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NEUTRON DEFICIENT RADIOACTIVE ISOTOPES OF TANTALUM AND TUNGSTEN

Geoffrey Wilkinson and Harry G. Hicks

August 18, 1949

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Geoffrey Wilkinson and Harry G. Hicks
Radiation Laboratory and Department of Chemistry
University of California, Berkeley, California

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ABSTRACT

A study has been made of neutron deficient radioactive isotopes of tantalum and tungsten. Bombardments were made, using the 60-inch cyclotron, of lutecium with 20, 30 and 38 Mev α-particles, and of hafnium with 10 Mev protons; tantalum was bombarded with deuterons and protons of various energies using the 184-inch cyclotron. Four new radioactive isotopes of tantalum and three of tungsten have been characterized; evidence has been obtained for the existence of an unreported stable isotope of tungsten, W^{178}.

NEUTRON DEFICIENT RADIOACTIVE ISOTOPES OF TANTALUM AND TUNGSTEN

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Radiation Laboratory and Department of Chemistry
University of California, Berkeley, California

I. Experimental

The techniques of bombardments and of measurement of radiation characteristics by absorption methods, together with assumptions involved in the interpretation of data, have been described previously[1,2].

(1) G. Wilkinson, Phys. Rev. 75, 1019 (1949).
(2) G. Wilkinson, H. G. Hicks, Phys. Rev. 75, 1370 (1949).

After bombardment on the 60-inch Crocker Laboratory cyclotron, the lutecium oxide was dissolved in nitric acid, while the hafnium oxide was dissolved in a mixture of strong nitric and hydrofluoric acids. In both cases, tantalum carrier, prepared by dissolving a known weight of the metal in nitric and hydrofluoric acids was added. In the lutecium bombardments, carrier for hafnium activities was added; in hafnium bombardments, lanthanum carrier was added for rare earth activities. Hold back carriers were also added for contaminating activities likely to be formed from target materials, e.g., copper, platinum, sodium silicate. After bombardment on the 184-inch cyclotron, the tantalum foil was dissolved in the hydrofluoric acid with the addition of the minimum of strong nitric acid necessary to obtain solution; tungsten and other carrier elements were then added.

The chemical separations were made using fluoride solutions, since not only is hydrofluoric acid necessary in the solution of target materials, but radiochemical exchange of hafnium, tantalum and tungsten occurs most effectively in such solutions. The fluoride ion was removed when necessary by complexing with excess boric acid. Further, under the given conditions, the chemical separations themselves are specific for the elements studied. No evidence of contaminating activities has been observed

in any of the separated tantalum and tungsten fractions. The following separations were employed. Lutecium precipitations and lanthanum scavenging precipitations were made from solutions 3N in nitric acid by addition of hydrofluoric acid. Hafnium was precipitated as barium hafnium fluoride by addition of excess barium nitrate solution to solutions 3N in both hydrofluoric and nitric acids; the precipitate was dissolved in nitric acid saturated with boric acid and the hydroxide precipitated. Tungsten was separated using the following procedure. To the hot hydrofluoric acid solution of the tantalum target, hydrazine sulphate was first added to reduce any nitric acid present. Solid ammonium thiocyanate was then added, together with sufficient strong hydrochloric acid to give a 6N solution. On adding excess boric acid to complex the fluoride, and shaking with mercury, the green thiocyanate complex of IV valent tungsten was formed, which was then extracted into ethyl acetate. The washed solvent layer was evaporated, holdback carriers for various elements were added and tungstic oxide precipitated by boiling with strong nitric acid. The oxide was dissolved in ammonium hydroxide, thiocyanate added to the solution and the reduction-extraction process repeated after acidification. Tantalum was recovered after removal of other elements by addition of excess boric acid followed by sodium hydroxide. The hydroxide was re-dissolved in hydrofluoric acid, and scavenging separations of the rare earths, hafnium and tungsten were made as above from the fluoride solution. The tantalum was finally precipitated as potassium tantalum fluoride by saturation of the solution with potassium fluoride. The tantalum precipitate was fumed with strong sulphuric acid to remove the fluoride, and the hydroxide then precipitated.

In all cases the oxides were prepared for counting and for estimation of chemical yields by ignition of the hydroxides.

II. Tantalum Isotopes

Four new tantalum activities of half lives 8.0 hours, 2.5 days, 15.4 days and ~120 days have been produced by bombardment of lutecium with alpha particles of various energies; a brief report on the first three isotopes has been given

previously.[3] From the yields, allocations have been made respectively to masses 176, 177, 178 and 179. The isotopes have been identified also in the tantalum fraction from Hf + p bombardments. The radiation characteristics of the isotopes are summarized in Table I.

(3) G. Wilkinson, H. G. Hicks, Phys. Rev. 74, 1733 (1948).

Table I

Production and Characteristics of Tantalum and Tungsten Isotopes.

Isotope	Type of Radiation	Half-Life	Energy of Radiation in Mev Particles	Energy of Radiation in Mev γ-rays	Produced by
Ta^{176}	K, e^-, γ, β^-?	8.0±0.1 hrs.	0.12, 0.18(e^-) ~1(β^-?)	L, K x-rays, ~2	Lu-α-3n
Ta^{177}	K, e^-, γ	2.50±0.05 days	0.11(e^-)	L, K x-rays ~1.4(weak)	Lu-α-2n, 3n Hf-p-n Ta-d-p5n W^{177} K decay
Ta^{178}	β^-, γ?	15.4±0.2 days	1.5		Lu-α-n, 2n Hf-p-n
Ta^{179}	K, e^-, γ	~120 days	~0.12(e^-)	L, K x-rays	Lu-α-n Hf-p-n Ta-d-p3n Ta-p-p2n
Ta^{180}	K, γ, β^-(~10%)	8.00±0.05 hrs.	0.7(β^-)	K x-rays 1.3	Ta-n-2n Ta-d-p2n Ta-p-pn
Ta^{180}	K or I.T., e^- γ, β^-(< 5%)	16±2 min.	0.12(e^-) 0.6 (β^-)		Ta-n-2n
W^{177}	K, e^-, γ	130±3 min.	0.13(e^-) ~0.4(e^-)	L, K x-rays ~0.45 1.2	Ta-d-6n Ta-p-5n
W^{179}	K	30±1 min.	No e^-	L, K x-rays	Ta-d-4n Ta-p-3n
W^{179}	K, e^-, γ	21 days	0.12(e^-) ~0.5(e^-?)	L, K x-rays	Ta-d4n Ta-p3n

8.0±0.1 hour Ta^{176}

The chemically separated tantalum from 38 Mev α particle bombardment of lutecium shows the 8.0 hour activity in high yield.

The decays (Fig. I) of the gross and electromagnetic radiations were followed separately through about eight half-lives; after correction for long-lived backgrounds, a half-life of 8.0 ±0.1 hours was obtained from several measurements.

In Fig. II is shown the aluminum absorption of the radiations of the 8.0 hour activity; the soft electromagnetic radiation was obtained by aluminum absorption after removal of electrons in beryllium foils. The lead absorption of hard electromagnetic radiation was also measured. No positrons were observed on a crude beta ray spectrometer. From the measurements, the radiations consist of electrons of total ranges ~19 mg/cm^2 (120 Kev), ~40 mg/cm^2(180 Kev), and ~400 mg/cm^2(~1 Mev) together with electromagnetic radiation of half-thicknesses, 16 mg/cm^2 aluminum (8.5 Kev), ~115 mg/cm^2 lead (58 Kev) and 15.5 g/cm^2 lead (2 Mev). From aluminum absorption measurements on an infinitely thin sample, from the lead absorption, and making the customary corrections and assumptions regarding counting efficiencies, the following ratios were obtained:

	120 Kev e^-	180 Kev e^-	~1 Mev e^-	L x-rays	K x-rays	2 Mev γ ray
=	~1	~0.04	~0.02	~0.5	1	0.6

The isotope thus probably decays by orbital electron capture followed by converted γ ray transitions from excited or metastable levels of the daughter nucleus.

2.50 ± 0.03 day Ta^{177}

After decay of the 8.0 hour activity, an activity of 2.5 days half-life has been observed in the tantalum fraction from Lu + α bombardments. The resolved gross decay of the 2.5 day activity was followed through eight half-lives, and the decay of electromagnetic radiation through nine half-lives to give a value of 2.50 ± 0.03 days (Fig. I, III) for the half-life.

