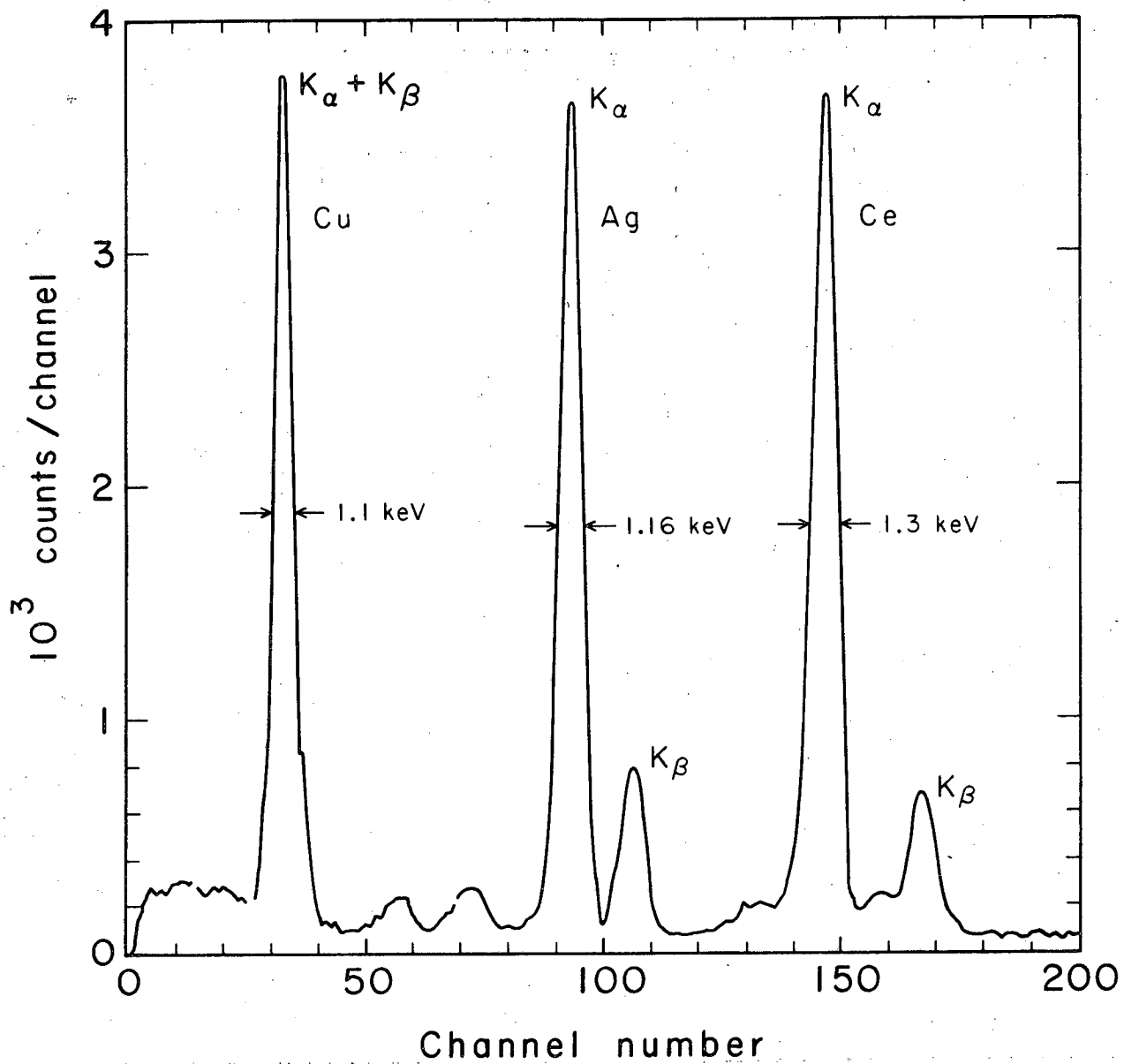
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Fig. 2



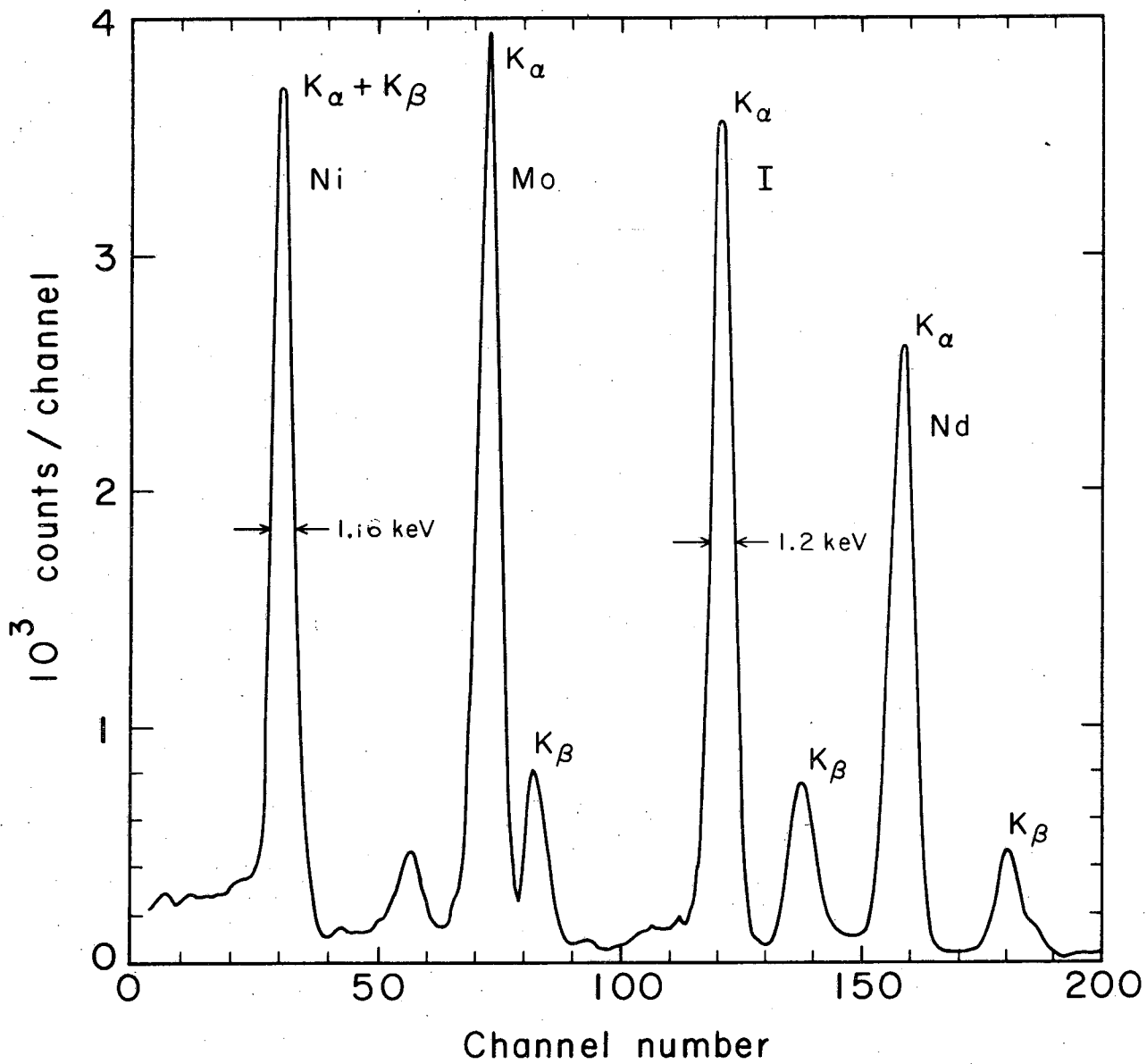
