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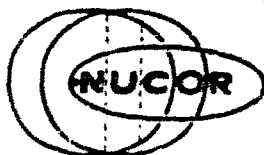
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RESEARCH CHEMICALS

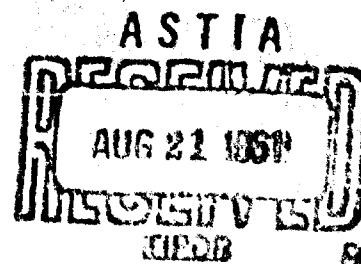
division of nuclear corporation of america

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Burbank, California

La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
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RC 179

RESEARCH CHEMICALS
division of Nuclear Corporation of America
Burbank, California

RARE EARTH INTERMETALLICS

Released to ASTIA by the
Bureau of **NAVAL WEAPONS**
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Third Bi-monthly Report
for the period
March 15 to May 15, 1961

DEPARTMENT OF THE NAVY
Bureau of Naval Weapons
Washington 25, D.C.

Contract NOW 61-0257-c
Control 0266-61

INTRODUCTION

Under contract NOW-61-0257-c, Research Chemicals Division of Nuclear Corporation of America is to investigate intermetallic compositions, emphasizing systems containing one rare earth metal but not limited thereto in a screening program whose criteria are as follows:

- (a) high melting point
- (b) oxidation resistance at elevated temperature
- (c) high strength weight ratio at elevated temperature
- (d) indications of ductility at normal temperatures

SUMMARY OF PROGRESS

Intermetallic compositions have been prepared by the arc melting procedures previously described (1).

Compositions prepared so far on this contract include dysprosium-silicon, hafnium-silicon, hafnium-rhenium and hafnium-boron (2). During this last period compositions of hafnium-boron and yttrium-phosphorus have been examined. Samples with phosphorus coatings were also investigated. All of the phosphorus coated samples showed improvement in corrosion resistance over uncoated alloys. For hafnium, the corrosion was reduced by a factor of ten (10). For zirconium the corrosion was reduced by a factor of two (2). The phosphorus coated molybdenum sample persisted for some time after the uncoated molybdenum sample has almost completely volatilized as molybdic oxide. Phosphorus coatings also improved the corrosion resistance of yttrium metal. Additional samples of hafnium-boron, confirmed previously reported corrosion rates.

Techniques and data obtained. Hafnium-boron compositions have been prepared by inert arc melting of the constituents and the data are shown in Figure 1. Corrosion

rates are in general good agreement with the previous run showing a decrease in corrosion as a function of boron content, when the boron content is greater than four percent. The corrosion process appears to be retarded in this system by virtue of the presence of a viscous fluid material on the surface of the sample. Table I shows sample condition vs total exposure time.

The phosphorus coated samples were prepared by heating metal specimens in quartz tubes with 100 milligrams of phosphorus, under a pressure of approximately 100 microns of argon. These ampules were heated at 350°C for approximately 15 hours after which the temperature was raised to 600°C for 2 hours and to 1000°C for 2 hours. Phosphorus was incompletely reacted in all save the zirconium tube and the unreacted phosphorus remained as a condensed white liquid. Figure 2 shows corrosion rate of zirconium and hafnium metals together with the corrosion rate of the zirconium and hafnium which have been treated with phosphorus. Figure 3 shows the corrosion rate of yttrium metal and yttrium treated with phosphorus. Figure 4 shows the corrosion rate of molybdenum metal and molybdenum samples coated with phosphorus. Table II shows total corrosion versus total time for phosphorus coated samples and Table III the sample condition versus total time. In the case of

yttrium, the phosphorus coated samples showed little corrosion with respect to uncoated yttrium for about one hour after which time the corrosion rates were almost the same.

It would appear from this, that the phosphorus coating inhibits corrosion until sufficient oxidation has occurred, that the material underneath the coating separates the phosphide surface from the sample. In yttrium and molybdenum this was clearly the case as the uncoated samples had approximately the same rate of corrosion as the coated samples near the end of the run. In the case of zirconium this point was reached later in the experiment, but does not show clearly on the graph. At the end of sixteen (16) hours at 1000°C both the zirconium samples look identical. Hafnium, however, has such a slow corrosion rate that the point at which the phosphorus no longer inhibits corrosion has not yet been reached.