In Fig. IV is shown the aluminum absorption after subtraction of the contribution (10%) of the 15 day tantalum activity at the time of measurement. Resolution shows electrons of total range 16 mg/cm^2(110 Kev) together with soft electromagnetic radiation of half-thickness 16 mg/cm^2(8.5 Kev). The lead absorption (Fig. IV) of hard electromagnetic radiation shows components of half thicknesses 120 mg/cm^2 lead (58 Kev) and ~13 g/cm^2

lead (1.4 Mev). The following ratios of the various radiations were obtained:

	110 Kev e^-:	L x-rays:	K x-rays:	1.4 Mev γ-ray
=	$\sim$0.3 :	0.3 :	1 :	0.005

It appears fairly safe to assume decay by orbital electron capture with electrons arising from conversions in subsequent transitions.

The 2.5 day activity has been observed in high energy particle bombardments of tantalum and has been shown to be produced by decay of a 130 minute tungsten activity. It has been also observed in the tantalum fraction from Hf + p bombardments.

15.4 ± 0.2 day Ta^{178}

The half-life of this isotope resolved from the tantalum gross decay (Fig. III) from Lu + α bombardment is 15.4 ± 0.2 days through six half-lives. The aluminum absorption (Fig. V) shows the radiation to consist almost entirely of hard electrons, range 610 mg/cm^2 (1.4 Mev). The shape of the absorption curve suggests beta particle radiation and study of the isotope on the crude beta ray spectrometer showed negative electrons with an energy distribution corresponding to that of a beta particle spectrum of maximum energy $\sim$1.5 Mev. The Feather range 770 mg/cm^2 corresponds to 1.65 Mev maximum energy.

Sufficient intensity was not available to allow a lead absorption of gamma radiation, but from the decay of tantalum electromagnetic radiation and aluminum absorption measurements, an upper limit of 0.002 γ or K x-rays per electron is obtained. Assuming 1% counting efficiency for such radiation the γ/β^- ratio is less than 0.2 thus confirming designation of the 15.4 day activity as a negative beta particle emitter. The isotope has been observed also in the tantalum fraction from Hf + p bombardment; the resolved decay and radiation characteristics agree with those from Lu + α bombardments.

~120 day Ta^{179}

After decay of shorter-lived activities, tantalum fractions from both Lu + α and Hf + p bombardments show a long-lived activity. The half-life at present appears to be ~ 120 days. In the bombardment of tantalum with 40 Mev protons, this activity has been observed after decay of the 8.0 hour Ta^{180}. In this case formation is possible directly by p,p2n reaction, or by decay from the tungsten parent isotope.

The aluminum absorption shows electrons, range ~ 20 mg/cm^2 (120 Kev), soft electromagnetic radiation, half-thickness ~ 16 mg/cm^2, and K x-rays or γ radiation.

8.0 hour and 16 minute Ta^{180}

Two activities of half-lives 8.2 hours and 14 minutes have been produced by n,2n and γ,n reactions in tantalum[4,5] and allocated to mass 180. These activities

(4) O. Oldenberg, Phys. Rev. 53, 35 (1938).
(5) W. Bothe, W. Gentner, Naturwissenschaften 25, 191 (1937).

have now been re-examined. Very pure tantalum was bombarded with fast neutrons from Be + d reaction; the short lived activity was studied without chemical separation, but separation was made for the 8 hour activity. The 8.0 hour activity is also produced in high yield by bombardment of tantalum by 40 and 50 Mev protons presumably by the p,pn reaction.

The decays of the gross and electromagnetic radiations of the longer lived activity were followed through ten half-lives and a value of 8.00 ± 0.05 hours obtained for the half-life. In Ta + n bombardments, a small, long-lived background was identified as the 117 day Ta^{181} produced by neutron capture. The aluminum absorption curve of the 8 hour activity (Fig. VI) shows beta particles, range 210 mg/cm^2 aluminum (0.6 Mev), Feather range 238 mg/cm^2 (0.7 Mev) and hard electromagnetic radiation. The lead absorption shows components of half thicknesses 115 mg/cm^2 (58 Kev) and 12.2 g/cm^2 (1.3 Mev); the energy of the former corresponds well with the energy of hafnium K x-radiation. From the measurements, with usual corrections and assumptions counting efficiencies, the following ratios were obtained:

$$0.7 \text{ Mev } \beta^- : \text{ K x-rays} : 1.3 \text{ Mev } \gamma = 0.13 : 1 : 0.02$$

The isotope thus appears to decay predominantly by orbital electron capture. Taking one K x-ray quantum as representing one disintegration by orbital electron capture, the branching by beta particle emission is around 0.1.

The short lived isomer was studied in short neutron bombardments of tantalum and a half life of 16 $\pm$ 2 minutes obtained. This activity is formed in very low yield, the saturation intensity being only about a hundredth that of the 8 hour activity. A rapid measurement of the aluminum absorption of the mixed activities was made and the contribution of the 8 hour activity subtracted. The resolved aluminum absorption curve of the 16 minute activity (Fig. VII) shows very soft electrons, range $\sim$ 20 mg/cm^2 (120 Kev), hard electrons or beta particles, range $\sim$180 mg/cm^2 ($\sim$0.6 Mev) together with K x-ray or γ ray background. The approximate ratios of the radiations are

$$120 \text{ Kev } e^- : 0.6 \text{ Mev } \beta^- : \text{K} + \gamma \text{ rays} = \sim 0.2 : \sim 0.05 : 1$$

Since the very soft electron was not observed in the decay of the 8.0 hour activity, it seems unlikely that these electrons arise from γ-ray transitions in hafnium following orbital electron capture, and the 16 minute activity may decay, partially at least, by isomeric transition. The hard particle present suggests a few percent branching by beta particle emission.

Discussion

The yields of the tantalum isotopes have been estimated for 38, 30 and 20 Mev γ-particle bombardments of lutecium. Since the decay schemes of all but the 15.4 day activity are obscure, the K x-radiation was used as a reference and the yield of the most abundant activity in each bombardment taken as unity. The yields of the various isotopes, and the variations with bombarding energy were similar to those found for the production of thulium[2] and rhenium[6] activities by α-particle

(6) G. Wilkinson, H. G. Hicks, Phys. Rev. (in press).

bombardments of holmium and tantalum respectively. Since lutecium has two stable isotopes, tantalum isotopes of masses 177 and 178 can be formed by both α,3n and α,2n and α,2n and α,n reactions respectively, while isotopes of masses 176 and 179 can be formed only by Lu^{175}-α,3n and Lu^{176}-α-n reactions respectively.

The yields of the 2.5 day, 15.4 day and 120 day activities in 10 Mev protons bombardments of hafnium agree with present allocations. The presence of the 8.0 hour Ta^{176} is masked in this case however by simultaneous formation of the 8.0 hour Ta^{180}.

Since the allocation of the 15.4 day activity is made to mass 178, and particularly since its radiations appear to consist almost entirely of negative beta particles, it seems necessary to postulate the existence of a previously unreported beta stable isotope of tungsten of mass 178. Additional evidence for stability of this isotope has been obtained from bombardments of tantalum with deuterons and protons of various energies from the 184 inch cyclotron, where careful examination of the decays of the chemically separated tantalum fractions has shown no evidence for the 15.4 day activity. The yield of the 15.4 day activity is certainly less than a thousandth that of the 2.5 day Ta^{177} or the 120 day Ta^{179} activities. The 120 day Ta^{179}, 8.0 hour Ta^{180} and 117 day Ta^{181} activities were observed in all cases, but the 2.5 day Ta^{177} was detected only at bombarding energies of 50 Mev deuterons and above. With 50 and 60 Mev deuterons, the yield of the 15.4 day and 2.5 day activities should have been comparable.

III. Tungsten Isotopes

The bombardment of tantalum with deuterons and protons of various energies from the 184 inch cyclotron has led to the recognition and characterization of three new radioactive isotopes of tungsten. The 140 day W^{181} activity produced by d,2n and p,n reactions in tantalum has been described previously.[7]

(7) G. Wilkinson, Nature 160, 864 (1947).

