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A
STUDY OF THE HAEFFNER
EFFECT IN MERCURY

BY

JAMES NICHOLSON BAYSDEN

A

THESIS

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Wm A. Frad

ABSTRACT

In 1953, E. Haeffner (1) reported that when he passed a direct electric current through a capillary tube filled with mercury, he obtained a separation in the isotopes of mercury with the heavier isotopes being enriched at the negative terminal and the lighter isotopes at the positive terminal. This effect has since been referred to as the Haeffner Effect. Since the original experiment with mercury, other investigators have observed this effect in many other liquid metal systems.

This investigation of the Haeffner Effect in mercury involves the use of a radioactive isotope of mercury (Hg 203) as a tracer to follow the separation of the isotopes as it occurs in a capillary system. The capillary system used in this investigation was constructed so that no reservoir was attached to the system thereby allowing an observation as to whether equilibrium conditions existed in a relatively short time compared to other investigations of the Haeffner Effect.

ACKNOWLEDGEMENT

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The author is also grateful to Dr. A. H. Larson and Professor R. V. Wolf for their consultations and their aid in obtaining the equipment for this investigation.

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I. INTRODUCTION

In 1953 it was successfully demonstrated that an isotopic separation in a liquid metal could be obtained by passing a direct current through a capillary tube containing the liquid metal. Since then, this phenomenon has been observed in many liquid metals and once in a solid metal. In all of these investigations it was demonstrated that the lighter isotopes were concentrated in the direction of electron flow, while the heavier isotopes were concentrated in the direction opposite to that of electron flow. With a few exceptions these investigations were conducted in a system in which a reservoir was attached to the capillary tube, either in the middle of the tube or at one end or both ends of the tube. In each case the presence of the reservoir, with its comparatively large volume of liquid metal, made it difficult to obtain equilibrium in the system.

It is the purpose of this investigation to determine:

(1) If equilibrium may be attained in a relatively short time, compared with the 50 to 120 days of previous investigations, in a capillary tube to which no reservoir has been attached.

(2) If a radioactive isotope can be used as a tracer to determine the separation of isotopes by successive measurements of the activity of the mercury at positions along the length of the tube. As was the case in the majority of the previous investigations, this one will be conducted in a bath held at constant temperature.

II. REVIEW OF LITERATURE

The first successful isotopic separation in a liquid metal was accomplished in 1953 by E. Haeffner (1). In his investigation the liquid metal used was mercury. The system used by Haeffner consisted of a capillary tube, 14 centimeters in length and an inside diameter of 2 millimeters, filled with small glass beads. One end of the tube was sealed with an electrode in place, while the other end consisted of an open reservoir. The tube, when filled with mercury, was placed in a vertical position in a bath whose temperature was maintained at 15° C. An average current of 22.6 amperes at 4.3 volts was passed through the mercury for 2000 hours. Immediately after the current was turned off the reservoir was emptied and its contents analyzed by mass spectrometer. The polarity of the electrodes was reversed and the electrolysis repeated. Results of the electrolysis with the reservoir as the anode are given in Table 1, and the results with the reservoir as the cathode are given in Table 2. The results of the electrolysis with the reservoir as the anode showed an increase in concentration of lighter isotopes, while the electrolysis with the reservoir as the cathode showed an increase in concentration of the heavier isotopes, illustrating that the lighter isotopes were concentrated in the direction of electron flow, while the heavier isotopes were concentrated in the direction opposite to that of electron flow.

Since the original experiment, the effect has been observed and reported in liquid metals by several investigators and, in one instance, was reported in a solid metal. Additional studies of mercury have been made (2, 3, 4), as well as studies of molten gallium (5, 6), rubidium (7, 8), lithium (9), potassium (8, 10), indium (11), zinc (12), cadmium (12), and tin (12), and in solid uranium (4). With a few exceptions these investigations were

TABLE 1

Mass Spectrographic Analysis of the Anode Volume

Relative Abundance

Mass No.		Control Sample	Anode Sample	Change in Relative Abundance
198		10.10%	10.35%	2.5 ± 0.3%
199		17.05	17.35	1.76 ± 0.2
200		23.25	23.40	0.6 ± 0.2
201		13.27	13.22	-0.4 ± 0.2
202		29.60	29.15	-1.54 ± 0.2
204		6.67	6.44	-3.45 ± 0.4
	Total	99.94%	99.91%	

TABLE 2

Mass Spectrographic Analysis of the Cathode Volume

Relative Abundance

Mass No.		Control Sample	Cathode Sample	Change in Relative Abundance
198		10.06%	9.68%	-3.8 ± 0.4%
199		17.03	16.78	-1.5 ± 0.3
200		23.32	23.20	-0.5 ± 0.3
201		13.27	13.35	0.6 ± 0.3
202		29.60	30.10	1.7 ± 0.2
204		6.66	6.94	4.2 ± 0.4
	Total	99.94%	100.05%	

made in systems similar to that of Haeffner's with reservoirs attached to the capillary tube.

Since the original experiment, there have been several mechanisms proposed to explain the effect of isotopic concentration by a direct current. Although none of these theories have gained unanimous support, a few of the most prominent will be discussed.

The first theory advanced was proposed by Klemm (13). Starting with the assumption that a metal consists of a mixture of immobile and mobile ions and electrons, he proposed that, as part of an orderly lattice arrangement, the immobile ions were less likely to suffer as many interactions with the conduction electrons as the disorderly arranged mobile ions. Thus, as a result of numerous interactions, the mobile ions were concentrated towards the anode. Among these mobile ions were both light and heavy isotopes, but, as a result of their greater degree of ionization, the lighter species interacted more strongly with the electrons and were, therefore, concentrated in the direction of electron flow.

Another mechanism proposed for the Haeffner effect was that of Bresler and Pikus (14). In their treatment Bresler and Pikus proposed that, since the vibrational frequency of a light isotope of a given element is greater than that of a heavy isotope, the "radius" (wave function) of the lighter isotope is larger than that of the heavier isotope. Therefore, the lighter isotope interacts to a greater degree with its neighbors than the heavier isotope. Assuming then that the net flux of material is towards the cathode or in the direction of the electric field, the heavier isotope would be concentrated in this direction, because of its smaller degree of interaction and, therefore, greater mobility.

A somewhat different treatment was given by Jacobs (15). In the mechanism proposed by Jacobs, the basic approach was similar to that of

Bresler and Pikus in that a negative free energy gradient was proposed and that the diffusion rate was proportional to the vibrational frequency of the ions. However, Jacobs proposed that the negative free energy gradient was in the direction of the positive terminal and that since the vibrational frequency was inversely proportional to the mass of the oscillator, the rate of diffusion toward the positive terminal would be greater for a lighter isotope than for a heavier isotope. As a result, there would be a concentration of the lighter isotopes in the direction of the positive terminal and a corresponding enrichment of the heavier isotopes in the direction of the negative terminal.

Another mechanism proposed was that of de Gennes (16), which considered the relationship between the coefficient of diffusion and viscosity, as well as the mean frequency of oscillation of an ion and the energy transitions of free electrons whereby they impart energy to the ions of the liquid metal. Although, de Gennes predicts that the lighter isotopes have a greater effective cross section and will be carried along in the direction of electron flow, calculations based on his proposals give a considerably lower value of separation than has been found experimentally.

E. Haeffner, T. Sjoborg, and S. Lindre (4) studied the separation of isotopes in mercury by a direct current in a system in which the capillary tube was enclosed in a water-jacket to maintain a constant temperature. The capillary was 17 centimeters long with an inside diameter of 1 millimeter and was filled with small glass beads. When filled, the anode volume contained 18,230 milligrams of mercury. The capillary was kept at constant temperature, 47.0 ± 0.5° C, while a current of 4.3 amperes at 5.8 volts was passed through the mercury for 42 days. The tube was divided into 1 centimeter sections and the contents of each section analyzed by mass spectrograph. The results of the analysis showed that the maximum enrichment of mercury (198) to mercury (204) was 1.608, which occurred near the anode. An unsuccessful attempt was made by these investigators to measure the isotopic enrichment in a 1 millimeter uranium wire.

Investigation of isotopic separation in mercury by I. V. Bogoiavlenskii and coworkers (2) at 41° C and -10° C led to their conclusion that the process was temperature dependent and greater at 41° C than at -10° C. Their results also showed that a saturation of separation occurred at some characteristic time and further current flow failed to increase separation.

R. E. Croy (3) conducted an investigation of the separation of mercury isotopes at several current densities. The stable mercury was tagged with radioactive mercury (203), and the separation was to be determined by counting the activity of the mercury at positions along the capillary tube. In his investigation Croy used a capillary system in which reservoirs at each electrode were inherent in the design of the system. The volume of each reservoir was near 0.13 milliliters. Although Croy succeeded in obtaining isotopic separation in part of his investigation, these reservoirs were cited as being mainly responsible for the failure to detect isotopic separation by the counting method. When the capillaries used in part of the investigation were broken in half and the contents analyzed, the results showed that the relative change in isotopic concentration in the cathode half was 8.6% after passing 1507 A/cm^2 for 50 days and 9.7% after passing 3312 A/cm^2 for 60 days.

M. Goldman, F. Nief, and E. Roth (6) investigated the effect of temperature on isotopic separation in liquid gallium and found that the separation factor defined as

$$P = \frac{\Delta\left(\log \frac{C_1}{C_2}\right)}{\Delta V} \qquad (1)$$

increases from 0.049 at 325° K to 0.077 at 560° K, where P = separation factor, C_1 = original concentration, C_2 = final concentration, and V = voltage.

An investigation of separation in rubidium was made by Lodding (7), and the separation was again shown to be a function of temperature. However, the separation in this instance increases through 415 ± 6°K, then decreases sharply through 538 ± 12°K.