The emphasis to date in this program has been on corrosion resistance and melting point, although precise melting point data are not yet available. While these two screening parameters will be further investigated, emphasis in the program will be shifted to determination of mechanical properties of these systems. We have observed in a

qualitative way that while the hafnium-boron compositions are very hard, they are not excessively brittle.

Program for the ensuing period. The forthcoming period studies will include:

- (a) an examination of the strength and ductility of these materials at room and elevated temperatures
- (b) continued screening by means of oxidation studies and melting points

Ductility is here defined as the absence of a brittle fracture. While percent elongation or reduction in area are of significance, of course, in forming operations, the knowledge of the kind of fracture experienced in a given material is more valuable for design considerations in structures. This knowledge will be obtained by an examination of fractured surfaces, or by a comparison, between notched and un-notched specimens in an impact or tensile test.

While we really do not expect intermetallic compounds to be quite ductile, we should like by means of this testing to be able to distinguish between those which are very brittle and those which are merely brittle, and these new compounds may then become commercially useful, as "brittle" cast iron has been for centuries.

REFERENCES

- (1) Research Chemicals; Rare Earth Intermetallics,
RC 167, First Bi-Monthly Report. Research Chemicals
Division of Nuclear Corporation of America, Burbank,
California (December 1960)
- (2) Research Chemicals, Rare Earth Intermetallics,
RC 173, Second Bi-Monthly Report. Research Chemicals
Division of Nuclear Corporation of America, Burbank,
California (March 1961)

TABLE I

SAMPLE CONDITION vs TOTAL TIME IN HOURS

SAMPLE	Orig. Cond.	1/2 Hour	1 Hour	2 Hours	4 Hours	8 Hours
Hf-2%B	Shiny Silver	Dull Dark Grey	Grey Coat	White Coat	White Coat	White Coat
Hf-4%B	Shiny Silver	Dull Dark Grey	White Coat at cracked edge	White Coat at dges	Shiny black spotted white	White Coat
Hf-7%B	Shiny Silver	Dull Dark Grey	Grey spotted white	Black spotted white	Shiny black. spotted white	Shiny black spotted white
Hf-10% B	Shiny Silver	Dull black	Grey	Grey	Shiny black few white dots	Shiny black few white dots
Hf-15% B	Shiny Silver	Dull black	Dull black	Black	Shiny black	Shiny black

TABLE II

PHOSPHORUS COATED SAMPLES
(g/dm²)
TOTAL CORROSION vs TOTAL TIME

<u>SAMPLE</u>	<u>1/2 HOUR</u>	<u>1 HOUR</u>	<u>3 HOURS</u>	<u>4 HOURS</u>	<u>8 HOURS</u>	<u>16 HOURS</u>
Hf	.239	.339	.759	.920	2.954	6.330
Hf-P	.041	.067	.38	.130	.130	.302
Zr	.803	1.635	3.431	4.271	14.160	
Zr-P	.234	.344	1.018	1.585	7.687	29.327
Mo	-5.682	-12.740	-85.086			
Mo-P	.042	-.326	-49.245			
Y	.839	1.840	5.127	6.728	9.639	12.280
Y-P	.667	1.510	4.678	6.253	9.147	12.653

TABLE III

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PHOSPHORUS COATED SAMPLES
SAMPLE CONDITION VS TOTAL TIME

SAMPLE	ORIGINAL CONDITION	1/2 HOUR	1 HOUR	3 HOURS	4 HOURS	8 HOURS	16 HOURS
Hf	shiny silver	pinkish white coat	same	same	lt. yellow crust	same	same
Hf-P	dull black	same	same	same	dull black white spots	same	same
Zr	shiny silver	black white edges	white coat	white crust	lt. green crust	lt. green fragments	-
Zr-P	dull black	same	black white edges	white coat	white crust	white crust	lt. green fragments
Mo	shiny silver	shiny trans- parent crystals	same	sample reduced in size			
Mo-P	dull black	black	shiny trans- parent crystals	sample very small			
Y	shiny silver	tan coat	tan coat	tan crust	tan crust	tan fragments	tan fragments
Y-P	dull black	white coat	white coat	white crust	white crust	white fragments	tan fragments