130 minute W^{177}

This isotope was observed in bombardments of tantalum with deuterons of energy 45 to 60 Mev and protons of $\sim$ 40 to 60 Mev from the 184 inch cyclotron. The radiation characteristics were best measured using the chemically separated tungsten fraction from a 50 Mev proton bombardment of tantalum. The decays of electron and electromagnetic radiations followed separately through seven half-lives gave a value of 130 $\pm$ 3 minutes for the half-life. The radiations (Fig. VIII) consist of electrons, total ranges in aluminum of 21 mg/cm^2(0.13 Mev) and $\sim$100 mg/cm^2 (0.4 Mev), L and K x-radiation and γ radiation of half thicknesses in lead of 2.75 g/cm^2 (0.45 Mev) and 11.7 g/cm^2 (1.2 Mev). No positrons were observed on a simple beta ray spectrograph. The ratios of the various radiations, corrected for counting efficiencies, etc. are:

0.13 Mev e^-	0.4 Mev e^-	L x-rays	K x-rays	0.4 Mev γ ray	1.2 Mev γ ray
0.1	0.02	0.25	1	0.05	0.2

In order to characterize daughter activities of this isotope, the tungsten fraction containing the 130 minute activity was "milked" for tantalum activities. The tantalum fraction was found to contain only a pure 2.5 day activity whose decay was followed through six half-lives and whose radiations are identical with those of the 2.5 day Ta^{177} described above. The yield of the tantalum activity in successive separations shows it to be the daughter of the 130 minute tungsten activity. No evidence for shorter or longer lived daughter activities was obtained. Allocation of the 130 minute activity to mass 177 is made on the basis of recognition of the 2.5 day Ta^{177} daughter activity and also from considerations of yield in bombardments at various energies which agree with formation by a d,6n or p,5n reaction.

30 $\pm$ 1 minute $W^{178,9}$

As the yield of the 130 minute W^{177} activity decreased with decreasing bombarding energy, the yield of a second new tungsten activity of half-life 30 minutes increased to a maximum for both deuterons and protons of about 40 Mev. The activity has not

been observed in bombardment of tantalum with 20 Mev deuterons, where the d,3n reaction would be observable, and hence must be due to an isotope of mass less than 180. The radiation characteristics were obtained from aluminum beryllium and lead absorption measurements, and consist only of tungsten or tantalum L and K x-radiation (Fig. X). No evidence was obtained for either electron or hard electromagnetic radiation and an upper limit of one percent of the K x-radiation can be placed for each. The ratio of L to K x-radiation was 0.25:1. The decays measured through seven half-lives gave a value of 30 $\pm$ 1 minutes for the half-life (Fig. IX). The isotope may be either an isomer of the 21 day activity allocated to W^{179}, decaying by orbital electron capture, or an excited state of the postulated stable W^{178}. The latter possibility is the least likely, since no conversion electrons or gamma rays were observed.

21.0 $\pm$ 0.2 day W^{179}

In the bombardments of tantalum with lower energy deuterons or protons from the 184 inch cyclotron, the chemically separated tungsten fraction shows, after decay of shorter lived activities, an activity which decays with a 21.0 $\pm$ 0.2 day half-life. Qualitative estimates of the yields are in agreement with allocation of the activity to mass 179, and this allocation has been confirmed by chemical separation of the 120 day tantalum activity allocated to mass 179 after decay of a large sample of the tungsten fraction. The isotope would hence be formed by d,4n and p,3n reactions in tantalum. The radiations consist of very soft electrons, total range $\sim$15 mg/cm^2 (0.1 Mev), electrons range $\sim$120 mg/cm^2 (0.4 Mev) together with L and K x-radiation. Fig. XI. The decay measured through seven half-lives is 21.0 $\pm$ 0.2 days.

Acknowledgements

The authors are indebted to Dr. J. G. Hamilton, Mr. J. Vale and the crews of the 60-inch and 184 inch cyclotrons for their cooperation and assistance in bombardments. We wish to thank Professors G. T. Seaborg, and I. Perlman for their continued interest and advice. This paper is based on work performed under the auspices of the Atomic Energy Commission.

LEGENDS FOR FIGURES

Fig. I Decay of 8.0 hour Ta^{176}(B) and 2.5 day Ta^{177}(A) activities, with 15.4 day background (C) from Lu + α bombardment.

Fig. II Aluminum absorption of 8.0 hour Ta^{176} activity. K x-ray and γ-rays (A), electrons (B,C,D), L x-rays (E).

Fig. III Decay of 2.5 day Ta^{177} (C), 15.4 day Ta^{178}(B) and ~120 day Ta^{179} activities from Lu + α bombardment.

Fig. IV Aluminum and lead absorptions of 2.5 day Ta^{177} activity from Lu + α bombardment. Aluminum absorption: γ-ray background (A), 0.11 Mev electron (B) and L x-rays (C). Lead absorption: 1.4 Mev γ-ray (A), K x-rays (B).

Fig. V Aluminum absorption of 15.4 day Ta^{178} activity from Lu + α bombardment. X and γ-ray background (A), 1.3 Mev beta-particle (B).

Fig. VI Aluminum absorption of 8.0 hour Ta^{180} activity from Ta + n bombardment. X and γ-rays (A), 0.7 Mev beta particle (B).

Fig. VII Aluminum absorption of 16 minute Ta^{180} from short Ta + n bombardment. X and γ-rays (A), 0.6 Mev beta particle (B), 0.2 Mev electron (C).

Fig. VIII Aluminum and lead absorptions of 130 minute W activity from Ta + p bombardment. X and γ-rays (A), L x-rays (B), electrons (C,D).

Fig. IX Decay of 30 minute tungsten activity from Ta + p bombardment.

Fig. X Aluminum (A,B) and beryllium (C) absorptions of 30 minute tungsten activity.

Fig. XI Aluminum absorption of 21.0 day tungsten activity from Ta + p bombardment. K and γ-rays (A), L x-rays (B), electrons (C).