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Fig. 3



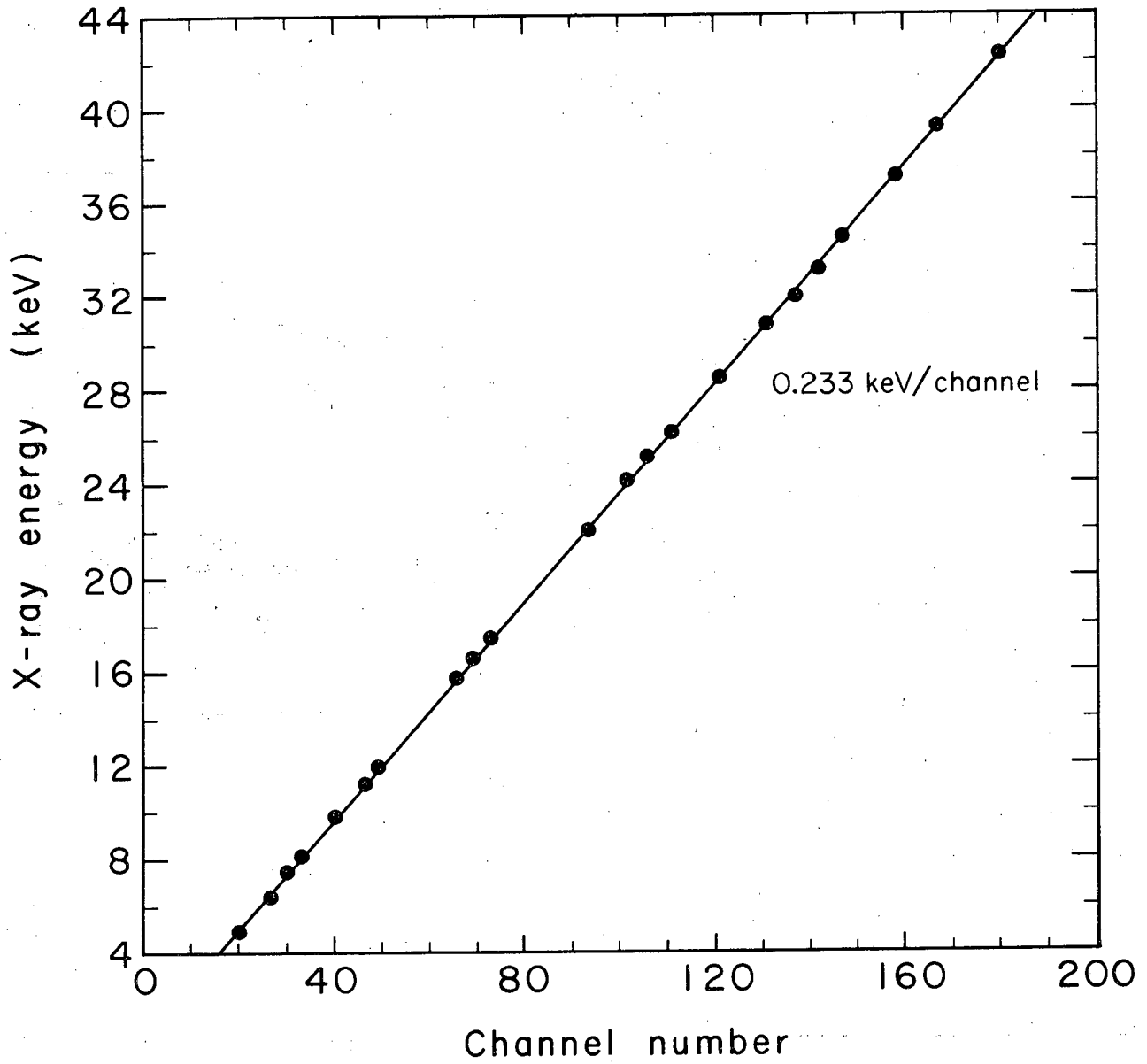
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Fig. 4