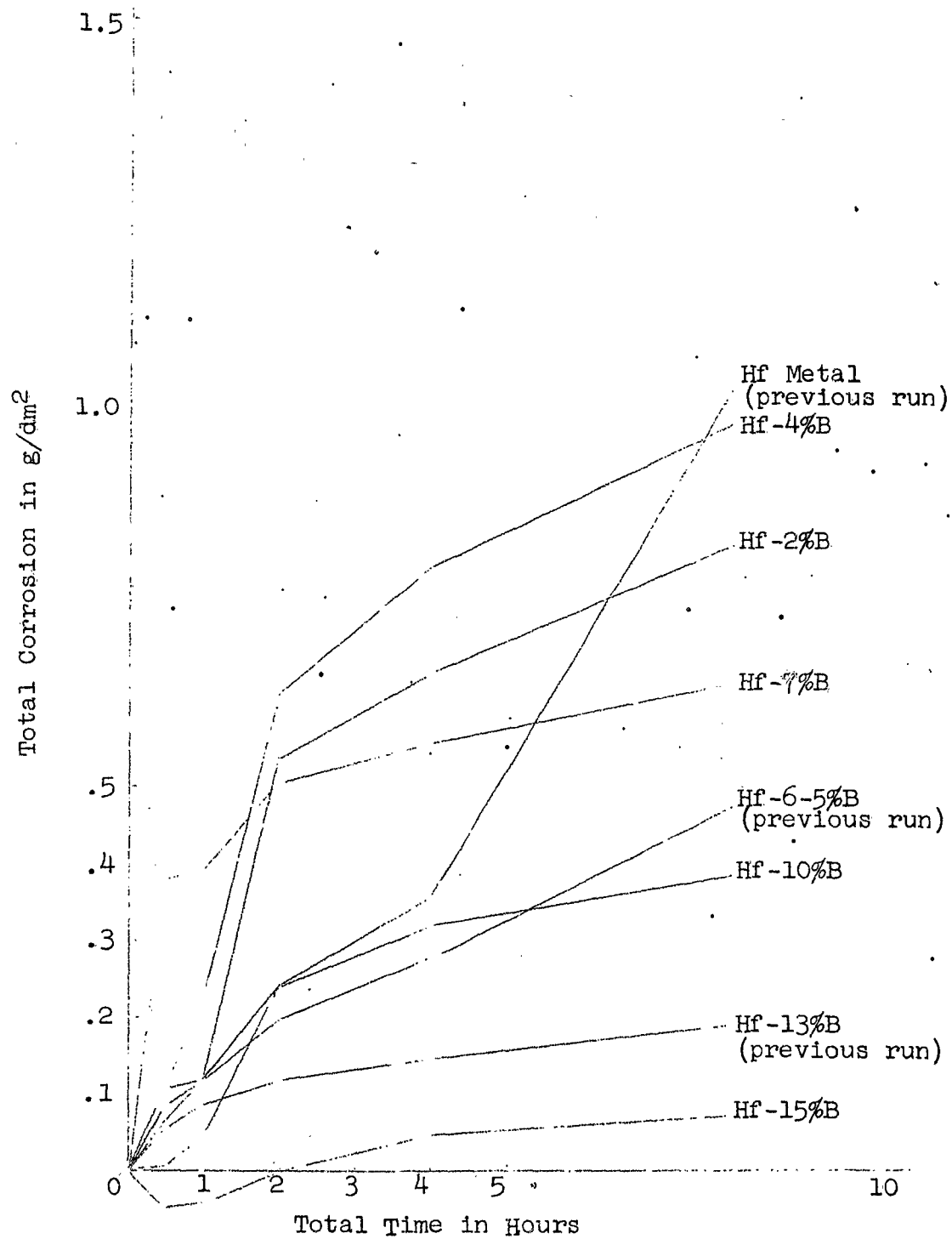


Figure 1 Hf-B System (.1 Scale). Total Corrosion in g/dm^2 vs Total Time in Hours at 1000°C