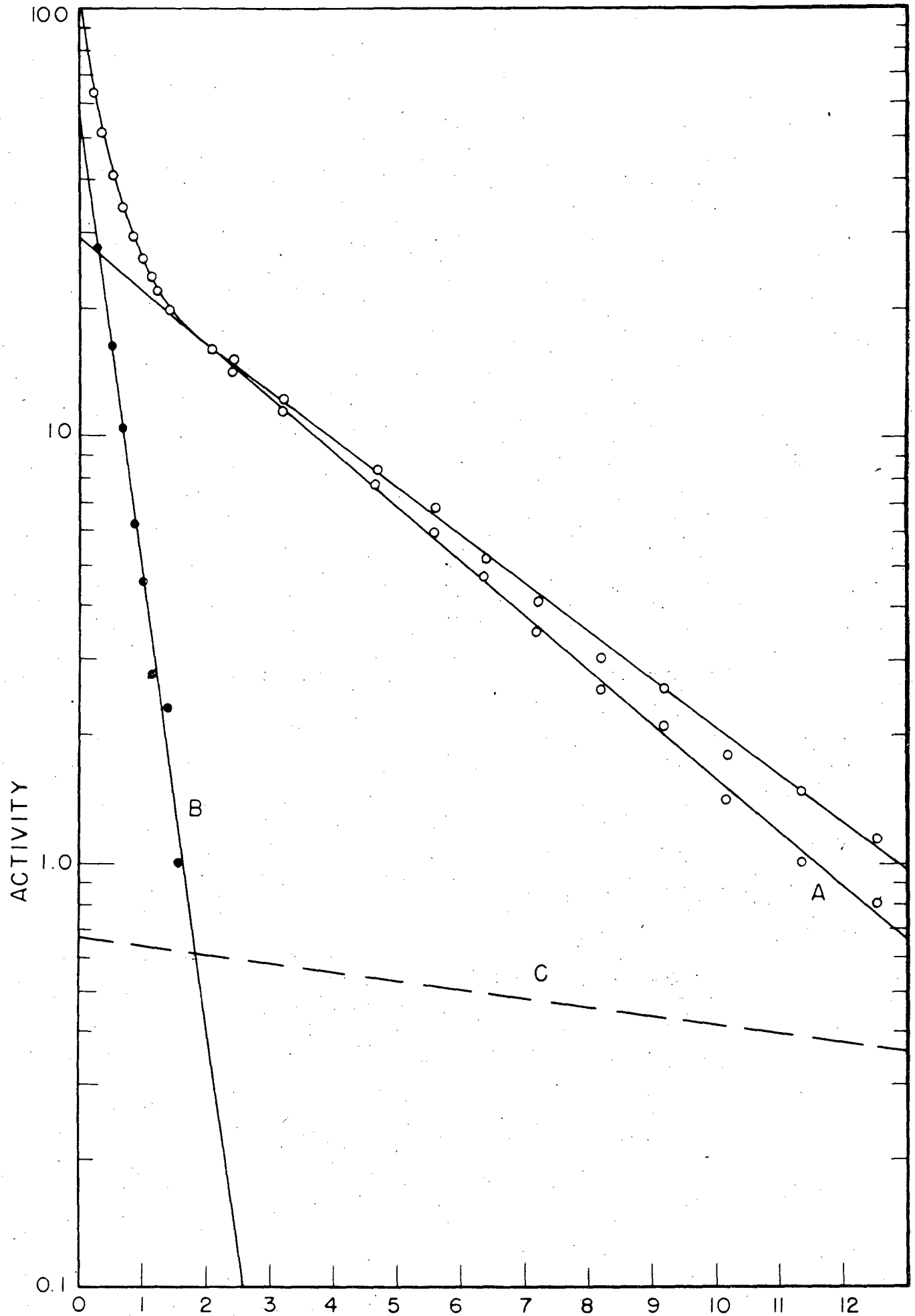


FIG. 1

13428-1

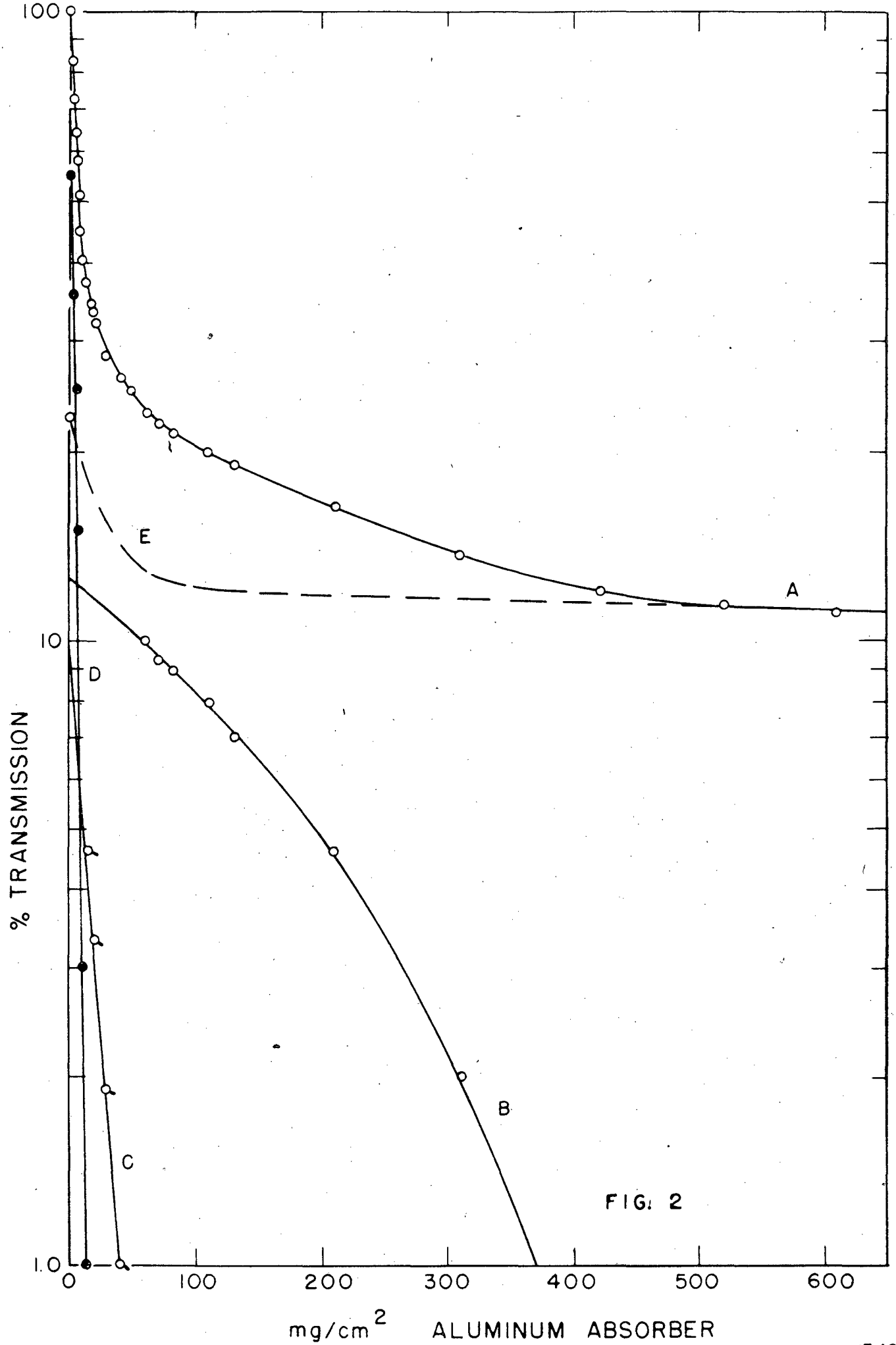

100
10
1.0
% TRANSMISSION
0
100
200
300
400
500
600
mg/cm^2 ALUMINUM ABSORBER
A
B
C
D
E

FIG. 2

13426-1

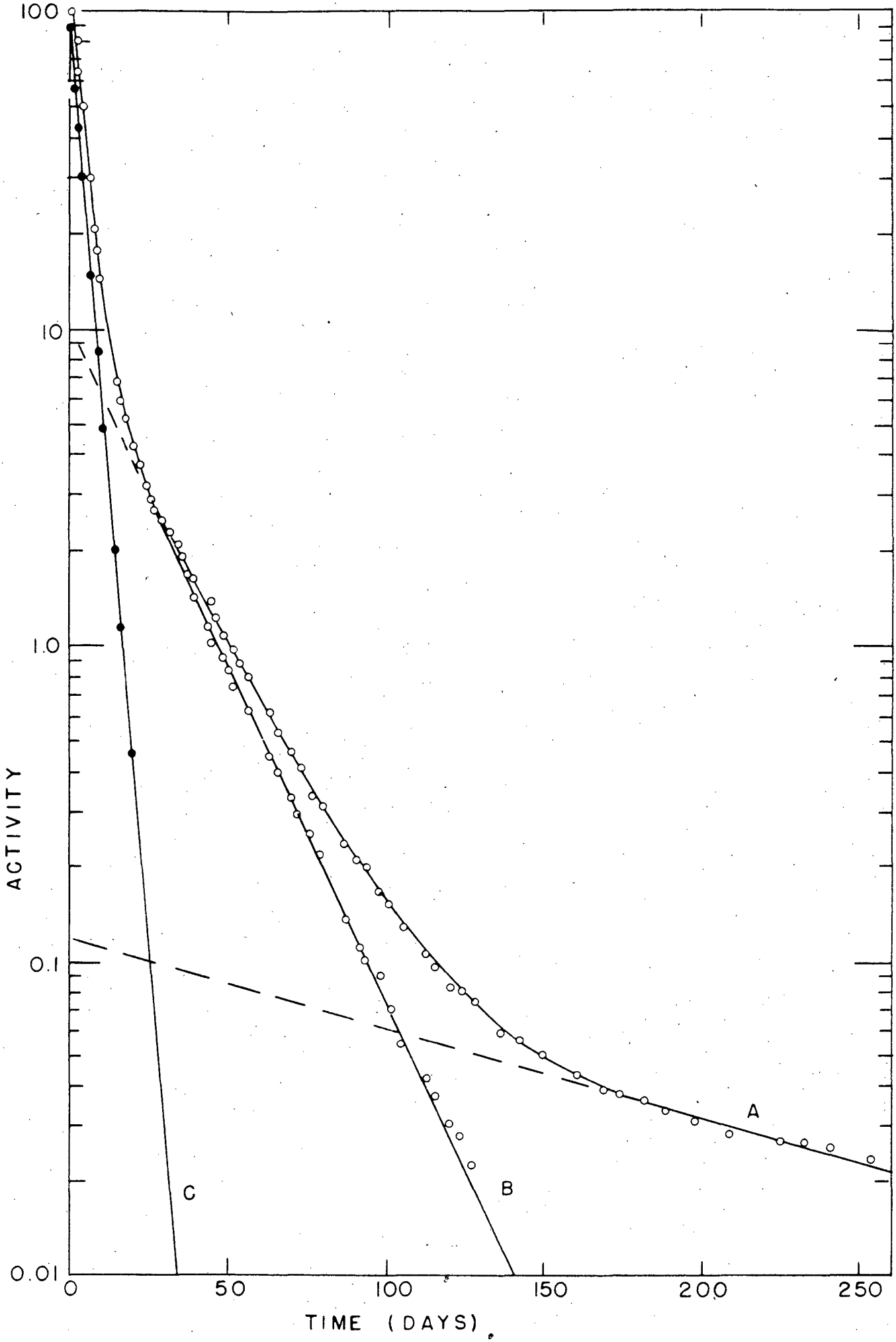
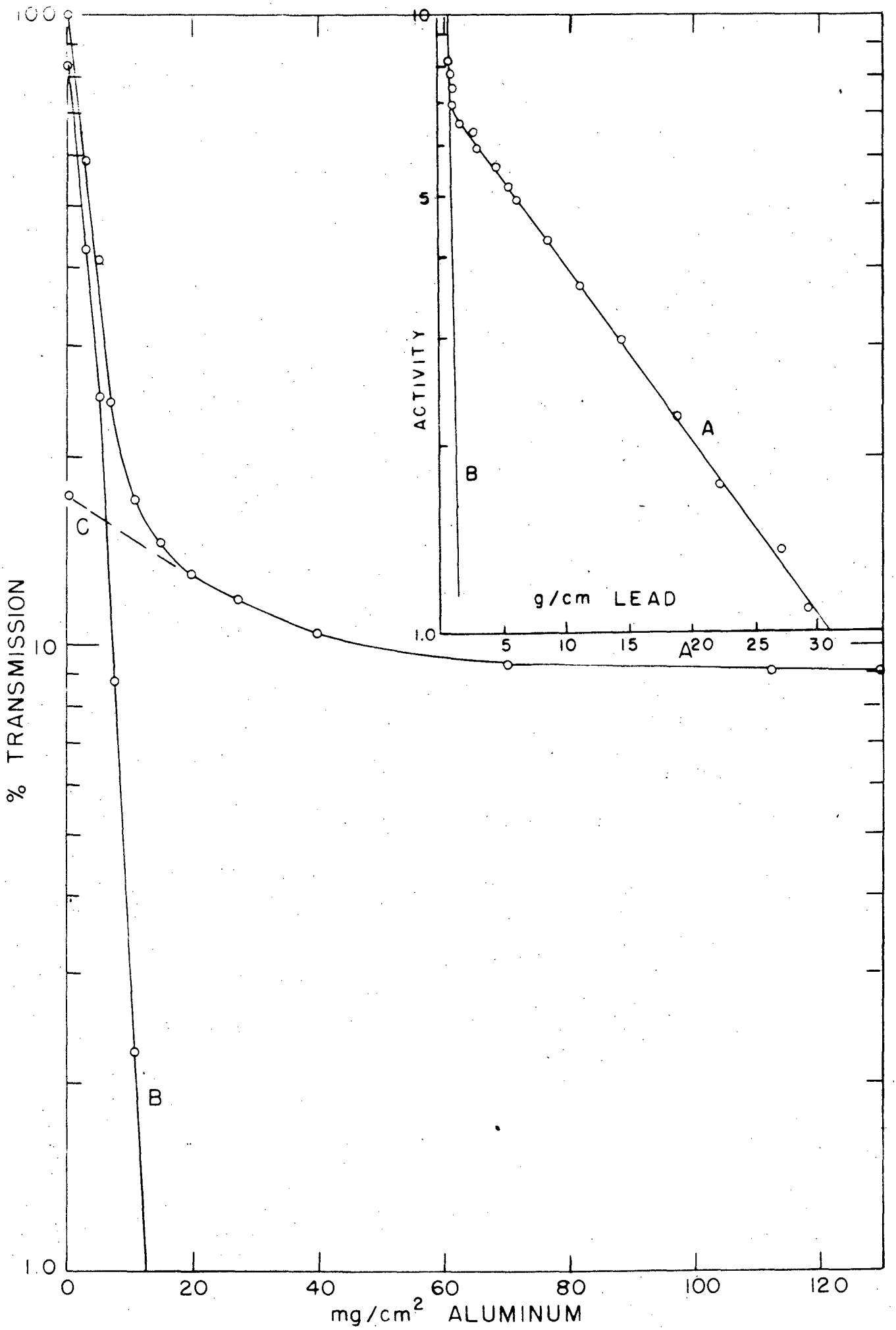


FIG. 3