A. Lunden, A. Lodding and W. Fischer (9) studied the effect of isotopic separation in molten lithium by a direct current utilizing stainless steel as a capillary tube. The investigation was conducted at 300° 10° C with a direct current of 37 amperes for 159 hours. Their results showed that the ratio of Li (6) in the anode to Li (6) at various positions along the capillary decreased from 1.27 ± 0.03 near the cathode to 0.97 ± 0.03 near the anode.

A. Lunden, C. Reutersward and A. Lodding (10) passed a direct current of density $2900 A/cm^2$ through a 40 centimeter capillary of molten potassium for 14.8 days. Their results showed an enrichment of K (41) at the cathode and an enrichment of K (39) at the anode with a separation factor of 1.12 between the anode and the cathode.

Similar investigations with similar results have been conducted with indium (11), zinc (12), cadmium (12), tin (12) and in solid uranium (4).

III. EXPERIMENTAL PROCEDURE

The predominant reasons for choosing mercury as the metal to be investigated are as follows: (1) mercury is in the liquid state at room temperature, and, since the investigation was to be conducted in a capillary tube, this eliminated many of the problems of filling and emptying the tube, as well as eliminating the need for an extraneous heat source; (2) mercury has a radioisotope with a sufficiently long half-life; a mass that is sufficiently different from that of the average atomic mass of stable mercury, and is readily detectable; (3) a comparison of results with those of other investigators can be made.

The radioisotope, mercury (203), was purchased in a 10 millicurie quantity by the University of Missouri School of Mines and Metallurgy from the Oak Ridge National Laborabory. The radioactive isotope of mercury was received in the form of mercuric nitrate in a nitric acid solution. The volume of the solution was 0.7 milliliters and the normality was 15.2. The radioactive properties of the isotope properties are given in Table 3.

A. Preparation of the Tagged Mercury

In order for the radioactive mercury isotope to be used in this investigation it had to be recovered from the acid solution in its elemental form. This was accomplished by electrolysis in the following manner (Figure 1): A small amount of stable double distilled mercury was placed in the bottom of a separatory funnel. One end of a platinum wire was placed beneath the surface of the stable mercury. The portion of the wire extending above the surface of the mercury was enclosed in a glass capillary tube so that no portion of the wire was exposed to the acid solution during electrolysis. This arrangement made the stable mercury in the bottom of the funnel the cathode, and all radioactive mercury separated from solution would be deposited on the stable mercury. A small sheet of platinum served as the anode in the

TABLE 3

The Radioactive Properties of Mercury-203

Half-life	Energies of Radiations (in Mev)
47.2 ± .7 days	β^- 0.208
	γ 0.286
	e^- 0.20, 0.27

APPARATUS

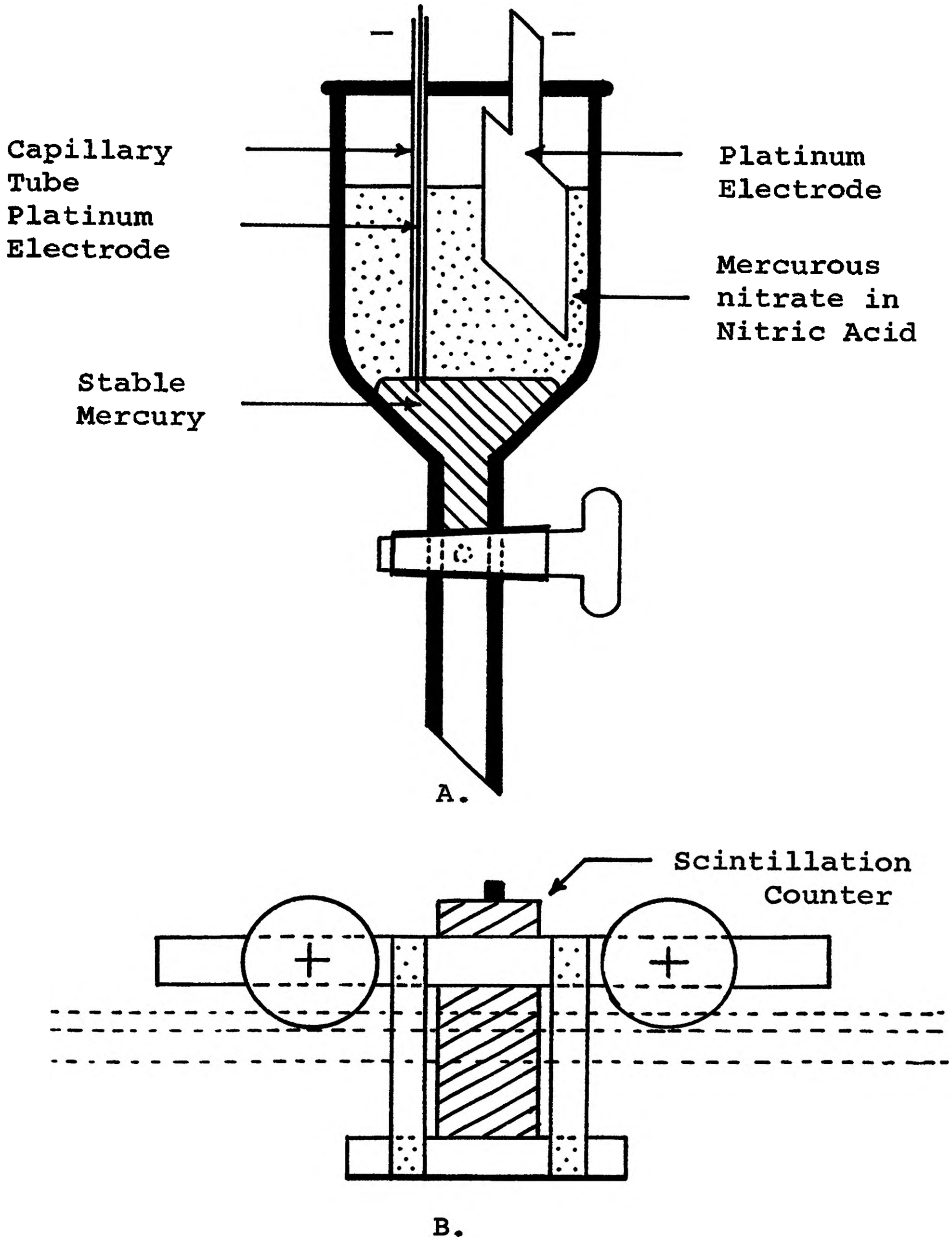

Figure 1

A. Electrolytic Cell
B. Cart to hold scintillation counter

electrolysis. The radioactive solution was mixed with 100 milliliters of 2 normal nitric acid and placed in the 250 ml, wide-mouth separatory funnel. A current of 0.2 ampere was passed through the solution for 5 hours at room temperature. No attempt was made to completely recover the radioactive isotope from solution, the primary purpose of the electrolysis being only to recover enough of the radioisotope to be used in this investigation. The tagged mercury was removed from the funnel by opening the stopcock. This tagged mercury was then cleansed by successive washing and decanting in distilled water.

B. Purification

The tagged mercury was purified by double distillation in a pyrex distillation tube. The condensate was collected under distilled water in a small beaker cooled by an ice bath, and thoroughly mixed before use in this investigation.

C. Description of Separation Cell

The separation cell consisted of a capillary tube 1 millimeter inside diameter, 1.65 millimeters outside diameter and 24 centimeters in length. These values were the average values obtained by microscopic measurement. Two centimeters of the tube at each end was bent at an angle of 45° giving the straight portion of the tube a length of 20 centimeters (Figure 2). A capillary tube was used in this investigation to avoid as much as possible any convection due to thermal gradients through the system. In addition, heating as a result of the current passing through the tube dictated that the capillary have a thin wall for rapid heat dissipation.

D. Filling of the Separation Cell

The capillary was heated slightly before filling to rid it of possible moisture. After allowing the capillary to cool a short length of plastic tubing was attached to one end of the capillary. A syringe was attached to