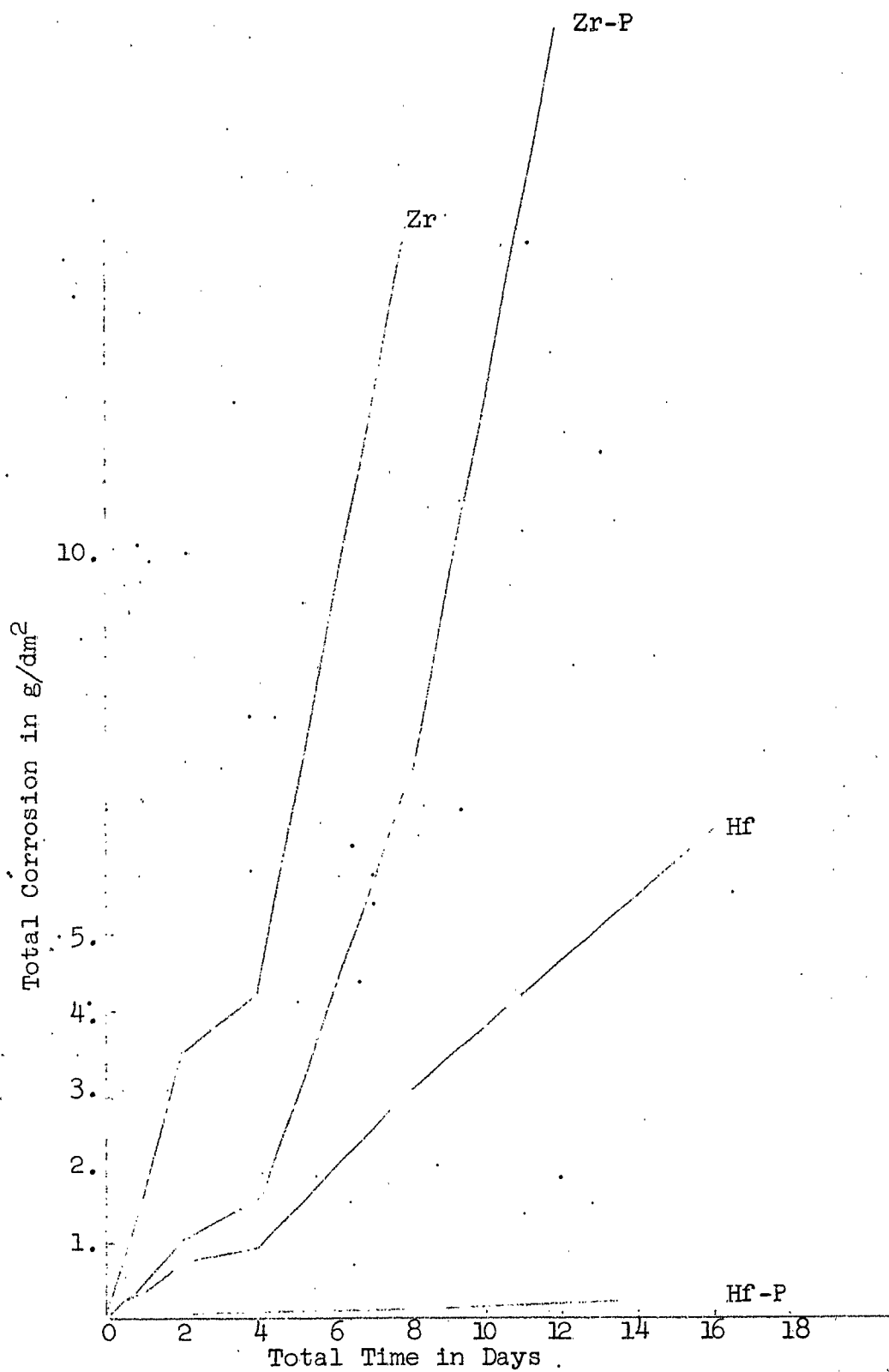


Figure 2 Corrosion Rate of Phosphorus Coated Samples at 1000°C

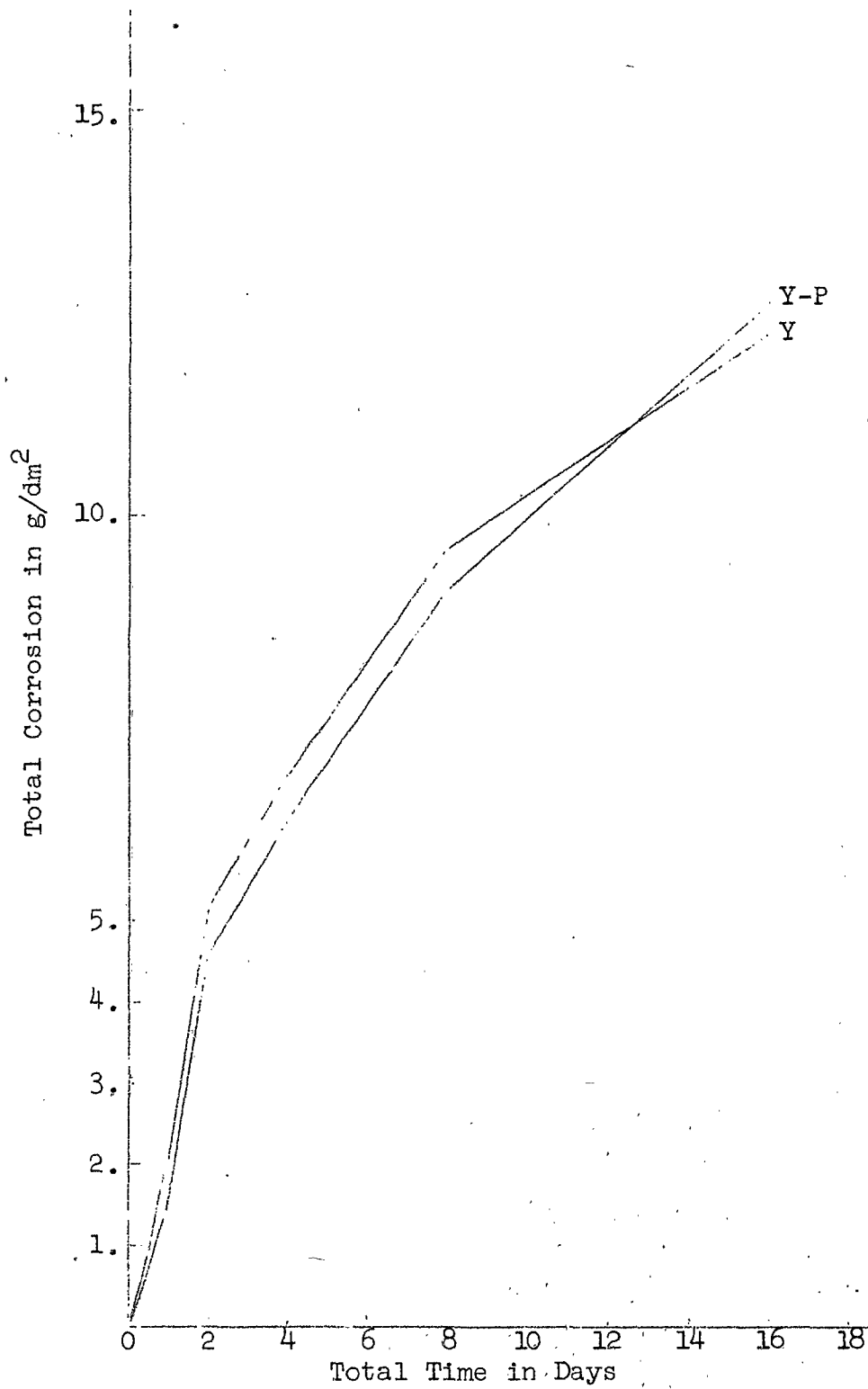