13427-1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10
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% TRANSMISSION
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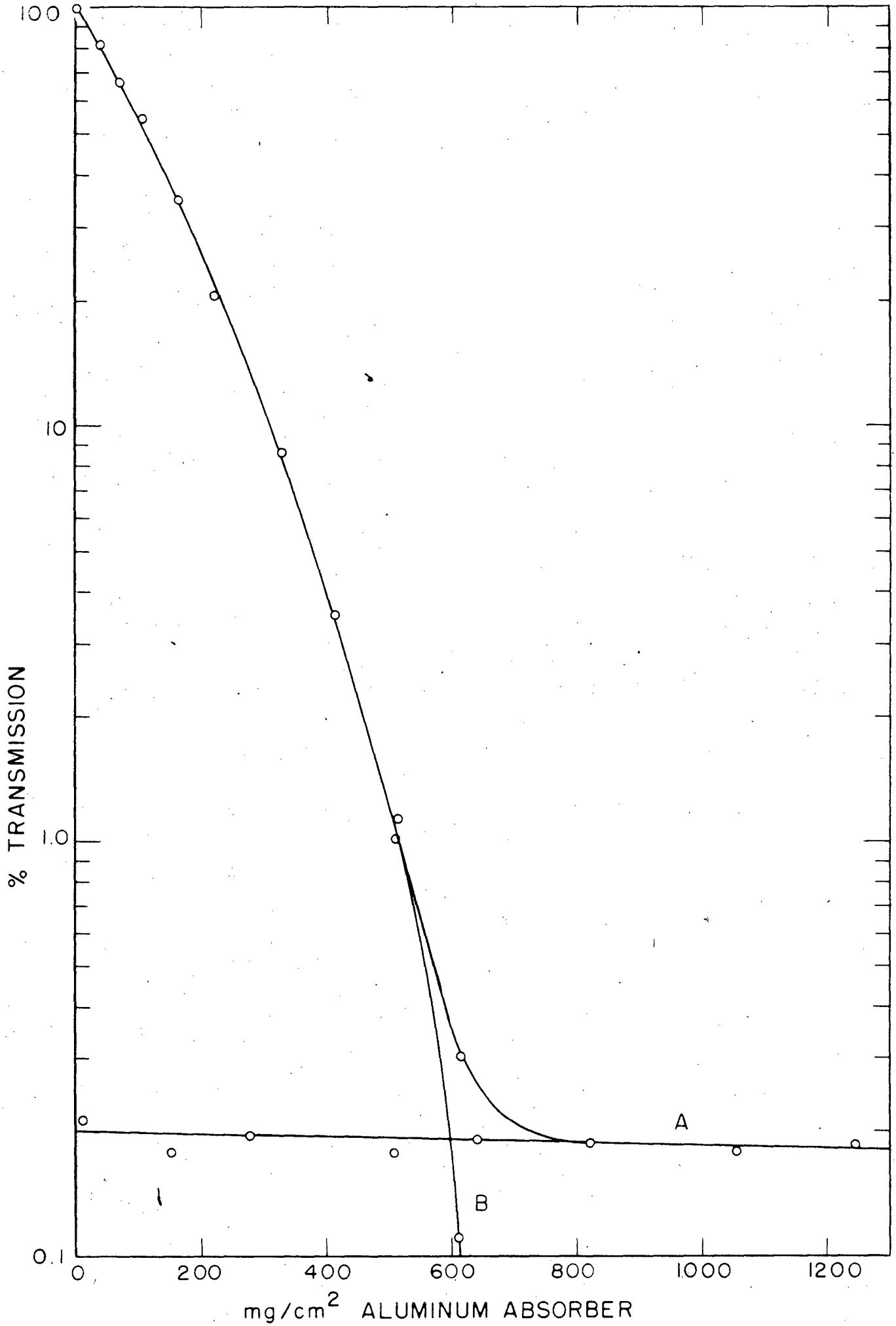
20
40
60
80
100
120
mg/cm² ALUMINUM
B
C
10
5
1.0
ACTIVITY
A
B
g/cm LEAD
5
10
15
20
25
30