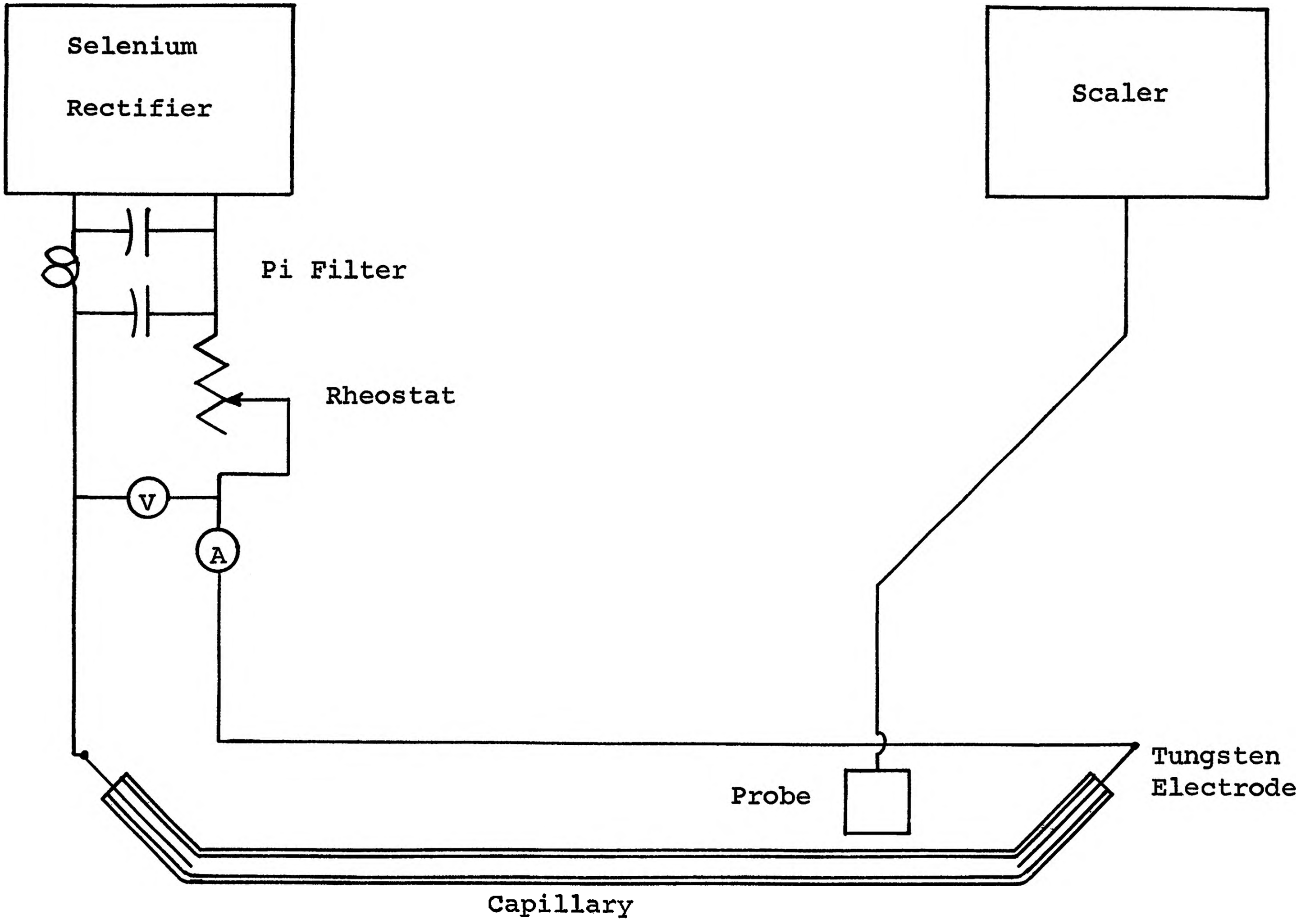

Figure 2. Schematic of Apparatus

the other end of the plastic tubing and a quantity of the tagged mercury sufficient to fill the 20 centimeter portion of the capillary was withdrawn from the mixture of stable and radioactive mercury. This amounted to 0.157 milliliters or 2.127 grams.

E. Counting

All counting in this investigation was done with an automatic decade scaler type N530F manufactured in England by Ekco Electronics Ltd. For detection of the activity of the radioisotope, a NaI (Tl) crystal scintillation counter was placed in an upright position on top of a moveable platform which moved on stationary rails. Grooves slightly larger than the platform wheels were machined into the rails for constant positioning of the counter probe with respect to the capillary tube. The counter probe was shielded with 0.5 centimeter thick lead plate. With the capillary tube in place on the counting stand, the counter probe was 7 centimeters from the capillary tube. A hole in the lead shielding of the counter probe allowed 2 centimeters of the capillary tube to be counted at each position. Each 1 centimeter of the length of the capillary tube was counted for 100 seconds every hour for 12 hours for the first 2 days, every other hour for 12 hours the second 2 days, and at 9:00 A.M., 3:00 P.M., and 9:00 P.M. the duration of the investigation.

F. Separation

The capillary tube was placed in holders in a plexi-glass tank. Tungsten electrodes were inserted at each end of the capillary, which was left unsealed to allow for thermal expansion of the mercury and for the escape of any dissolved gases that might be evolved by the heat generated in the capillary. The tank was then filled with oil and the bath kept at a constant temperature of $30^{\circ} \pm 4^{\circ}$ C. A direct electrical current of 4 amperes, obtained from a single phase selenium rectifier and filtered by a Pi-section filter, was constantly passed through the capillary of mercury. The calculated current density was 509.3A/cm^2 at 6 volts D. C.

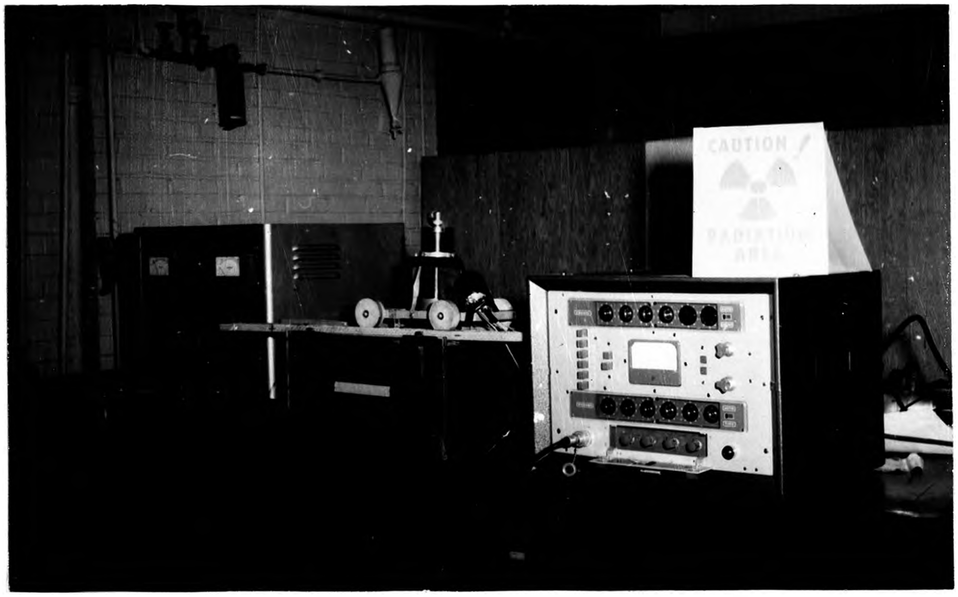

Figure 3. Apparatus used in separation.

IV. DISCUSSION OF RESULTS

In the graphs of the data obtained in this investigation the count rate of the radioactive isotope taken at 24 hour intervals were used in order to maintain a fixed time interval between graphs. All counts were corrected for radioactive decay before recording.

In presenting the data from this investigation, a correction was made for geometric effects. This correction involved the use of a ratio to obtain a dimensionless value. The count rate at a particular position, after a time x, were corrected for radioactive decay to give C_x, the original count for this same position at zero time, C_o, was subtracted from C_x and this value divided by the original count, C_o, to obtain a dimensionless ratio. This is presented in the graphs as the counting ratio, which is plotted versus the counter position.

It is apparent from the data obtained in this investigation that the method of using a radioactive isotope as a tracer to detect and follow the separation of isotopes in mercury can be used with good results. However, the data obtained by this method are somewhat complicated by the presence of the many isotopes of stable mercury. The data obtained do not have a smooth relationship as one would normally expect. This will be discussed in more detail below.

The results of this work are similar to those of other investigators in that separation occurred in the same direction. As in previous investigations, the heavier radioactive isotope, Hg 203, was enriched at the negative end of the capillary tube and depleted at the positive end of the capillary. This can be explained in terms of displacement of the Hg 203 by the lighter isotopes migrating toward the positive end of the capillary tube, as proposed by Jacobs (15) in which he postulated that electron interaction causes the lighter isotopes to migrate toward the positive terminal. This migration is due to a negative free energy gradient in the direction of the positive terminal, and, since the diffusion rate is proportional to the vibrational frequency of the atoms, which is inversely proportional to the mass of the atom, the lighter isotopes will diffuse more rapidly than the heavier isotopes in the direction of the positive terminal. Therefore, due to the incompressibility of the liquid, the heavier isotopes will be forced in the opposite direction toward the negative terminal.

Upon inspection of the graphs of the data one notices that at position one, the negative end of the capillary, there is a decrease in the counting ratio (Figures 4, 5, 6, 7, 9, 10, 12, 13, and 14). This can be explained in terms of the heaviest isotope of mercury present, Hg 204. As stated previously, the heavier isotopes are enriched at the negative end of the capillary. Since Hg 204 is heavier than Hg 203, this isotope would be enriched the greatest amount at the negative end of the capillary and the maximum enrichment of Hg 203 would occur next along the length of the capillary. In stable mercury the relative amount of Hg 204 is 6.8 per cent. Therefore, in a capillary tube 20 centimeters in length, one would expect the maximum enrichment of Hg 204 to occur in the first centimeter at the negative end with the maximum enrichment of Hg 203 very close to this position. This is illustrated in the figures previously cited.

In all of the graphs of the data, from 24 hours to 336 hours, there is a secondary maximum at approximately position eleven, or the middle of the capillary, which appears to reach equilibrium condition in 120 hours. This characteristic has never been reported in previous investigations of the isotopic separation in mercury. If a model is assumed in which the isotopes of mass 200 and below are treated as one part and the isotopes of mass greater than 200 are treated as one part, then each part will comprise approximately fifty per cent of the total due to the relative amounts present in stable mercury. The relative amount of the radioactive tracer which was added is negligible. If complete separation between the two parts is assumed, the isotopes of mass greater than 200 will be concentrated in the negative half of the capillary while those of mass 200 and below will be concentrated in the positive half of the capillary. Assume that before current was passed through the system the mixture of isotopes was homogeneous throughout the capillary tube. After the current began passing through the capillary, the lighter isotopes migrated in the direction of the positive terminal or in the direction of electron flow. Since the mass difference between the lighter isotopes and Hg 203 is larger than the mass difference between the heavier isotopes and Hg 203, the lighter isotopes will migrate more rapidly with respect to the Hg 203 than will the heavier isotopes. Therefore, one would expect a slight pile-up of Hg 203 near the center of the capillary that has been displaced by the lighter isotopes but has not been displaced by the heavier isotopes. This is the pile-up or secondary maximum illustrated in the plots of counting ratio versus counter position.