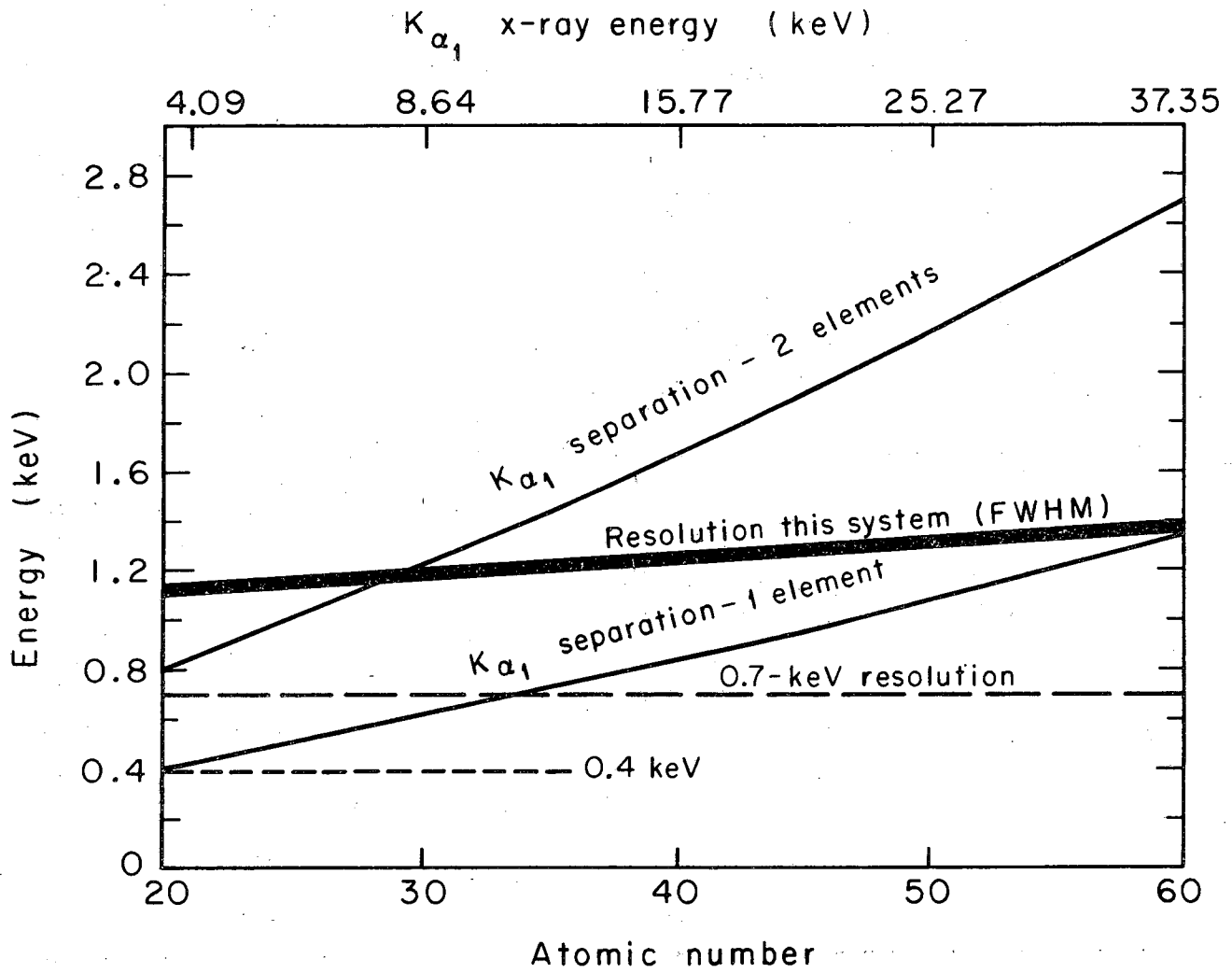
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Fig. 5



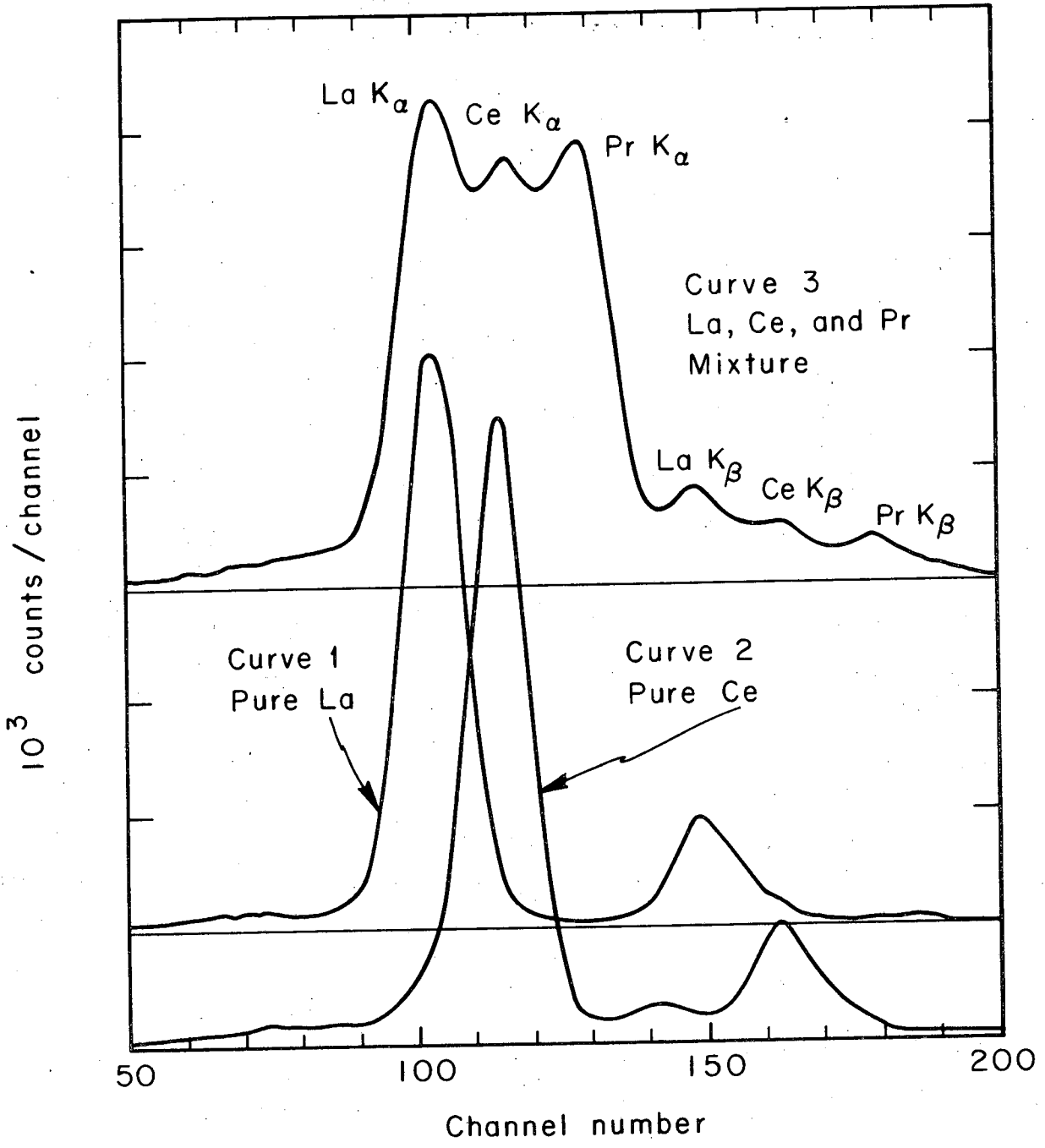