FIG. 4

13429-1


100
10
1.0
0.1
% TRANSMISSION
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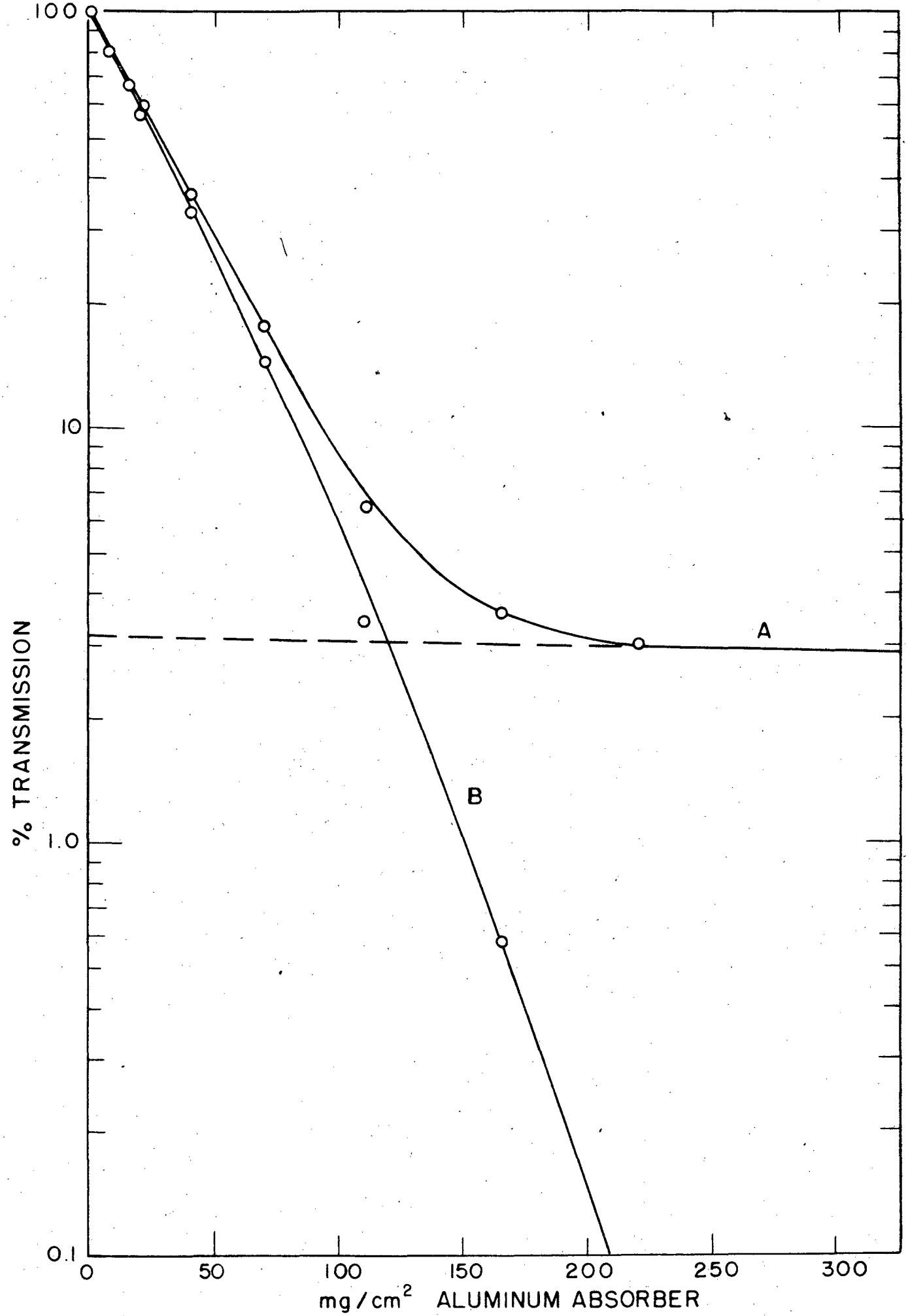
200
400
600
800
1000
1200
mg/cm2 ALUMINUM ABSORBER
A
B

FIG. 5

13425-1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10
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0.1
% TRANSMISSION
0
50
100
150
200
250
300
mg/cm² ALUMINUM ABSORBER
A
B

FIG. 6

13447-1

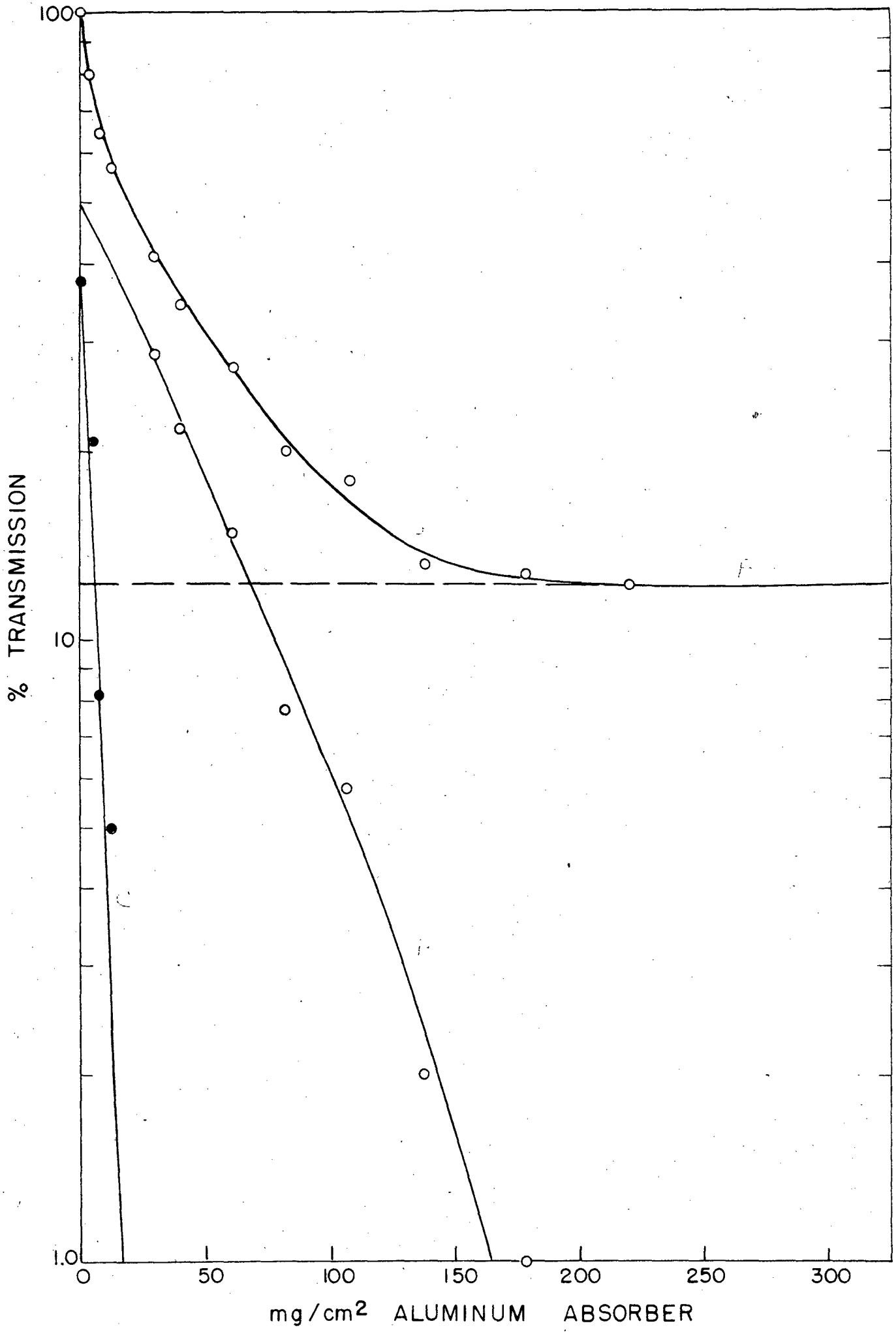
100
10
1.0
% TRANSMISSION
0
50
100
150
200
250
300
mg/cm2 ALUMINUM ABSORBER

