

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 method<sup>1</sup> in the determination of phosphorus pentoxide as magnesium ammonium phosphate with the earlier method in which ammonium acetate is not used.<sup>2</sup> 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

<sup>1</sup> Schmitz, *Z. anal. Chem.*, **65**, 46 (1924).

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

its volume of ammonium hydroxide (sp. gr. 0.95) was added; it was stirred vigorously, allowed to stand for ten minutes and then filtered. After the precipitate was transferred from the beaker the contents of the crucible was well washed with 2.5% ammonium hydroxide. The last washing was made with 4-5 drops of an ammoniacal ammonium nitrate solution (saturated); it was then dried, ignited and weighed.

In Method B the solution containing the alkali phosphate (volume 5-10 cc.) was treated with several drops of 1:10 hydrochloric acid, 30 cc. of magnesia mixture and 20 cc. of a saturated solution of ammonium chloride. After heating the mixture almost to boiling, 2.5% ammonium hydroxide was added slowly, while constantly stirring, until a precipitate began to form, and then the addition of ammonium hydroxide was regulated so that about four drops per minute were added. As the precipitate increased, addition of ammonium hydroxide was quickened until phenolphthalein gave a red color to the solution. After cooling (1-1½ hours), one-fifth of the volume of strong ammonium hydroxide (sp. gr. 0.95) was added, stirred for about one minute, allowed to stand for ten minutes and then filtered. The precipitate was transferred to the crucible, washed well with 2.5% ammonium hydroxide and finally with 4-5 drops of an ammoniacal solution of ammonium nitrate (saturated), dried, ignited and weighed.

The author has found that addition of ammonium nitrate before the ignition is carried out insures formation of a pure white magnesium pyrophosphate. It aids the oxidation of any organic material present which might tend to reduce pyrophosphate in the determination of various phosphates. This procedure is especially desirable in Method A as occasionally a trace of ammonium acetate clings to the precipitate and is easily oxidized by the ammonium nitrate.

All analyses were made from definite quantities of pure recrystallized monopotassium hydrogen phosphate. Each filtrate was allowed to stand for not less than twenty-four hours. Any trace of phosphate could be recognized by rubbing the bottom of the beaker with a stirring rod. This test was found to be entirely satisfactory. All filtrates gave a negative test for phosphorus. The crucible with the precipitate was placed in a porcelain crucible containing a platinum foil in the bottom.<sup>3</sup> The outer crucible was at first heated very gently with a Bunsen burner, the temperature being gradually increased to full red heat. The ignitions were carried out to constant weight with a Méker burner.

Since excellent results were obtained using Gooch crucibles in the determination of arsenic pentoxide as magnesium ammonium arsenate,<sup>4</sup> the same crucibles were used in the determination of phosphorus. With Method A the results obtained from a series of analyses using the porce-

<sup>3</sup> McNabb, *THIS JOURNAL*, 49, 891 (1927).

<sup>4</sup> McNabb, *ibid.*, 49, 1451 (1927).

lain Gooch crucible varied as much as 0.4 mg. with a 0.2000g. sample of phosphate, and there was as great a variation as 0.9 mg. for a 0.5000g. sample.

Due to these variations, the Monroe crucible was substituted. Results of the analyses are given in the table. Ignitions were carried out in the manner described above and gave a snow-white magnesium pyrophosphate. It is easier to carry out the precipitation in the presence of excess ammonium acetate. Acetate ions seem to prevent formation of secondary products and without the acetate it is tedious to produce pure magnesium ammonium phosphate. Sometimes a precipitate resembling aluminum hydroxide will form and is hard to filter, invariably giving high results.

Since phosphorus pentoxide is often determined as magnesium ammonium phosphate in the presence of molybdate, a check of Method A with B was made. According to Treadwell and Hall,<sup>5</sup> "if the solution contains more than 0.1 g. of phosphorus pentoxide, then aliquot parts for the analysis should be used." For this reason only 0.2000 g. of alkali phosphate was used for the determination of phosphorus pentoxide in the presence of molybdate ions. In analyses made by both methods and ignited to constant weight with a Méker burner, some of the ignited products retained a gray color and the results ranged from 0.1 to 0.3% high.