In previous investigations of the Haeffner Effect in mercury in which reservoirs were attached to the capillary tube to facilitate filling, no attempt has been made to determine the equilibrium conditions. In the previous investigations experiments were run for periods up to 120 days without knowing whether equilibrium was reached. In this investigation, no reservoirs were attached to the capillary and frequent measurements were made in an attempt to attain equilibrium conditions. Close inspection of the graphs of the data obtained in this investigation reveal that after 264 hours (Figure 14), further separation of the isotopes is negligible. This indicates that equilibrium conditions have been attained in the system in 264 hours or in eleven days.

In future investigations of the Haeffner Effect in mercury, the temperature dependence of the separation should be determined. This could be done by making several studies at different temperatures for the same length of time. It would also be interesting to determine the effect of voltage upon the separation. If the radioactive tracer method is used in further studies, it would be desirable to standardize the counting system before each count rate is taken.

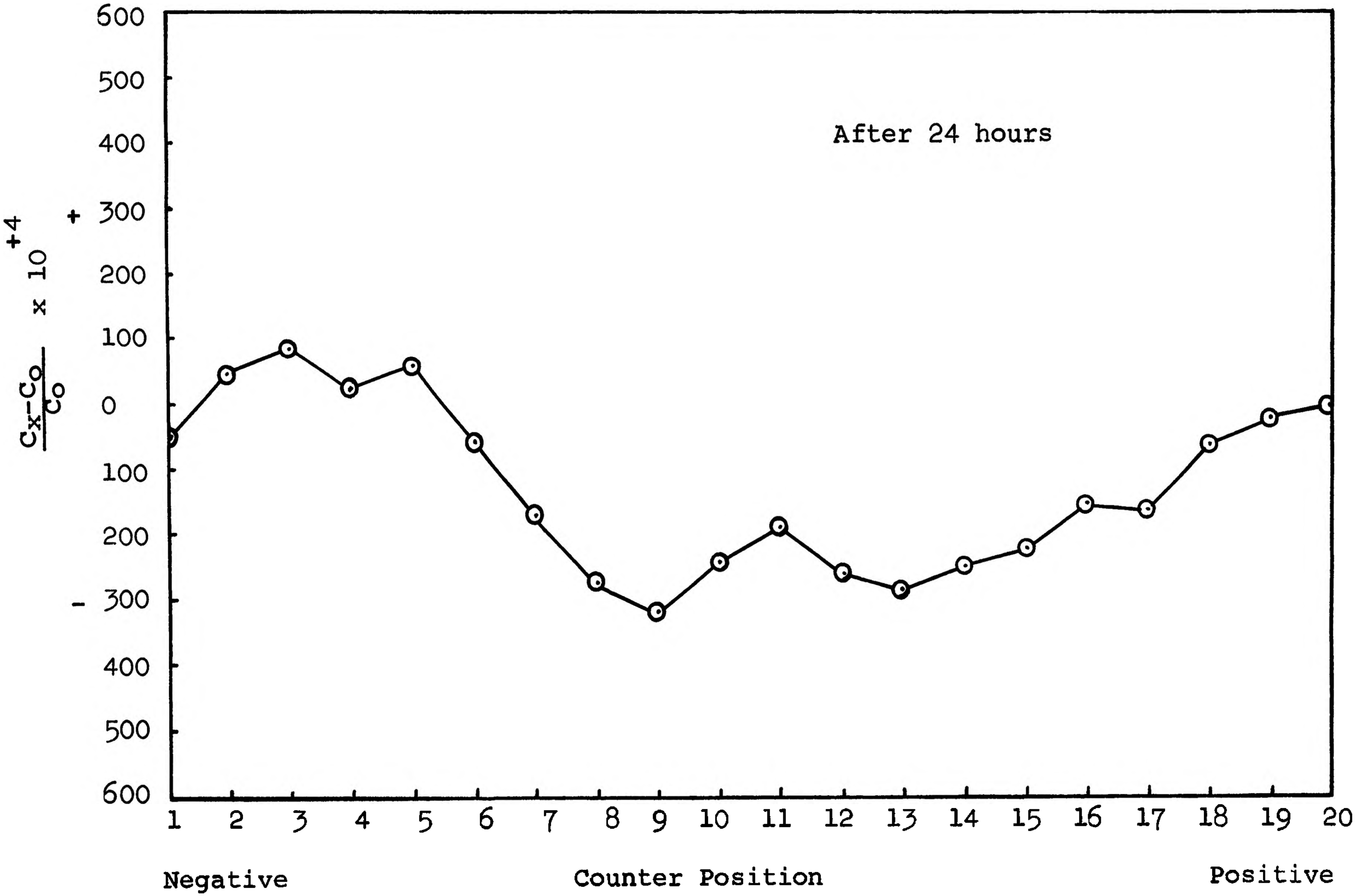

Figure 4. Counting Ratio vs. Counter Position

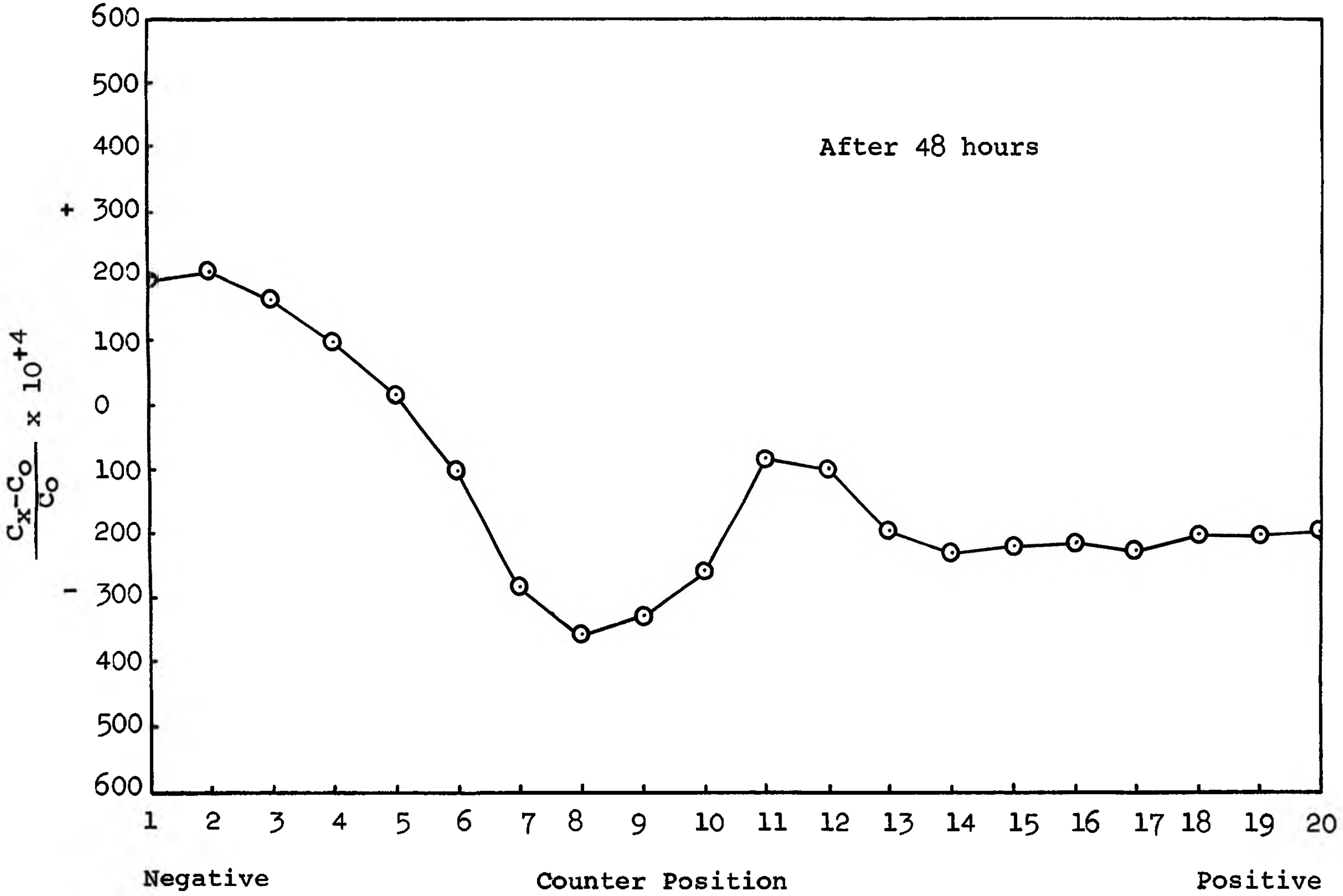

Figure 5. Counting Ratio vs. Counter Position