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Fig. 6



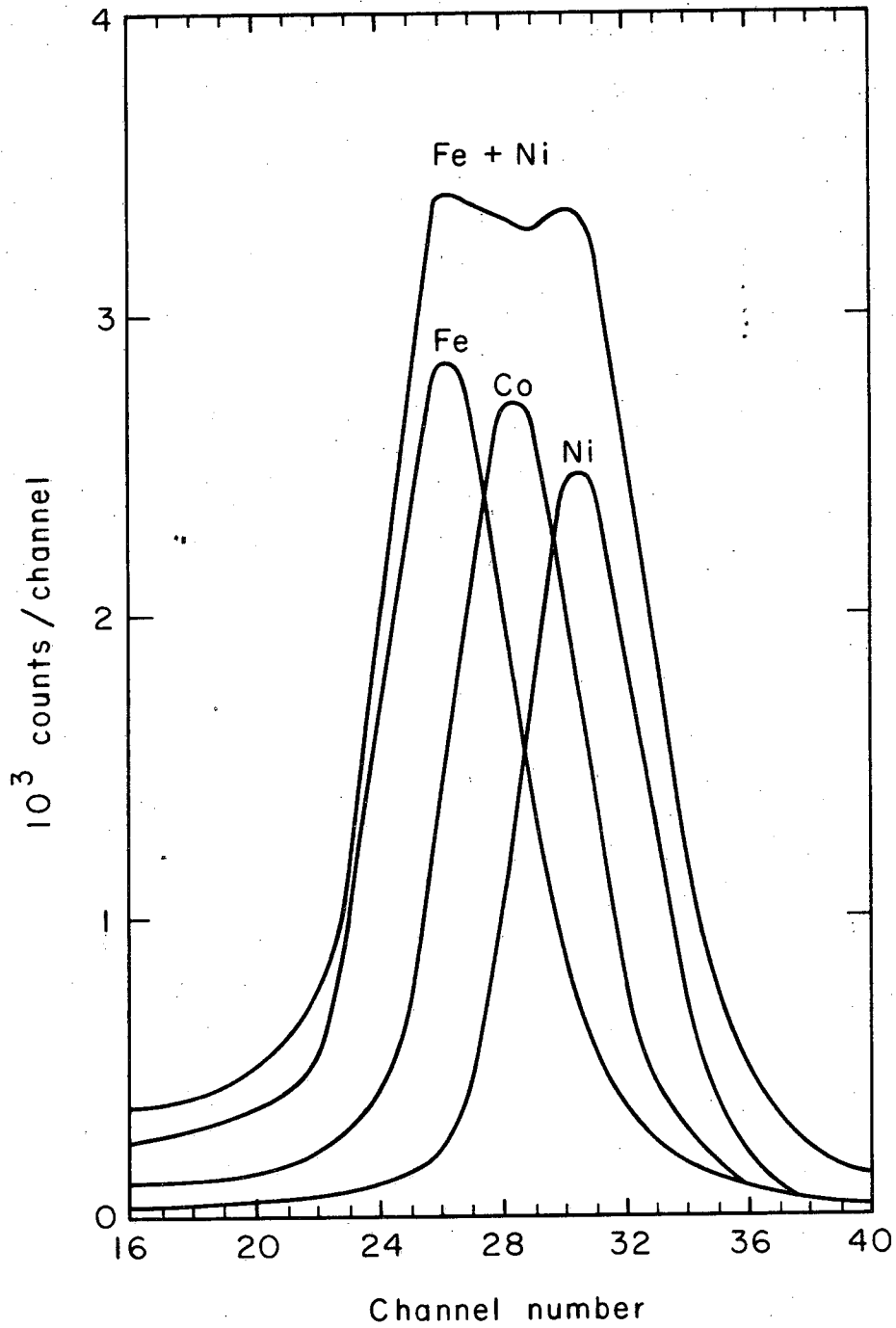
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Fig. 7