According to McCandless and Burton<sup>6</sup> "precipitates burned properly in a muffle do burn white and the molybdenum present is volatilized almost completely."

The table gives results of analyses using the two methods in the presence of 25 cc. of a 14% solution of ammonium molybdate, the volume of each solution being kept at 150–200 cc. Final ignitions were carried out in an electric muffle at 1000–1100° to constant weight. All ignited products on crushing were found to be white throughout. In the presence of molybdate ions a nice crystalline precipitate is easily formed using either method.

TABLE I  
RESULTS OF ANALYSES  
Method A

KH <sub>2</sub> PO <sub>4</sub> , 0.2000 g.; P <sub>2</sub> O <sub>5</sub> calcd., 0.1043 g.			KH <sub>2</sub> PO <sub>4</sub> , 0.5000 g.; P <sub>2</sub> O <sub>5</sub> calcd., 0.2609 g.		
Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> , g.	P <sub>2</sub> O <sub>5</sub> , g.	P <sub>2</sub> O <sub>5</sub> , diff., g.	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> , g.	P <sub>2</sub> O <sub>5</sub> , g.	P <sub>2</sub> O <sub>5</sub> , diff., g.
0.1635	0.1043	0.0000	0.4097	0.2613	+0.0004
.1633	.1042	— .0001	.4090	.2609	.0000
.1637	.1044	+ .0001	.4094	.2611	+ .0002
.1636	.1044	+ .0001	.4093	.2611	+ .0002
Av. .1635	.1043	.0000	Av. .4094	.2611	+ .0002
Av. % error calcd. from 0.2000g. sample = 0.00.			Av. % error calcd. from 0.5000g. sample = +0.04.		

<sup>5</sup> Ref. 2, p. 382.

<sup>6</sup> McCandless and Burton, *Ind. Eng. Chem.*, 19, 406 (1927).

TABLE I (Concluded)

Method B					
KH <sub>2</sub> PO <sub>4</sub> , 0.2000 g.; P <sub>2</sub> O <sub>5</sub> calcd., 0.1043 g.			KH <sub>2</sub> PO <sub>4</sub> , 0.5000 g.; P <sub>2</sub> O <sub>5</sub> calcd., 0.2609 g.		
Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> , g.	P <sub>2</sub> O <sub>5</sub> , g.	P <sub>2</sub> O <sub>5</sub> , diff., g.	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> , g.	P <sub>2</sub> O <sub>5</sub> , g.	P <sub>2</sub> O <sub>5</sub> , diff., g.
0.1638	0.1045	+0.0002	0.4088	0.2608	-0.0001
.1636	.1044	+ .0001	.4086	.2606	- .0003
.1631	.1040	- .0003	.4083	.2605	- .0004
.1633	.1042	- .0001	.4087	.2607	- .0002
.1635	.1043	.0000	Av. .4086	.2607	- .0002
Av. .1635	.1043	.0000			
Av. % error calcd. from 0.2000g. sample = 0.00.			Av. % error calcd. from 0.5000g. sample = -0.04.		

Method A			Method B		
In presence of molybdate ions					
KH <sub>2</sub> PO <sub>4</sub> , 0.2000 g.; P <sub>2</sub> O <sub>5</sub> calcd., 0.1043 g.			KH <sub>2</sub> PO <sub>4</sub> , 0.2000 g.; P <sub>2</sub> O <sub>5</sub> calcd., 0.1043 g.		
Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> , g.	P <sub>2</sub> O <sub>5</sub> , g.	P <sub>2</sub> O <sub>5</sub> , diff., g.	Mg <sub>2</sub> P <sub>2</sub> O <sub>7</sub> , g.	P <sub>2</sub> O <sub>5</sub> , g.	P <sub>2</sub> O <sub>5</sub> , diff., g.
0.1633	0.1042	-0.0001	0.1635	0.1043	0.0000
.1635	.1043	.0000	.1634	.1042	- .0001
.1634	.1042	- .0001	.1634	.1042	- .0001
.1634	.1042	- .0001	.1634	.1042	- .0001
Av. .1634	.1042	- .0001	.1634	.1042	- .0001
			Av. .1634	.1042	- .0001
Av. % error calcd. from 0.2000g. sample = -0.05.			Av. % error calcd. from 0.2000g. sample = -0.05.		