FIG. 7

13455-1

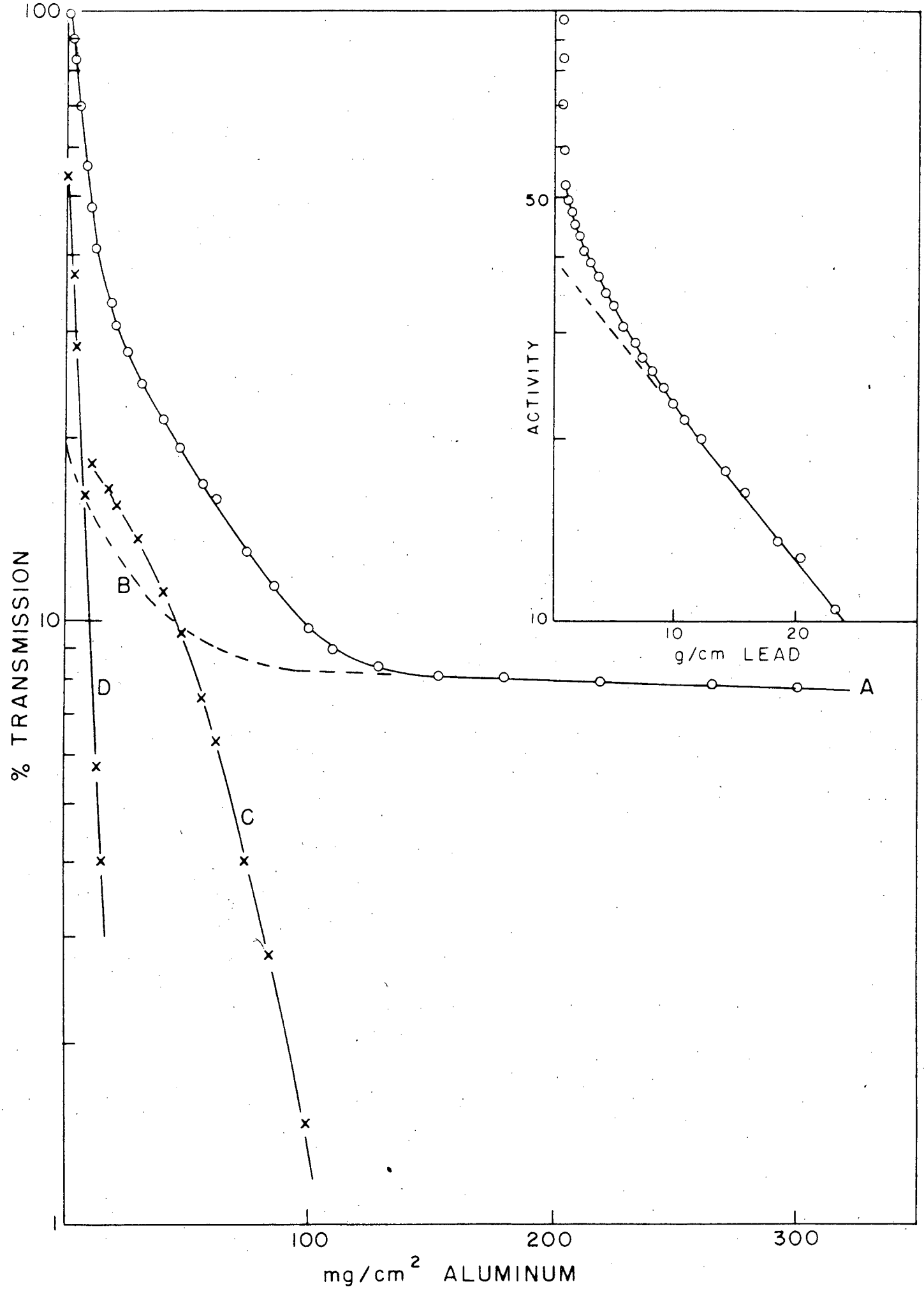
100
10
1
% TRANSMISSION
100
200
300
mg/cm² ALUMINUM
A
B
C
D
50
10
ACTIVITY
10
20
g/cm LEAD

