found it satisfactory, but also determined the arsenic which had been volatilized.

Lead arsenate.	Magnesium pyroarsenate. Found.	Calculated.
0.2965 gram gave	0.1022 gram.	0.1028 gram.

Two portions of a mineral containing lead, arsenic acid, and phosphoric acid, were exposed to the action of the acid gas. The volatilized arsenic equaled in (a) 10.42 per cent, and in (b) 10.39 per cent. None remained in the residue contained in the boat.

Experiments have not yet been made with antimonates.

Separations of vanadic and arsenic acids from tungstic and chromic acids are now being carried out, and with encouraging results. The action of hydrobromic, hydriodic and hydrofluoric acid in gas form upon vanadates, nitrates, arsenates and phosphates, has received attention with results which will appear later.

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[CONTRIBUTION FROM THE JOHN HARRISON LABORATORY OF CHEMISTRY. NO. 5.]

URANIUM OXYNITRIDE AND URANIUM DIOXIDE.

BY EDGAR F. SMITH AND J. MERRITT MATTHEWS.

Received May 31, 1895.

THE action of ammonia upon molybdenyl (MoO_2Cl_2) and tungstyl (WO_2Cl_2) chlorides has received attention in this laboratory. To complete the study of Group VI in this direction, uranyl chloride (UO_2Cl_2) was prepared, placed in a porcelain boat and heated in a glass tube in a current of dry ammonia. At a comparatively moderate temperature the material began to take on a dark color, and copious fumes of ammonium chloride were evolved. The heat was raised and continued until the ammoniacal salt was no longer produced. The residue was dull black in color. When fused in a nickel crucible with caustic potash, ammonia was slowly evolved. A portion of the substance introduced into aqueous silver nitrate caused the separation of a beautiful deposit of brilliant crystals of metallic silver. Chlorine was not found in the compound. When it was heated with sulphuric acid (1 : 2) in a sealed tube, complete solution ensued. Upon titration with standardized potassium permanganate, the presence of 6.83 per cent. of dioxide was disclosed. No hydrogen was discovered in the material examined for it. Four analyses were made and resulted as follows:

0.2311 gram of material gave 84.81 per cent. uranium.
0.3139 " " " " 2.19 " " nitrogen.
0.2412 " " " " 84.87 " " uranium.
0.1868 " " " " 2.24 " " nitrogen.

The mean of the uranium and nitrogen determinations was 84.84 per cent. uranium and 2.21 per cent. nitrogen, leaving a difference of 12.95 per cent. for oxygen. The formula we have deduced from these figures is $U_{11}N_5O_{25}$, which requires 84.88 per cent. uranium, 2.25 per cent. nitrogen, and 12.87 per cent. oxygen.

Uhrlaub[1] mentions an oxynitride of uranium which from its analysis approximates the formula $U_9N_4O_{18}$.

From further experiments made by us it is evident that these oxynitrides are bodies of extremely variable constitution, dependent upon the temperature and length of time to which the uranium body is exposed to the action of the ammonia. This conduct was also observed to prevail with the corresponding derivatives of both molybdenum and tungsten.

Preparation of Uranium Dioxide.—The preparation of this compound has been the subject of much discussion. It will be remembered that this is the oxide long looked upon as metallic uranium. Zimmermann[2] found that it resulted upon merely heating the oxide U_3O_8 in indifferent gases, such as carbon dioxide. Others have recommended the ignition of urano-uranic oxide in hydrogen, or together with sulphur, or with sulphur and ammonium chloride. We submit the course by which we obtained it. The oxide U_3O_8 was intimately mixed with a large excess of dry ammonium chloride in a porcelain crucible, and this then placed in a larger Hessian crucible and surrounded with closely packed charcoal. The crucibles were heated in a wind furnace at almost a white heat for about six hours. A reddish brown substance remained. It contained no nitrogen or chlorine. Two portions of it were dissolved in nitric acid and the uranium determined with these results: 88.23 per cent. and

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[1] Verbindungen einiger Metalle mit Stickstoff, Göttingen, 1859.

[2] Annalen, **232**, 283.

88.00 per cent., giving as a mean 88.12 per cent. A third portion, weighing 0.3363 gram, was heated for fifteen minutes over a Bunsen flame; the increase in weight was 0.0119 gram, and heating again for forty-five minutes there resulted an alteration of only 0.0011 gram, after which the weight continued constant. The total increase of the material taken by its conversion into urano-uranic oxide, was therefore 0.0130 gram, while the calculated increase should be 0.0132 gram.

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THE SEPARATION OF IRON FROM BERYLLIUM.

BY ELIZABETH A. ATKINSON AND EDGAR F. SMITH.

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THIS separation is generally effected through the solubility of beryllium hydroxide in ammonium carbonate. It is, however, not always satisfactory, and any method affording better results will be of interest to the analyst.

Ilinski and v. Knorre[1] called attention to the use of nitroso-β-naphthol in analytical work. For example, they separated cobalt from nickel and iron from aluminum by means of this reagent. Inasmuch as their study has not been continued, it seemed to us of interest to ascertain whether or not the same reagent could be used for the separation indicated at the head of this communication. We used the following solutions: (1) a fifty per cent. acetic acid solution of the nitroso-β-naphthol, (2) a ferric solution of which ten cubic centimeters contained 0.1278 gram of ferric oxide, and finally a beryllium chloride solution of which ten cubic centimeters contained 0.1248 gram.

The first step taken was to learn how completely the iron was precipitated by the proposed reagent. To this end ten cubic centimeters of the ferric solution ($=0.1278$ gram Fe_2O_3) were diluted with two hundred cubic centimeters of distilled water, and one hundred and twenty-five cubic centimeters of the nitroso-β-naphthol added to the cold liquid, which was then allowed to stand for a period of twenty-four hours. The iron precipitate was then filtered out, washed at first with fifty per cent. acetic acid, and subsequently with water, until a few drops of the washings left no residue upon evaporation and ignition upon a strip

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[1] *Ber. d. chem. Ges.*, **18**, 699, 2728.