Figure 6. Counting Ratio vs. Counter Position

Figure 7. Counting Ratio vs. Counter Position

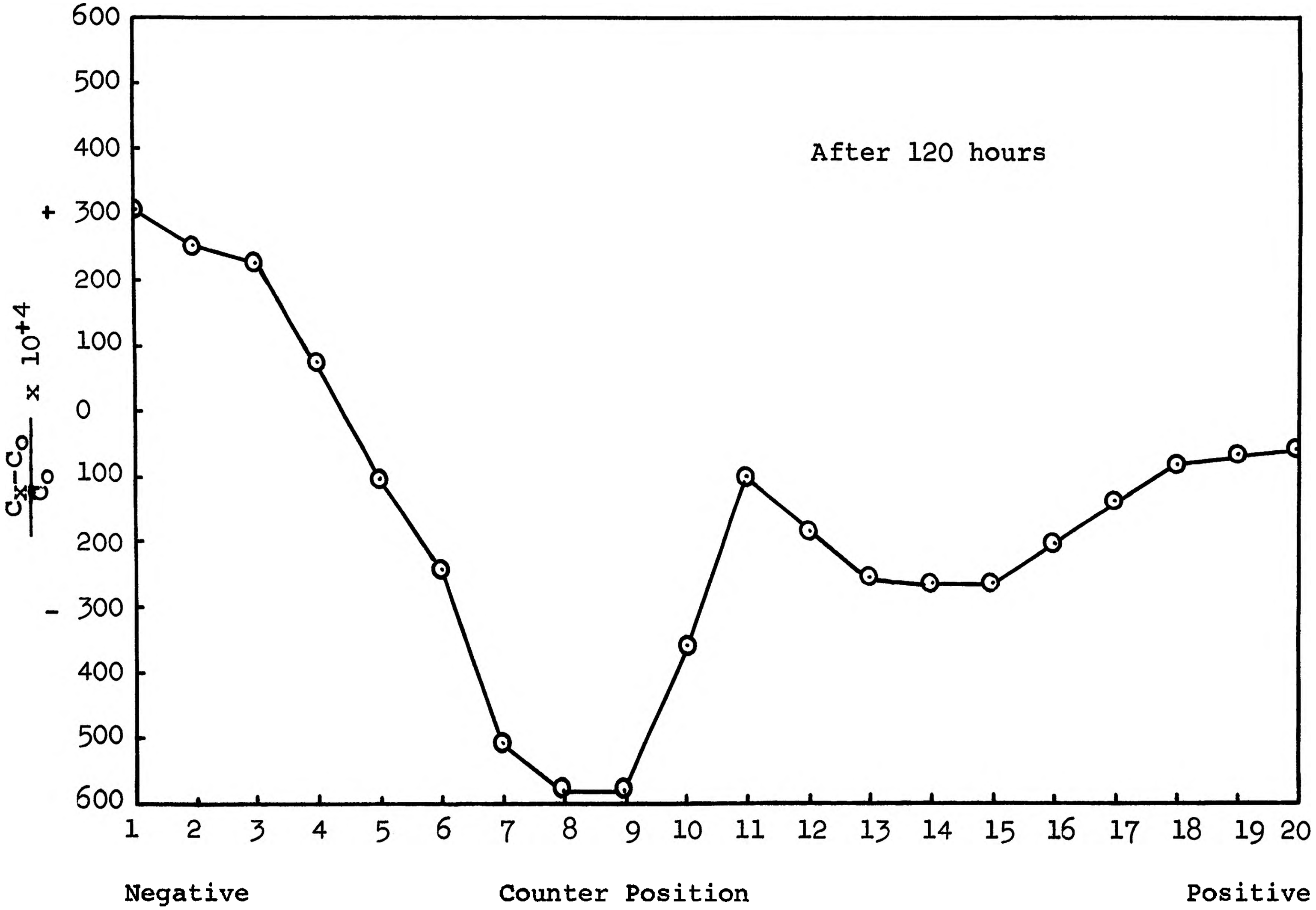

Figure 8. Counting Ratio vs. Counter Position

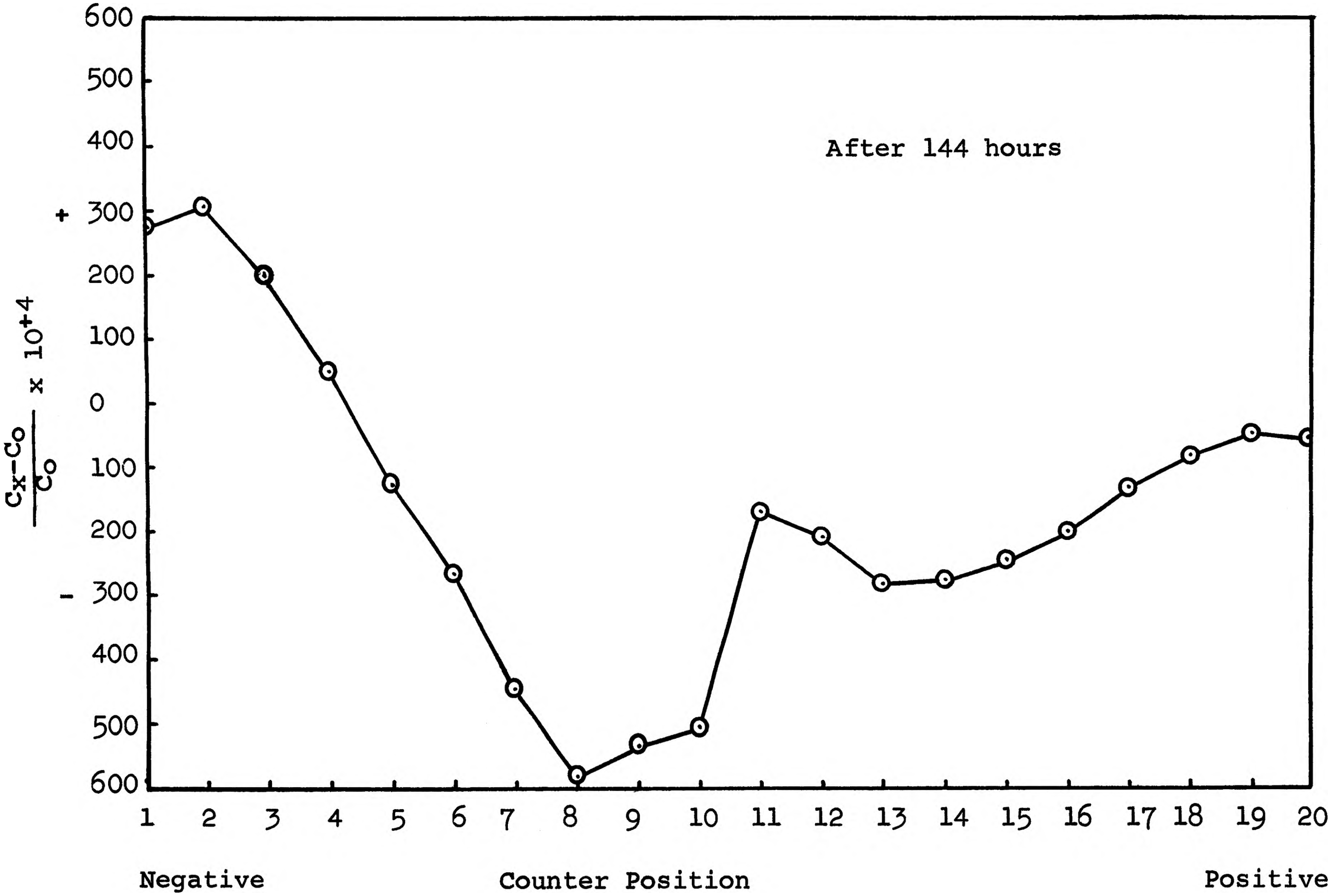

Figure 9. Counting Ratio vs. Counter Position

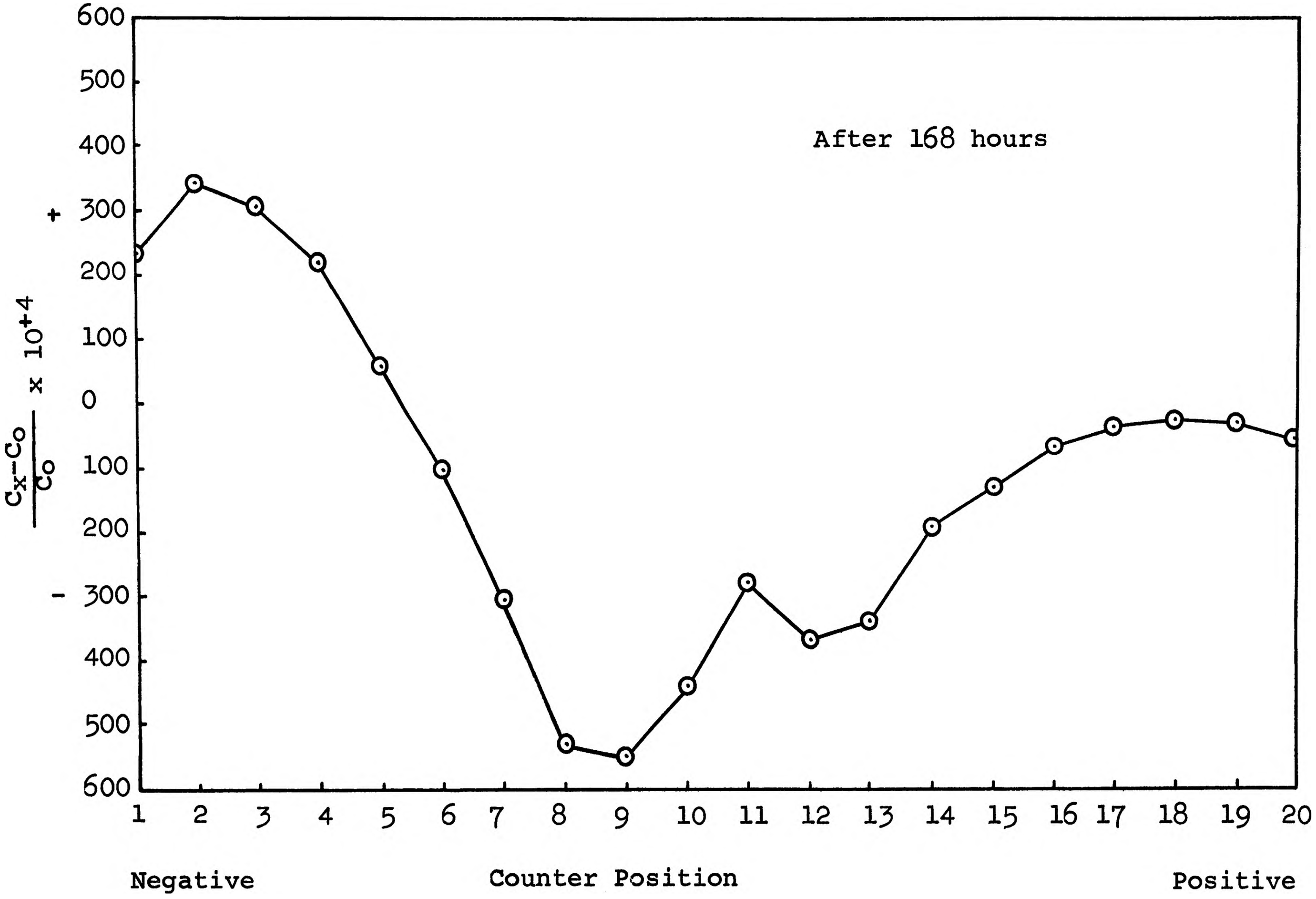

Figure 10. Counting Ratio vs. Counter Position

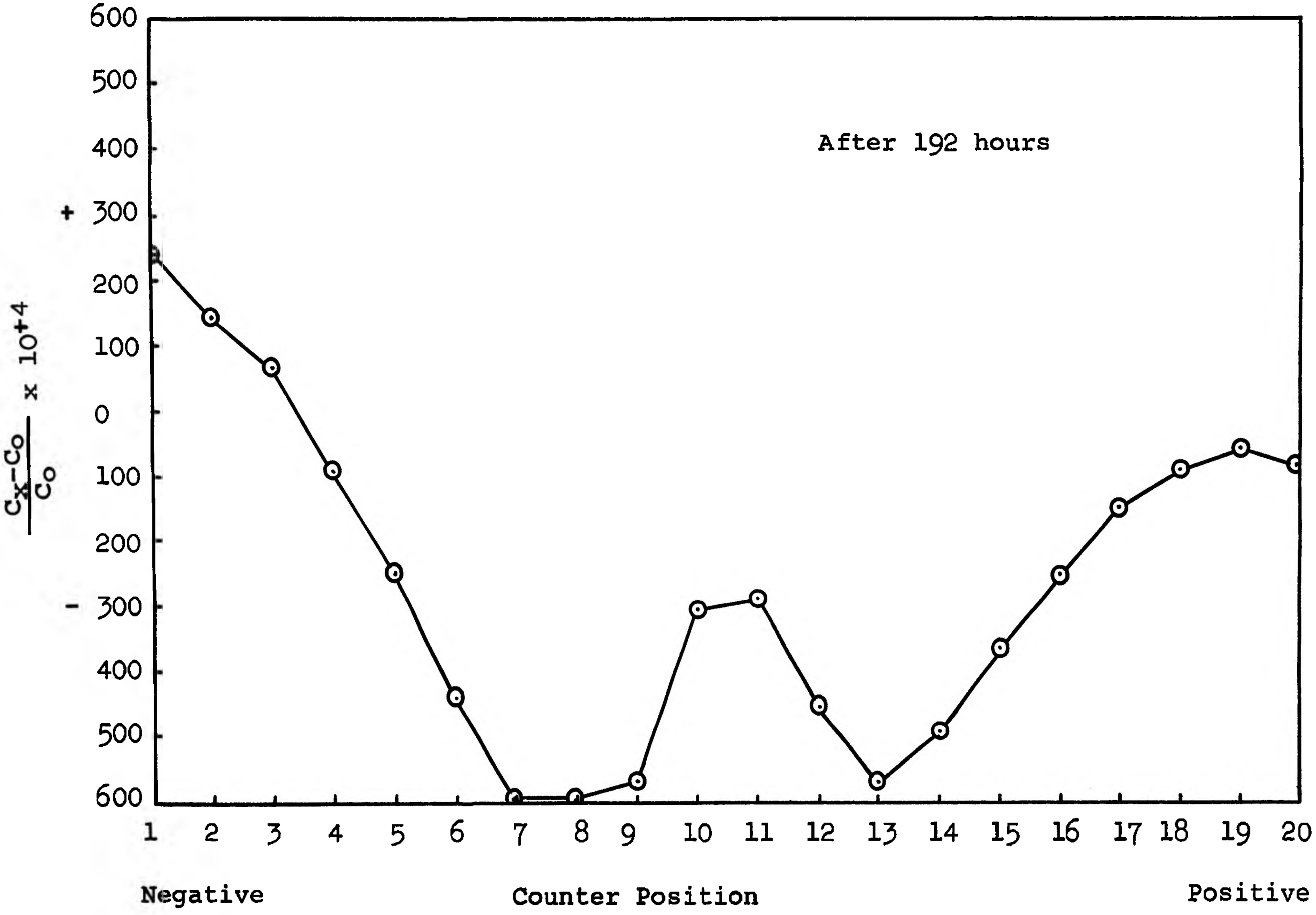

Figure 11. Counting Ratio vs. Counter Position

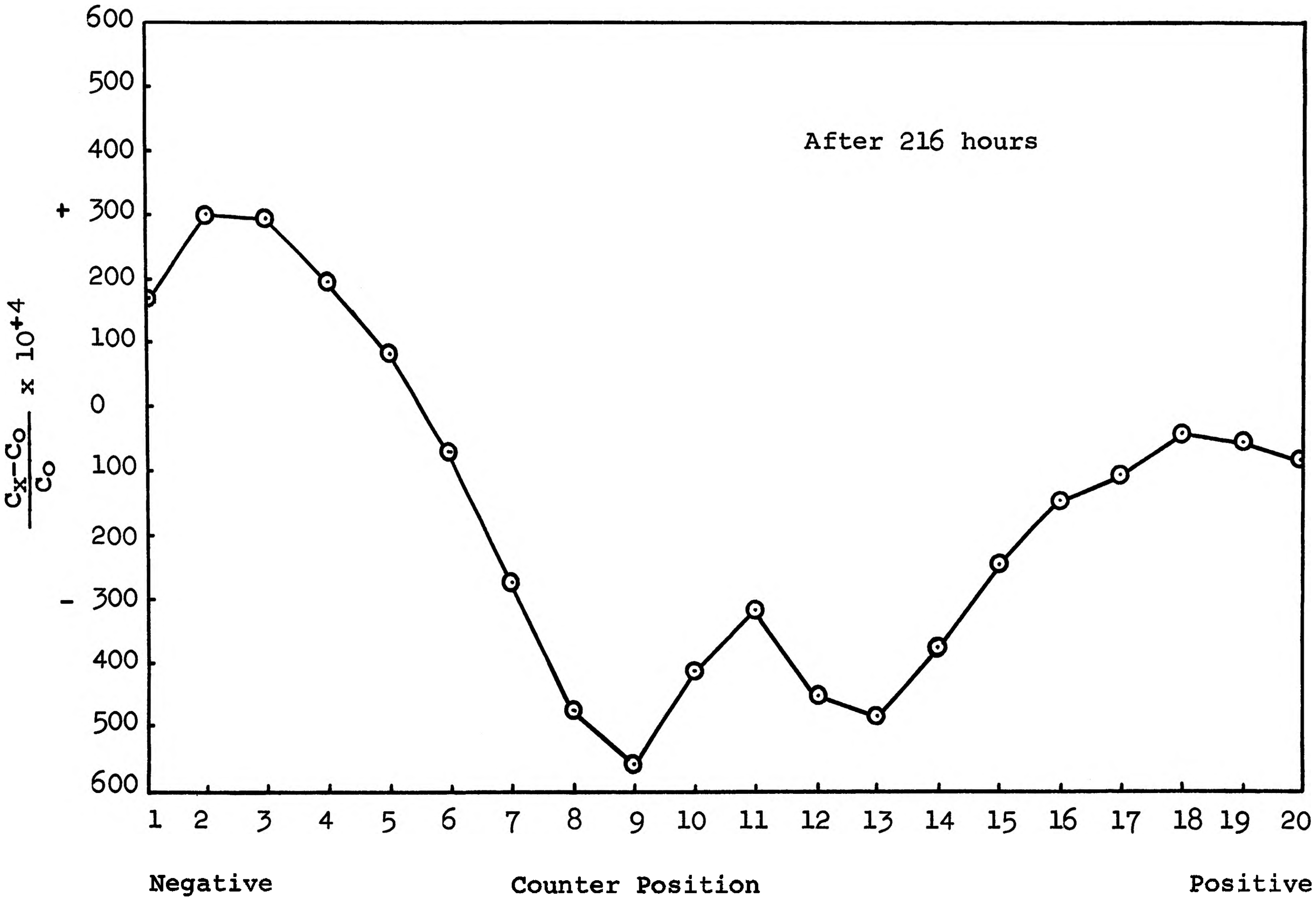

Figure 12. Counting Ratio vs. Counter Position

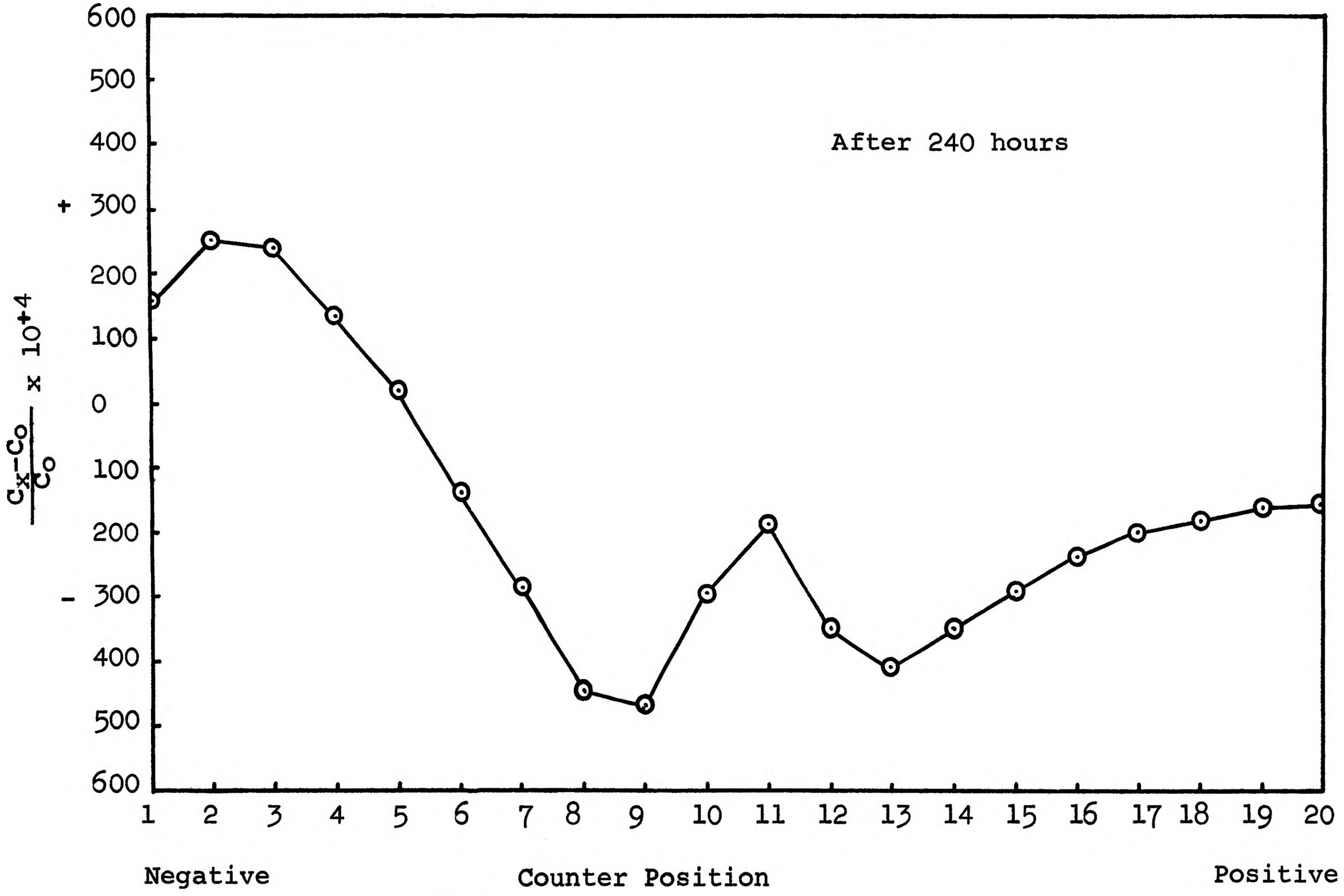

Figure 13. Counting Ratio vs. Counter Position

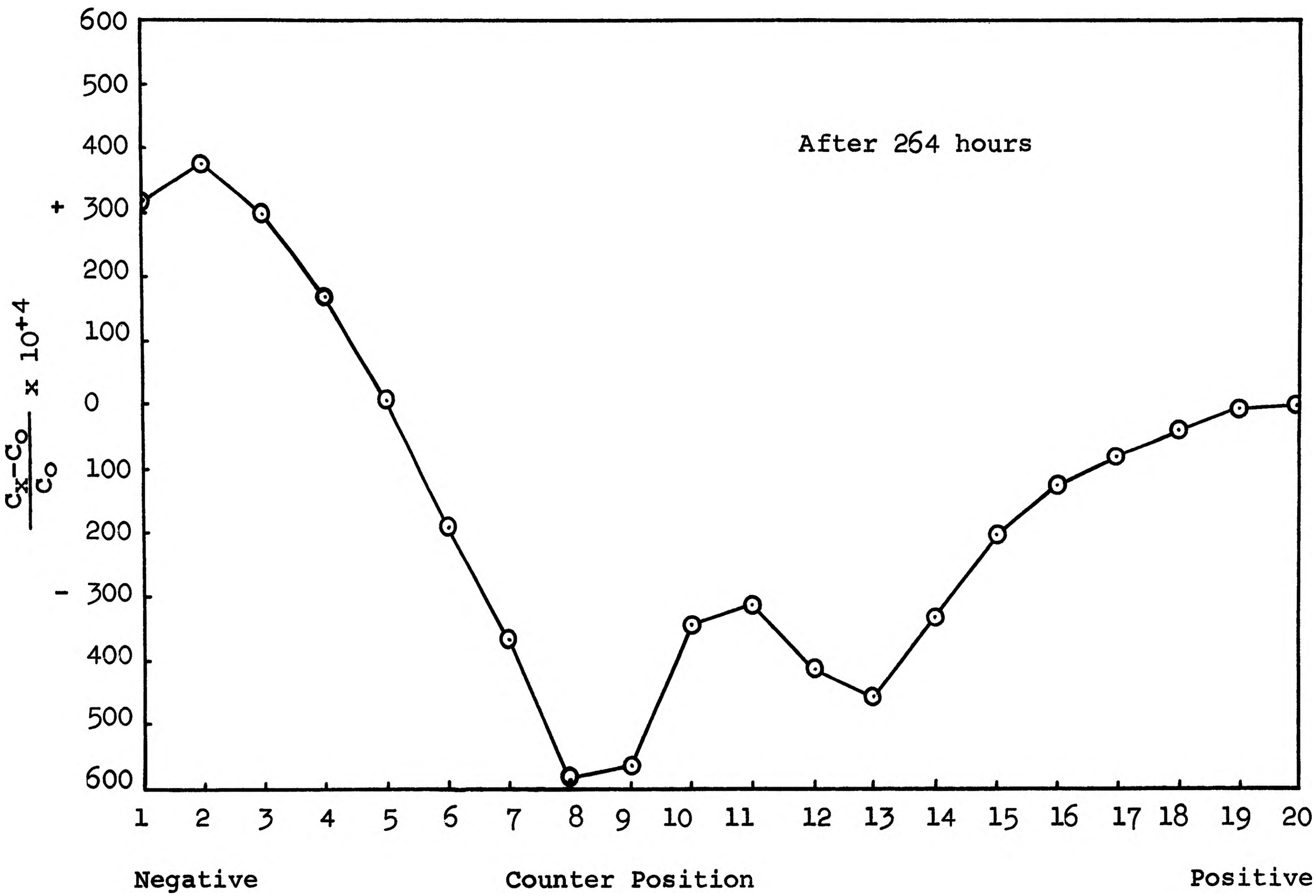

Figure 14. Counting Ratio vs. Counter Position