FIG. 8

13981-1

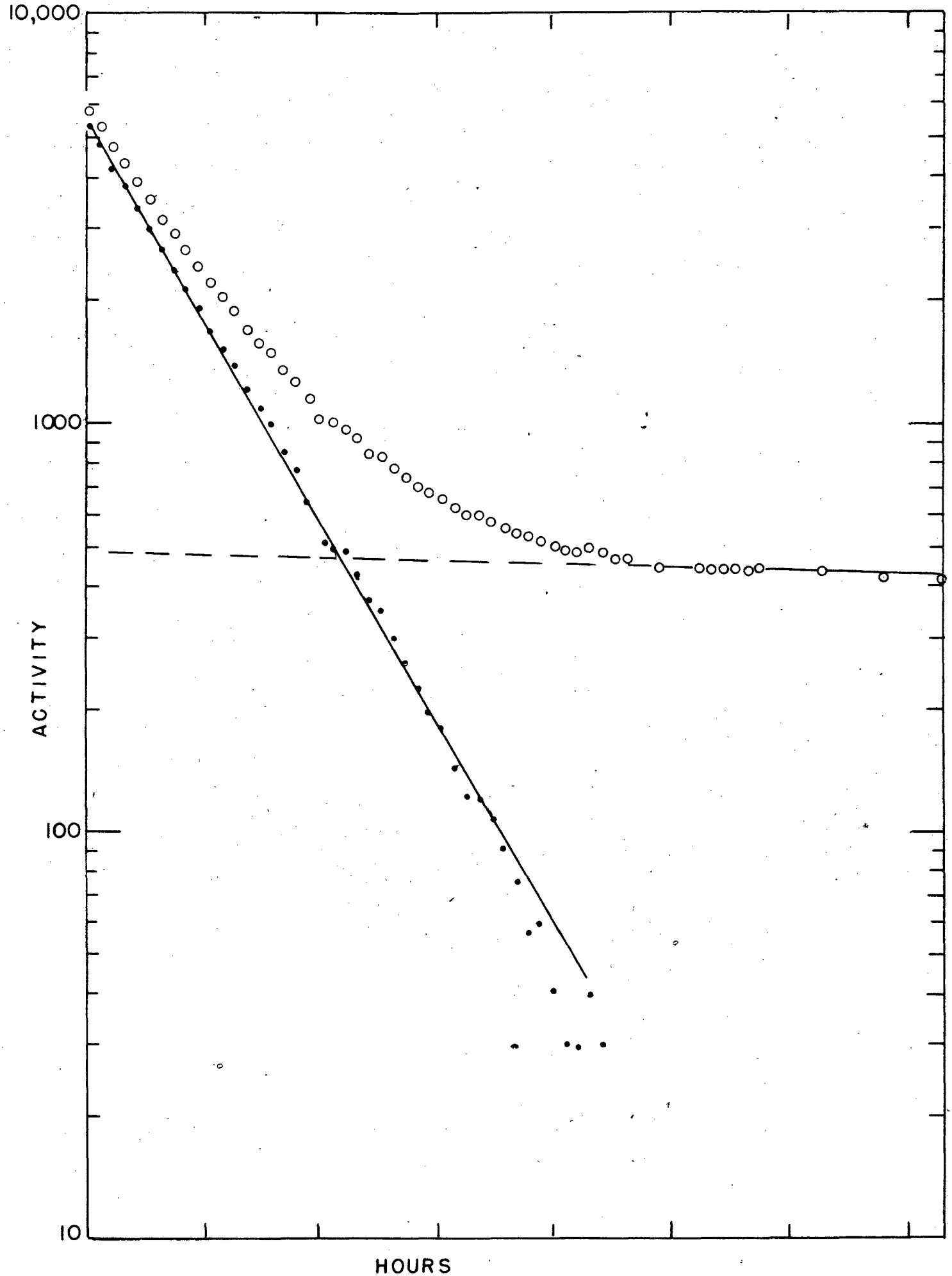

FIG. 9

13982-1

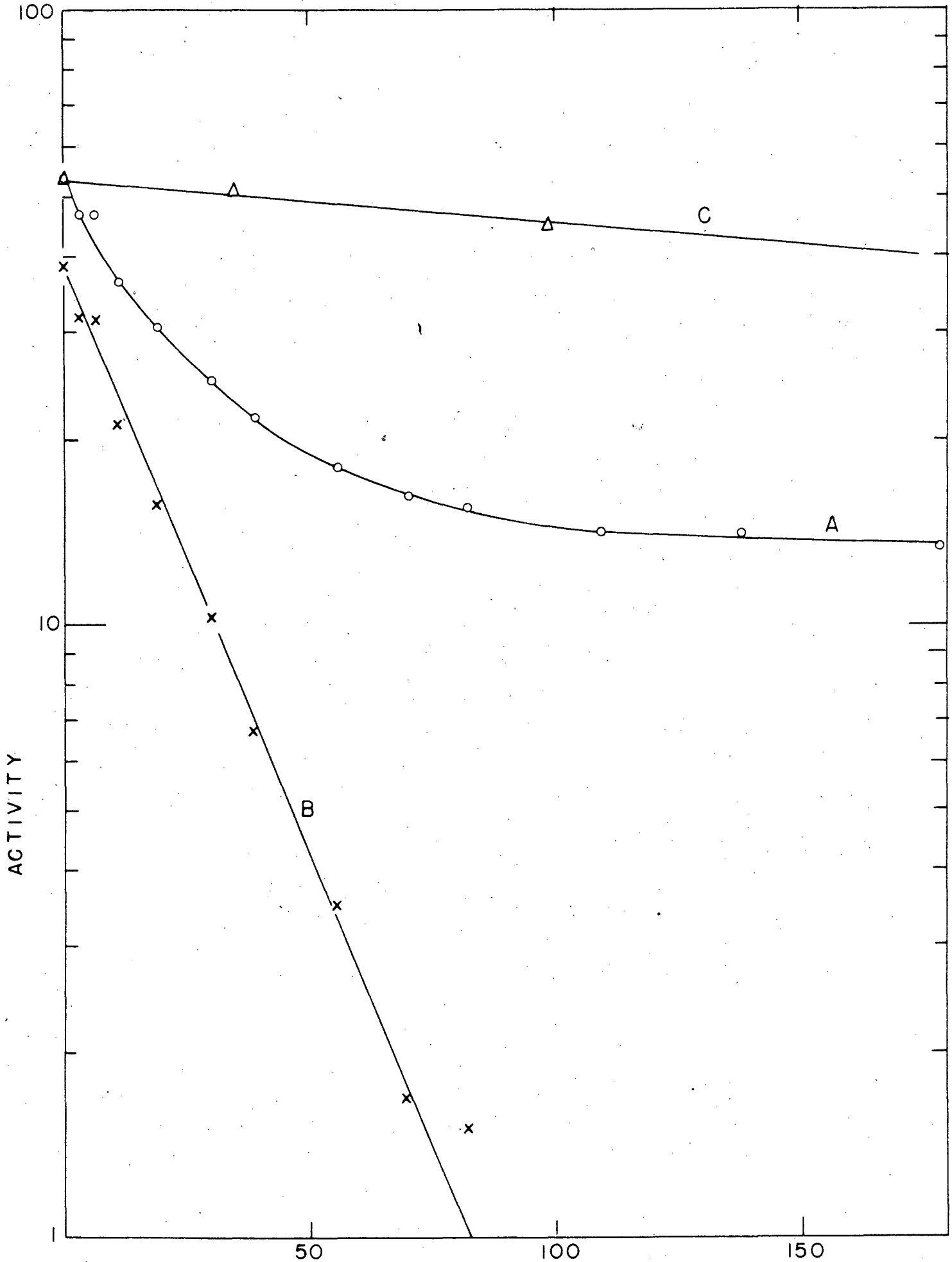

FIG. 10

13983-1

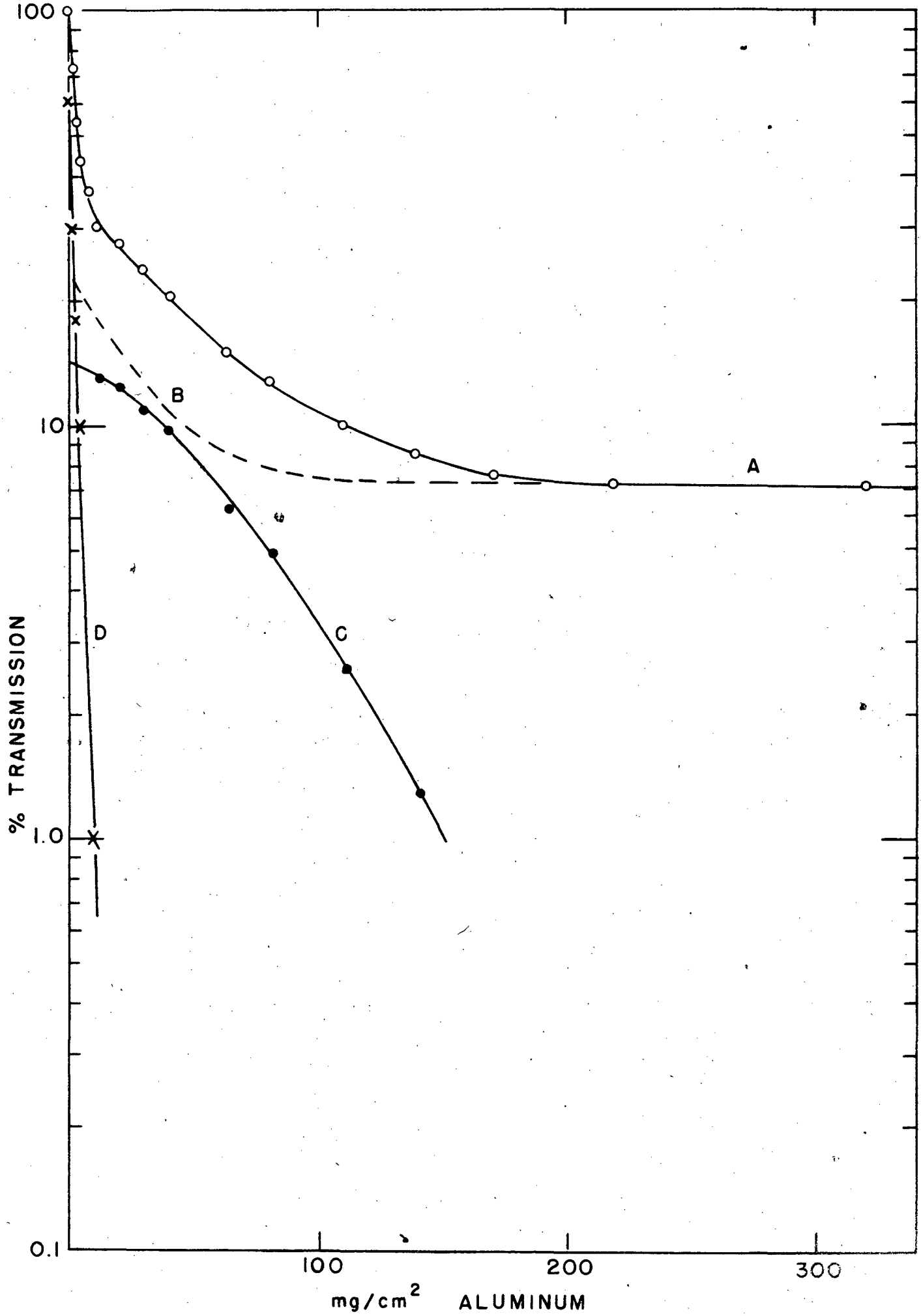

FIG. 11

13984-1