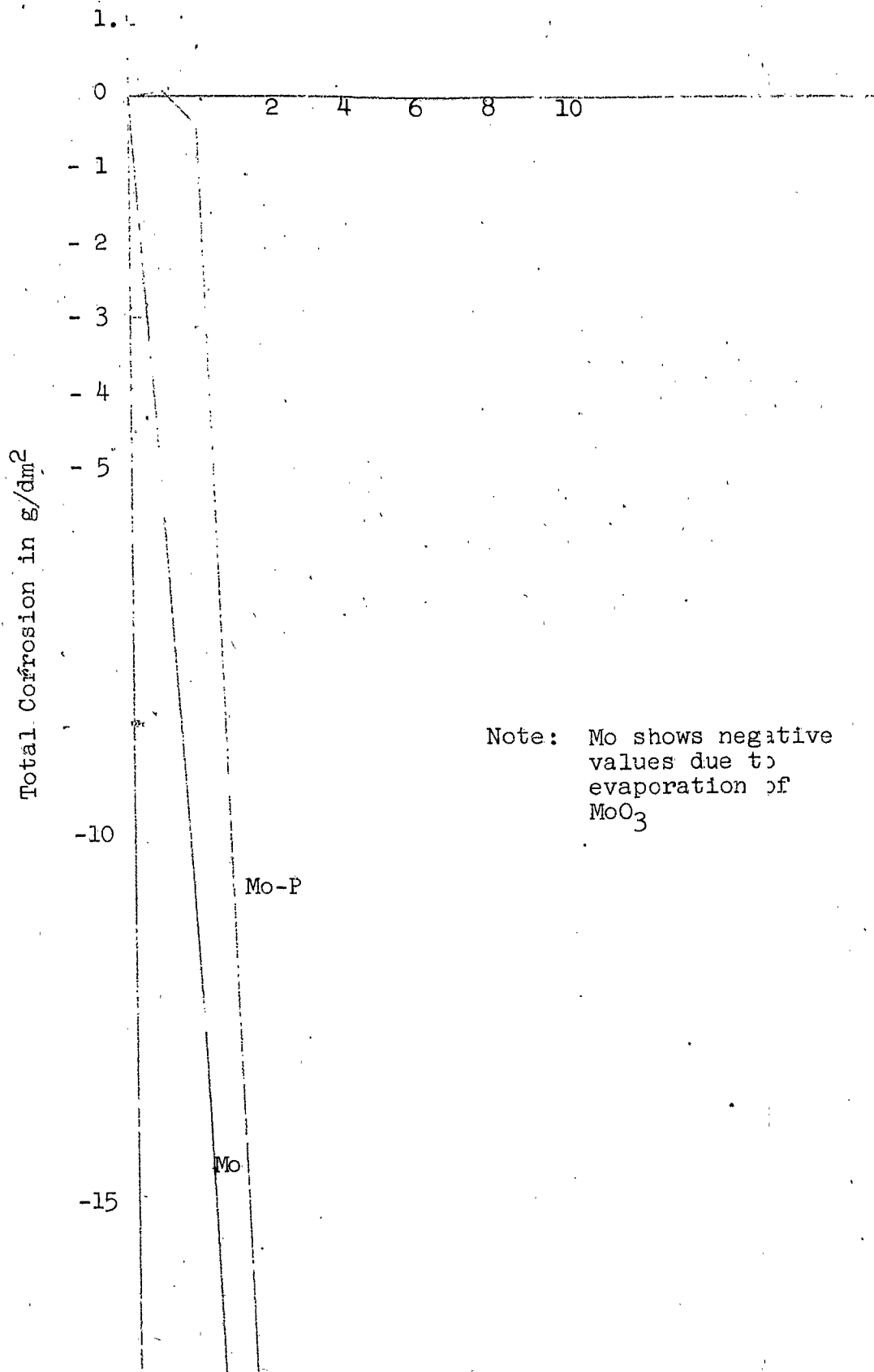


Figure 3 Corrosion Rate of Phosphorus Coated Samples at 1000°C

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Note: Mo shows negative values due to evaporation of MoO_3

Figure 4 Corrosion Rate of Phosphorus Coated Samples at 1000°C