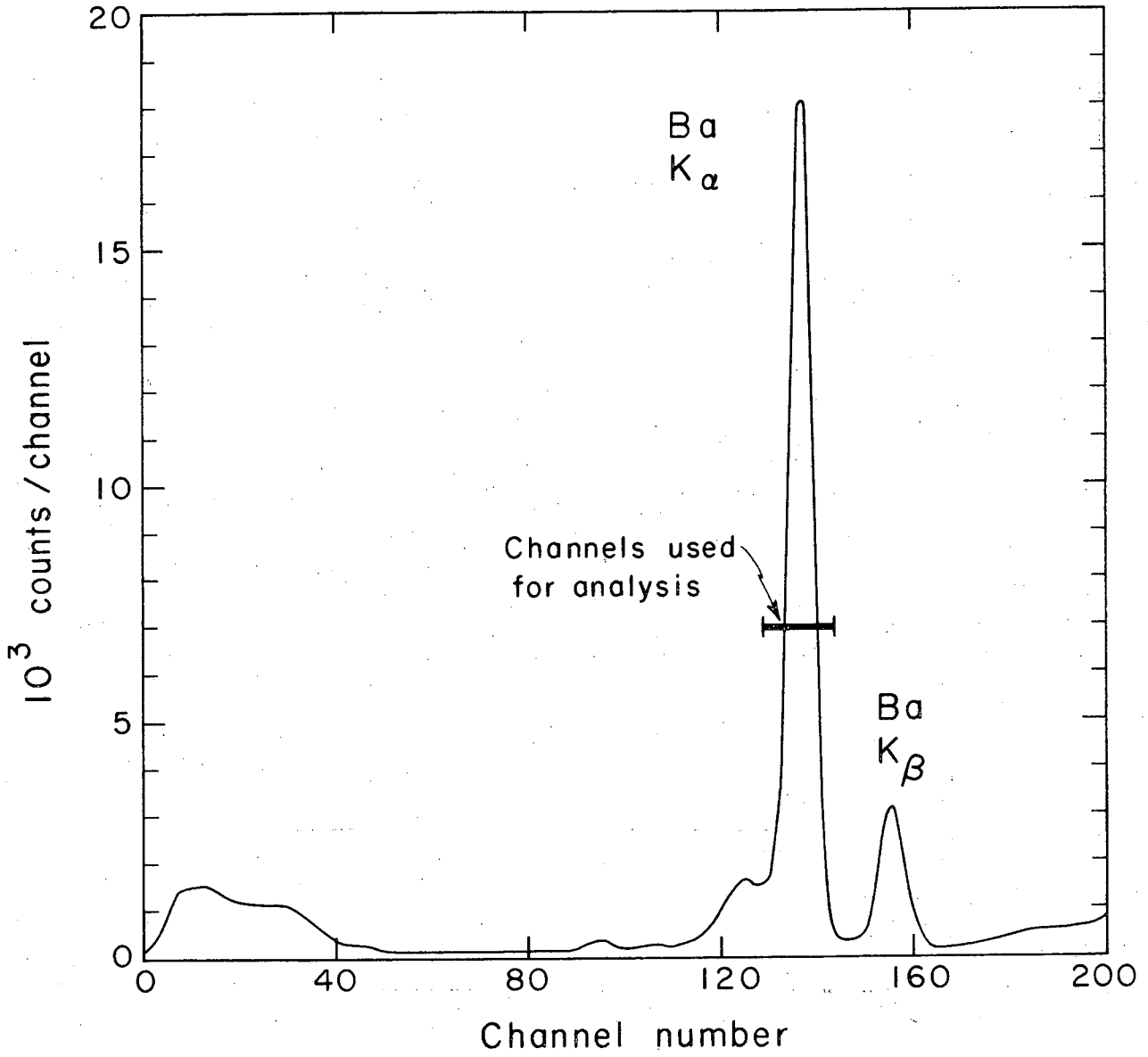
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Fig. 8



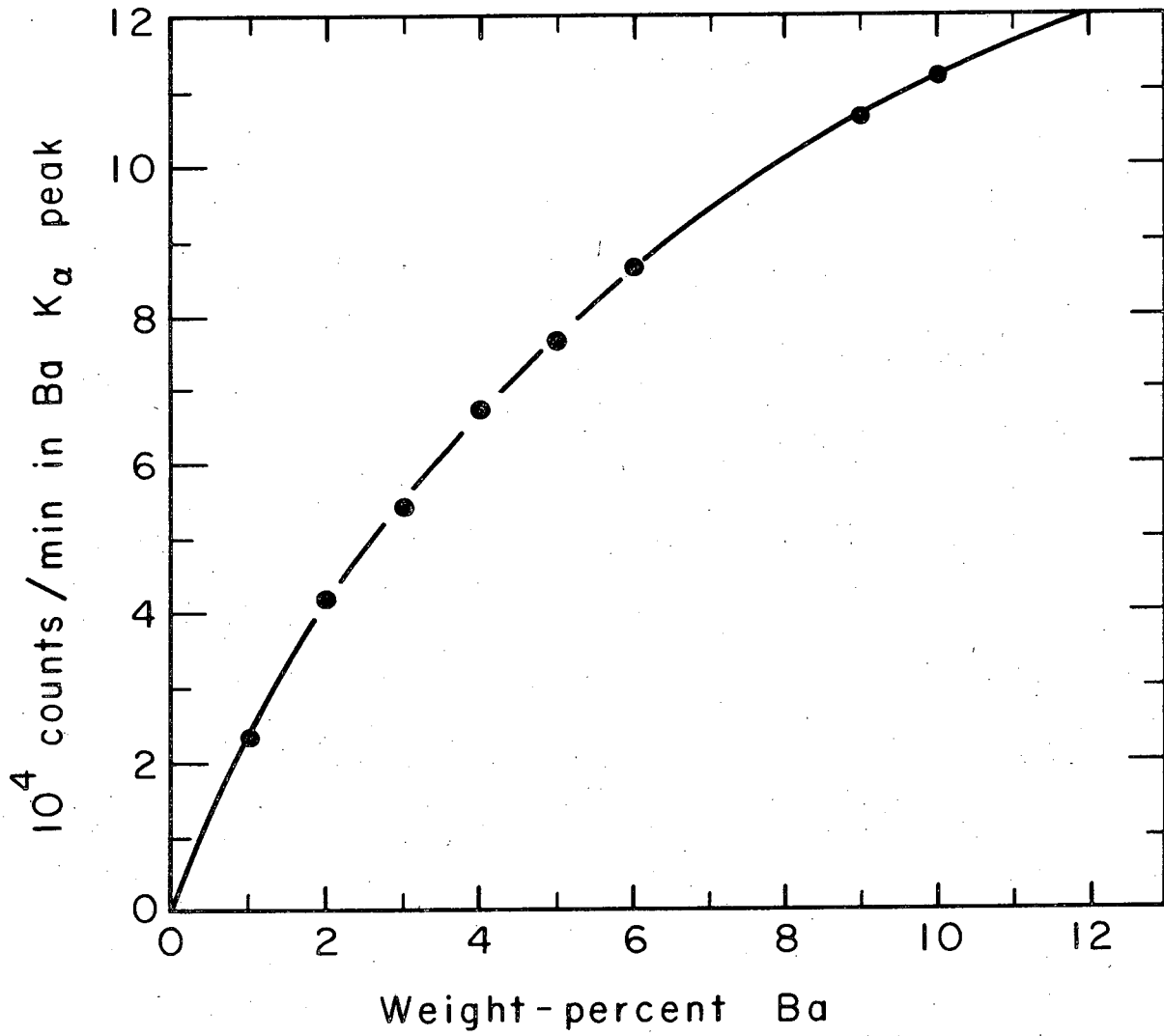