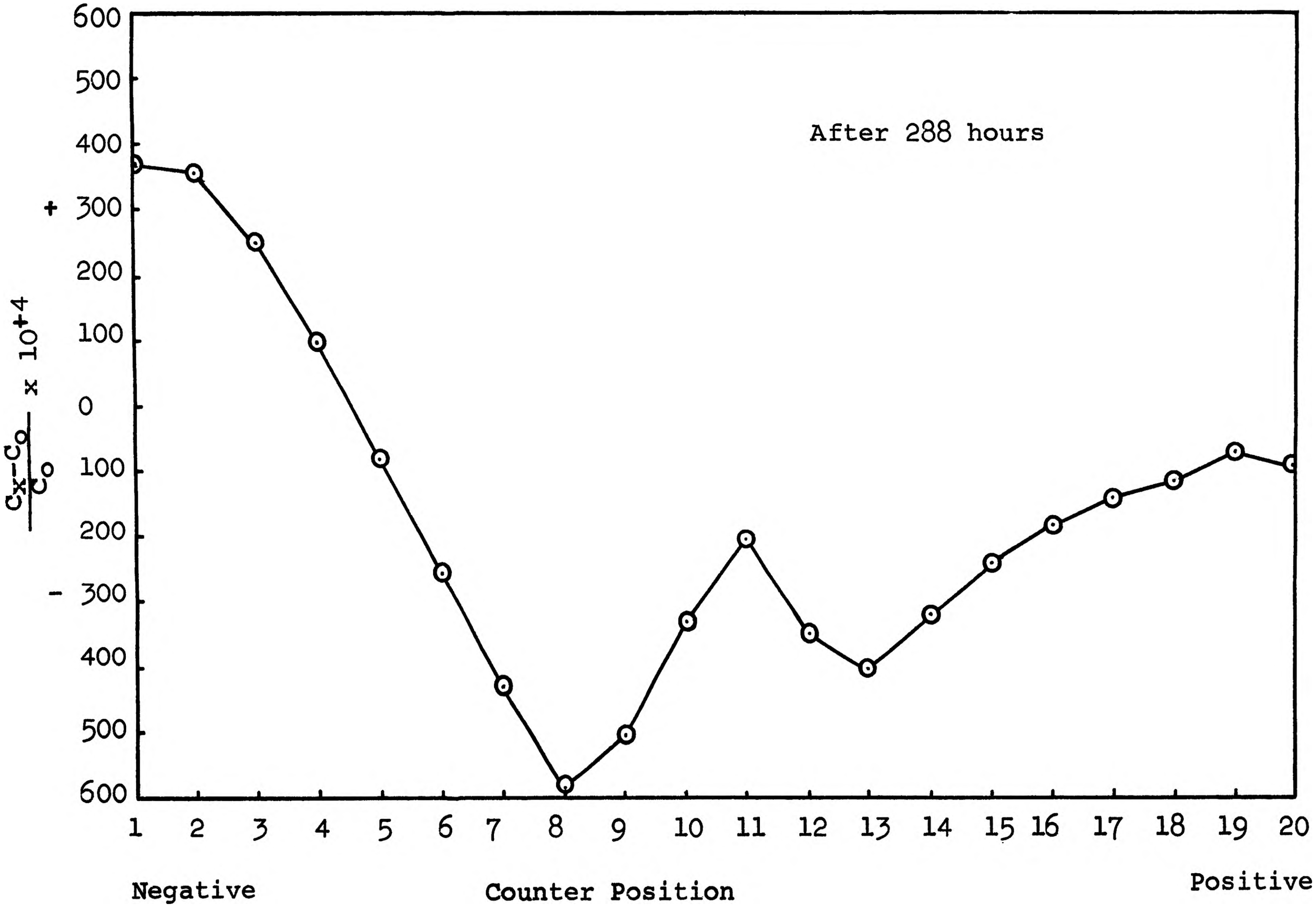

Figure 15. Counting Ratio vs. Counter Position

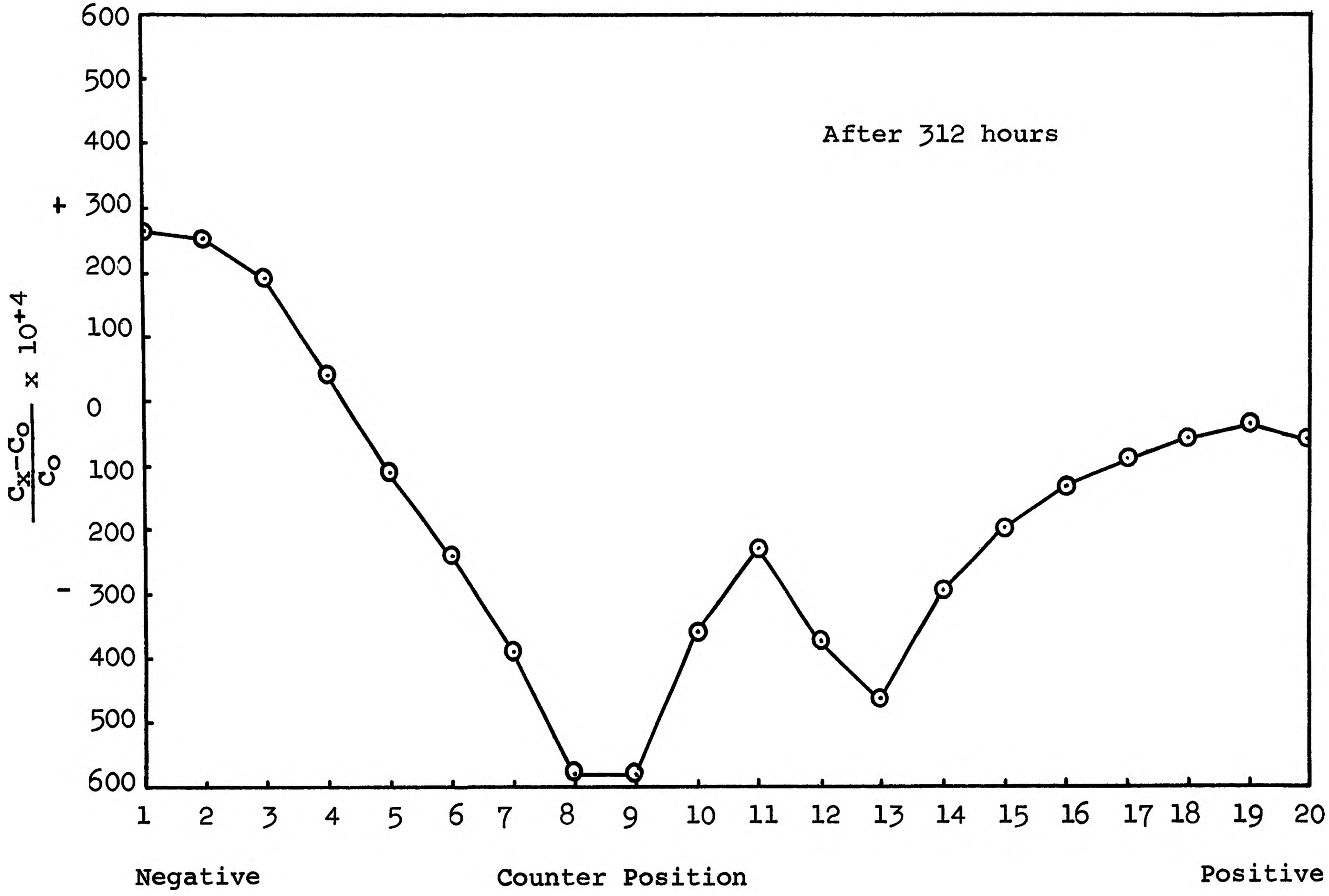

Figure 16. Counting Ratio vs. Counter Position

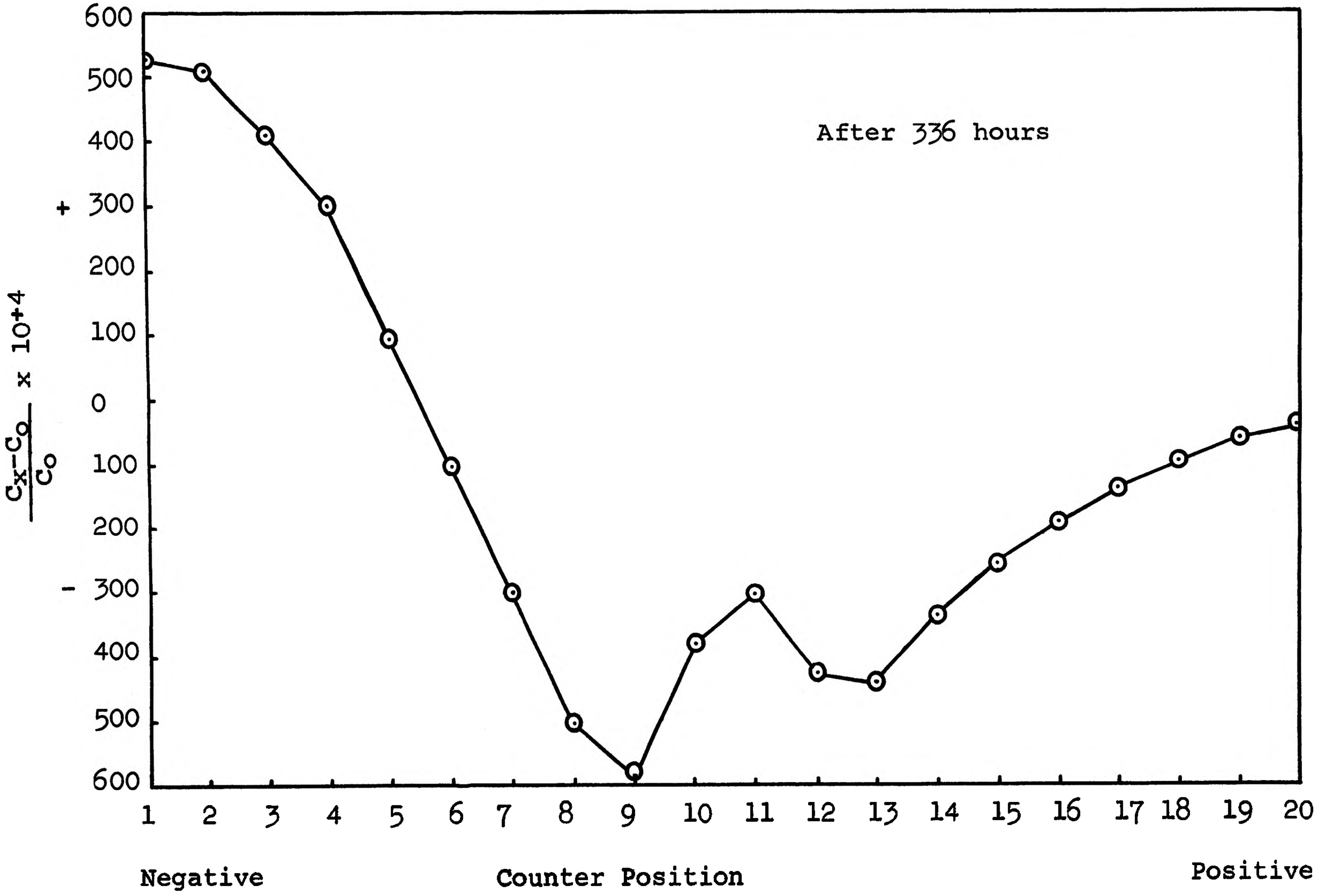

Figure 17. Counting Ratio vs. Counter Position

V. CONCLUSION

The separation of isotopes of liquid mercury occurs in the direction previously reported by other investigators of the Haeffner Effect. The heavier isotopes are enriched at the negative end of the capillary or in the direction opposite to electron flow.

It has been proven that the method of using a radioactive tracer to detect and follow the separation of isotopes in liquid mercury is a valid method that obtains excellent results. However, the use of this method in liquid mercury is somewhat complicated by the several isotopes of stable mercury.

A secondary maximum in the graphs of Counting Ratio versus Counter Position was observed. This may be explained in terms of the diffusion rate of the several different isotopes of mercury.

From the results obtained in this investigation, the time required to attain equilibrium conditions in a capillary tube 20 centimeters long and one centimeter inner diameter filled with mercury is approximately eleven days. This is in a capillary tube to which no reservoirs have been attached.

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