### Conclusions

1. A comparison of the two methods of Schmitz in determination of phosphorus pentoxide as magnesium ammonium phosphate has been made. The ammonium acetate method is designated as Method A and the one in which ammonium acetate was not used designated as Method B. Four to five drops of an ammoniacal solution of ammonium nitrate was used in the last washing to insure complete oxidation of any organic material occasionally found in the analysis of phosphates.

2. All analyses were made from definite quantities of pure, recrystallized monopotassium hydrogen phosphate.

3. Using Method A, results obtained with porcelain Gooch crucibles and ignition carried out to constant weight with a Méker burner varied from 0.4 mg. in a 0.2000g. sample to 0.9 mg. in a 0.5000g. sample of phosphate.

4. Using Monroe crucibles and igniting to constant weight with a Méker burner, Method A gave an average percentage error of +0.02 calculated from weight of samples ranging from 0.2000 g. to 0.5000 g. Method B gave an average percentage error of -0.02.

5. In applying both methods in the presence of molybdate ions high results ranging from 0.1 to 0.3% were obtained and some of the ignited products were of a gray color. Igniting in an electric muffle between

1000–1100° gave a white magnesium pyrophosphate. Both methods gave an average error of  $-0.05\%$  (0.2000g. sample of phosphate).

6. The advantage in Method A is that in the absence of molybdate ions it is easier to produce a crystalline precipitate.

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## THE ACTION OF ULTRAVIOLET LIGHT ON SOME COLLOIDAL DISPERSIONS OF GOLD<sup>1,2</sup>

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### Introduction

The study of the effect of radiation upon colloidal dispersions of gold has been the object of numerous investigations. The earliest of these dealt with the reducing action of light upon gold salts, with the accompanying formation of a colloidal dispersion.<sup>3</sup> The accelerating effect of light during the reduction of gold salts by various reducing agents has also been known for some time.<sup>4</sup>

A comprehensive study of the significance of light in the formation and stability of colloidal gold has been made by Nordenson.<sup>5</sup> His paper contains a valuable survey and criticism of previous work on this problem. He investigated the effect of light upon the reduction of chlorauric acid by hydrogen peroxide. Reduction proceeds in the darkness or diffuse daylight with the formation of a coarse, bluish suspension. The exposure to the radiation of a quartz mercury-vapor lamp, during the reduction, leads to the formation of stable sols. The degree of dispersion and the color depend upon the length of the exposure. The sols prepared in this manner were examined spectrophotometrically as well as with the ultramicroscope and the dependence of the properties upon time of exposure was demonstrated. In addition he observed a coagulating effect of the

<sup>1</sup> The abstract of a thesis presented to the Graduate Faculty of Columbia University by Ralph H. Muller, October, 1925, in partial fulfillment of the requirements for the degree of Doctor of Philosophy.

<sup>2</sup> Presented at the September, 1926, Meeting of the American Chemical Society.

<sup>3</sup> (a) Hellot, "Hist. de l'Acad. de Scien.," 1737, p. 101; (b) Scheele, see Plotnikow, "Lehrbuch der Photochemie," 1920, p. 361; (c) Krüss, *Liebig's Ann. Chem.*, 43, 237, 238, 276 (1887); (d) Rumford, Eders "Photochemie," p. 179; (e) Juch, Eders "Photochemie," p. 179; (f) Fulhame, *Ann. chim.*, 26, 58 (1798).

<sup>4</sup> (a) Pelletier, *Schweigg Jour.*, 31, 317 (1800); (b) Herschel, "Hunt's Researches on Light," 1844; (c) Döbereiner, *Schweigg Jour.*, 62, 86 (1831); (d) Sonstadt, *Eders Jahrbuch f. Phot. Ind.*, 1899, 466.

<sup>5</sup> Nordenson, *Inaug. Diss.*, Univ. of Upsala, 1914; *Z. physik. Chem.*, 90, 603 (1915).