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Fig. 9



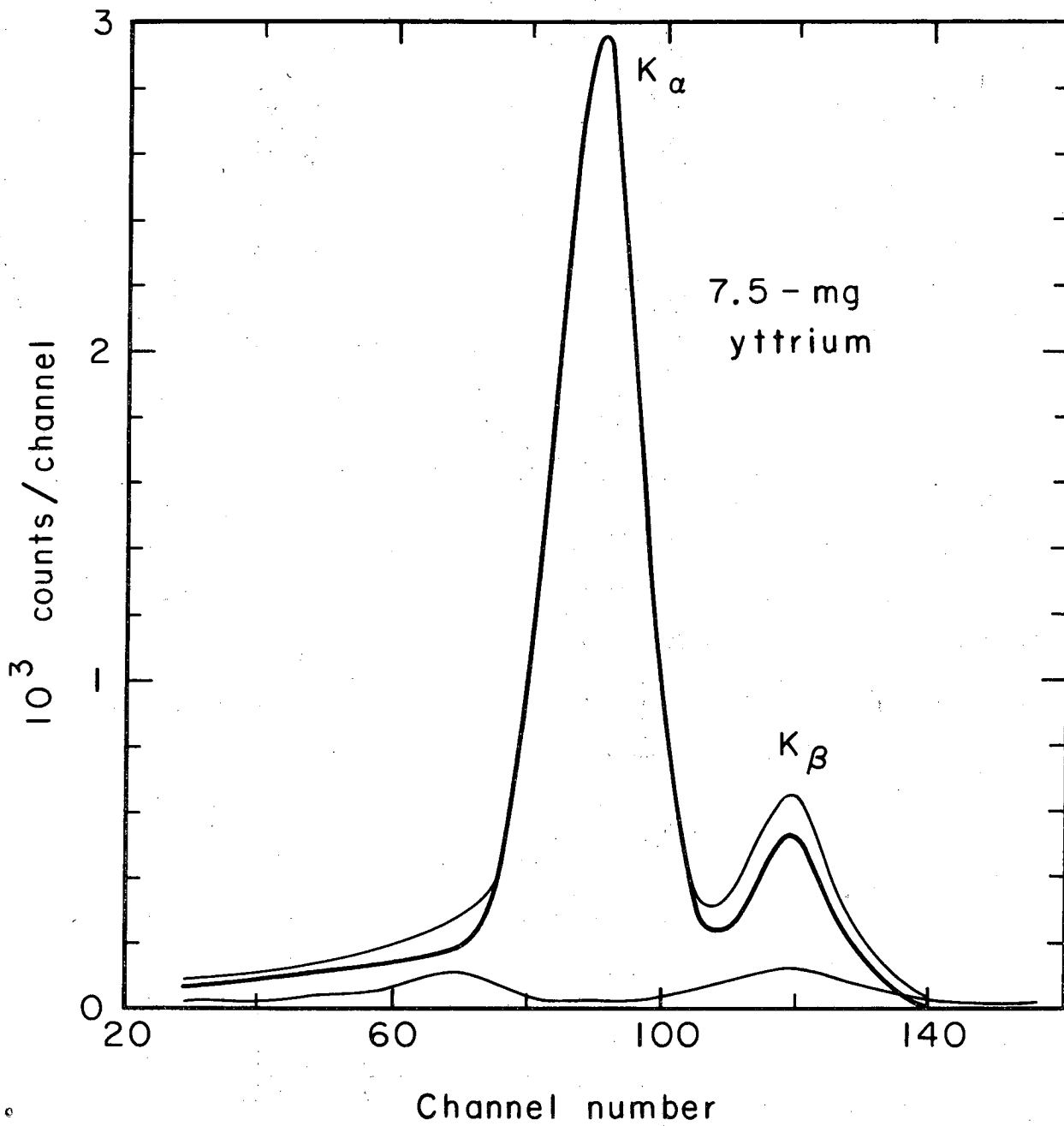
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Fig. 10



