

[CONTRIBUTION FROM THE CHEMICAL LABORATORY OF THE UNIVERSITY OF ILLINOIS]

OBSERVATIONS ON THE RARE EARTHS. XXVI. THE PURIFICATION AND ATOMIC WEIGHT OF ERBIUM

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Introduction

The object of the work described in this paper was to continue the purification of erbium material and to determine the atomic weight of erbium by using the chloride-to-silver ratio.

The accepted value for erbium is based on the work of Hofmann¹ on the oxide-to-sulfate ratio. The average of three determinations gave the value 167.68.

In 1917 Wichers, Hopkins and Balke² prepared erbium material of high purity and attempted to determine the oxide-to-chloride ratio. They found that the oxide, which they prepared by ignition of the oxalate, retained notable quantities of carbon dioxide, even after ignition at a temperature just below 900° for ten hours.

Sources of Material

The material used in the study of this element came from two different sources (1) Texas gadolinite and (2) Series M (bromates).

This material had been treated as described by Driggs and Hopkins³ until fractions could be removed which showed only yttrium and erbium, and which were well removed from those fractions containing holmium on the one hand and thulium on the other. The material in the final fractionation series showed no indications of the presence of any rare earths other than yttrium and erbium. This yttrium-erbium material had been further purified by being run as a nitrate fusion series by F. H. Driggs.

Separation of Yttrium and Erbium

The nitrate fusions were continued, using the furnace built by Driggs and Hopkins for the purification of holmium.³ After some fifty further fractionations, the four head fractions, on analysis by the permanganate method of Gibbs,⁴ showed values which were alike within the limits of experimental error. This close agreement of equivalent weights must be taken as evidence of the removal of yttrium. The arc spectrum of erbium is not sufficiently well mapped to be useful in determining the purity. The observation of the magnetic susceptibilities, as used by Driggs and Hopkins to show the absence of dysprosium from their holmium, is much

¹ Hofmann, *Ber.*, **43**, 2631 (1910).

² Wichers, Hopkins and Balke, *THIS JOURNAL*, **40**, 1615 (1918).

³ Driggs with Hopkins, *ibid.*, **47**, 363 (1925).

⁴ Gibbs, *Am. Chem. J.*, **15**, 546 (1893).

less sensitive in the case of an erbium-yttrium mixture than is the determination of the equivalent weight.

Since Wichers, Hopkins and Balke found that the method of partial precipitation with sodium nitrite gave a rapid separation of yttrium and erbium, some of the yttrium-rich ends of the nitrate fusion series were taken, combined and subjected to fractionation with sodium nitrite. In this way practically pure yttrium material was separated at the one end of the series and erbium material at the other. The two head fractions, 8D and 8E, were taken for analysis.

Preparation of the Reagents for Atomic Weight Study

The purification of the reagents in this investigation was carried out in the same manner as described in earlier publications of this Laboratory.⁵

The erbium nitrate obtained from the fractions 18, 19, 17, 8D and 8E was purified by alternate precipitations as oxalate and hydroxide, the second hydroxide and the second oxalate precipitation being from conductivity water. The final oxalate from each fraction was dried and ignited to the oxide in a platinum crucible.

The anhydrous chloride was formed under the conditions described by Wichers, Hopkins and Balke.

Ratio of Erbium Chloride to Silver

The ratio of erbium chloride to silver was determined in essentially the same manner as described by Driggs and Hopkins on the atomic weight of holmium.

In the following tabulated data the number of the fraction from which the material was taken is shown, together with the weight of the anhydrous chloride, the weight of silver necessary and the atomic weight of erbium. The number in parenthesis shows the determination in which the material had been previously used.

The weighings were made by the method of substitution, the tare flask being of quartz and differing from the reaction flask by only a few milli-

TABLE I

Determination	Fraction number	RESULTS OF DETERMINATIONS			
		Weight of anhydrous chloride	Weight of silver	Ratio	Atomic weight of erbium
I	18	2.25485	2.66270	0.84683	167.69
II	19	0.30165	0.35630	.84663	167.63
III	17	1.02242	1.20761	.84665	167.64
IV	8D	0.38998	0.46068	.84654	167.60
V	8E	.38219	.45144	.84662	167.63
VI (IV)	8D	.17445	.20604	.84668	167.65

Average 167.64

⁵ Kremers, Hopkins and Engle, THIS JOURNAL, 40, 598 (1918).

grams. The weights were standardized to 0.01 mg. and all weighings were corrected to the vacuum standard.

The following densities were used: erbium chloride, 4.1; silver, 10.5; platinum weights, 21.5; platinum plated brass weights, 8.4. The atomic weight of silver was taken as 107.88 and that of chlorine as 35.457.

Summary

1. Fractionation of erbium-yttrium material from gadolinite by the nitrate fusion method yielded erbium material of constant atomic weight, as did also fractionation of similar material by the method of fractional precipitation with sodium nitrite.

2. The ratio of erbium chloride to silver was determined in six analyses. The value 167.64 was obtained for the atomic weight as the mean of the six determinations. This value agrees with the accepted value for this element, 167.7.

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[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY, UNIVERSITY OF PENNSYLVANIA]

A COMPARISON OF TWO METHODS USED IN DETERMINATION OF PHOSPHORUS PENTOXIDE AS MAGNESIUM AMMONIUM PHOSPHATE

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The object of this method was to check the value of the ammonium acetate method¹ in the determination of phosphorus pentoxide as magnesium ammonium phosphate with the earlier method in which ammonium acetate is not used.² The ammonium acetate method is designated A and the earlier method B.

In Method A weighed samples of monopotassium hydrogen phosphate were dissolved in 5–10 cc. of water. To this solution was added several drops of dilute 1:10 hydrochloric acid, then 25 cc. of a 20% solution of ammonium acetate and 30 cc. of magnesia mixture. (The magnesia mixture was prepared by dissolving 55 g. of crystallized magnesium chloride and 105 g. of ammonium chloride in water, adding four drops of 1:10 hydrochloric acid and diluting to a volume of 1 liter.) After heating the solution almost to boiling, several drops of phenolphthalein indicator were added drop by drop with constant stirring until a red color appeared. The solution was allowed to stand until cold (1–1½ hours), then about one-fifth

¹ Schmitz, *Z. anal. Chem.*, **65**, 46 (1924).

² Treadwell and Hall, "Analytical Chemistry," John Wiley and Sons, Inc., New York, 1924, vol. 2, 6th ed., p. 380.