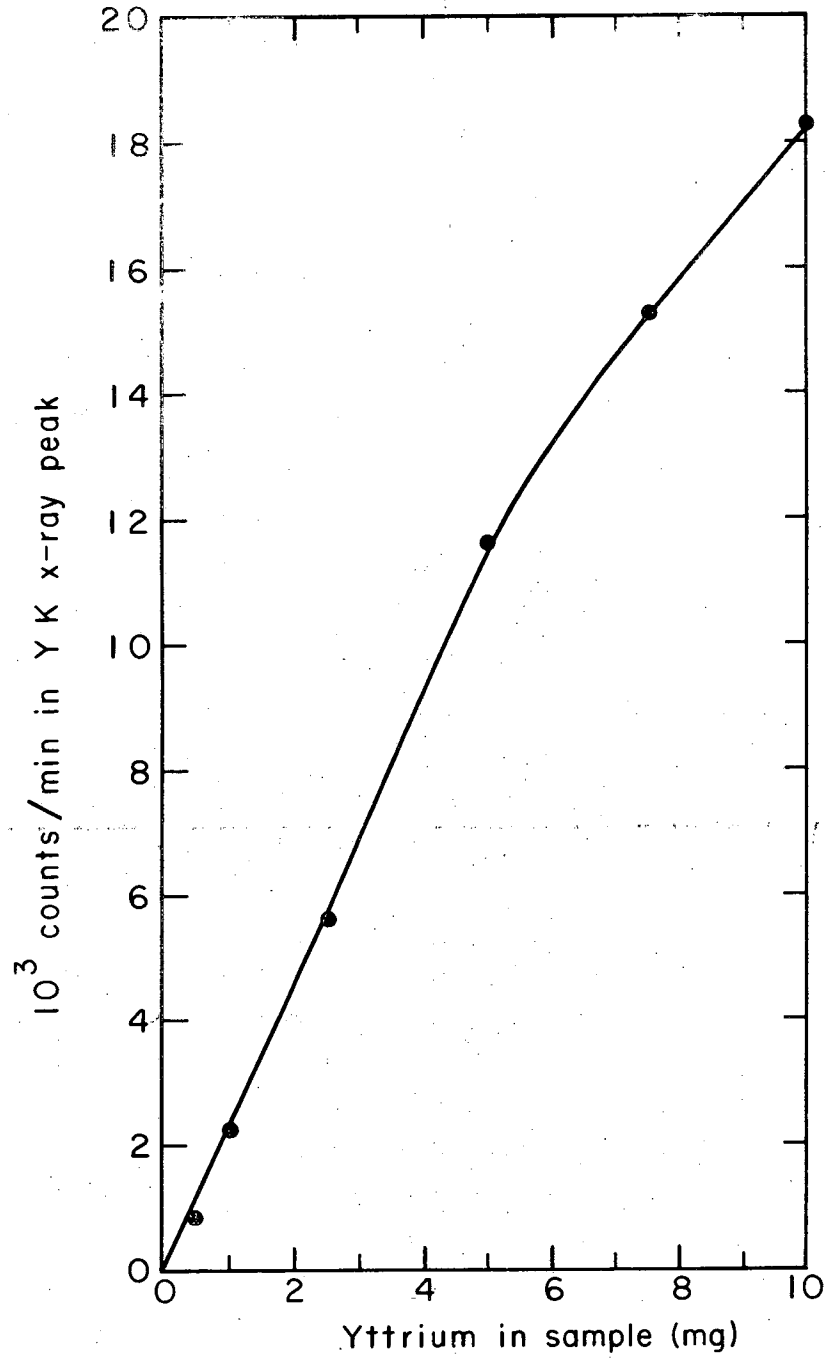
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Fig. 11



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Fig. 12



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Fig. 13

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