

*Summary.*—It has been shown that the photochemical decomposition of ammonia at low pressure into nitrogen and hydrogen is sensitized by mercury vapor when the radiation used is from a cooled quartz mercury-arc lamp and is filtered so as to remove wave-lengths below 2340 Å. The so sensitized decomposition has been found to be accompanied by diffuse-band fluorescence in the green and ultra-violet.

<sup>1</sup> Kuhn, *Compt. Rend.*, 177, 956 (1923).

<sup>2</sup> Coolidge, *J. Am. Chem. Soc.*, 45, 1637 (1923).

<sup>3</sup> Haber and Kerschbaum, *Zt. Elektrochem.*, 20, 296 (1924).

<sup>4</sup> G. Cario and J. Franck, *Zt. Physik*, 11, 161 (1922).

<sup>5</sup> Phillips, *Proc. Roy. Soc.*, 89, 44 (1913).

<sup>6</sup> Van der Lingen and Wood, *Astrophys. J.*, 54, 149 (1921).

<sup>7</sup> Wood, *Phil. Mag.*, 50, 774 (1925).

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## THE CONCENTRATION AND IDENTIFICATION OF THE ELEMENT OF ATOMIC NUMBER 61

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Communicated November 8, 1926

In making measurements of the wave-lengths of the X-ray *K* emission lines for the rare-earth elements,<sup>1</sup> very faint traces of lines corresponding to the *K* series of the element of atomic number 61 appeared on the plate with certain specimens of samarium (atomic number 62) and neodymium (atomic number 60). In tracing these compounds to their source a consistent effort has been made over a period of two years to increase the concentration of the element of atomic number 61. Many of the samples obtained have shown definite traces of lines where they should be expected in both the *K* and *L* X-ray wave-length regions but the photographic spectra have until now never been strong enough to permit photographic reproduction in a printed article and this was deemed necessary before the announcement of the discovery of the element was made.

In 1923, two of the authors commenced examining rare earth minerals for element 61. The minerals, which have been thoroughly investigated by employing large quantities, comprise gadolinite, xenotime (ytterspar) and monazite. These three minerals show an interesting variation in their rare earth constituents. Monazite is essentially a phosphate of the cerium group elements carrying a few per cent of the yttrium earths; gadolinite consists largely of the yttrium group with some of the cerium

group; ytterspar is just the reverse of monazite, in that it is composed of phosphates of the yttrium earths, with small amounts of those from the cerium group. The minerals were decomposed and the neodymium-samarium content separated by the usual methods of fractionation. In the case of gadolinite and monazite this Nd-Sa portion had to be put through an exhaustive fractionation before even the faintest line of 61 could be discerned. On the other hand, the ytterspar material gave faint lines after just a few crystallizations.

Ytterspar proved to be of exceptional interest owing to the fact that it was a very curious variety which carried a very much larger proportion of gadolinium and dysprosium than any other mineral met with. The solution of the rare earths, which showed only weak absorption bands of neodymium, gave upon treatment with sodium sulphate a copious precipitate of double sulphates. Since these were practically white, it was at first supposed that they consisted mainly of lanthanum and cerium. However, upon conversion to the double magnesium nitrates and fractional crystallization, it was found that the major part of the material quickly passed into the fractions more soluble than those of neodymium and samarium. These double nitrates gave an almost colorless solution showing weak dysprosium bands, indicating that the chief constituent was gadolinium.

In 1914 in an article upon terbium<sup>2</sup> it was stated that certain fractions coming between terbium bromate and the less soluble gadolinium salt showed some faint absorption lines attributed to neodymium. It would be concluded from this that Nd bromate was less soluble than Tb bromate. Apparently contradictory to this conclusion, J. Zernike in the winter of 1924-25 while attempting to facilitate the separation of Ho from Dy and Pr from Nd, by fractionating a mixture of the bromates of Pr, Nd, Ho, Dy and Tb, observed that terbium was less soluble than neodymium.

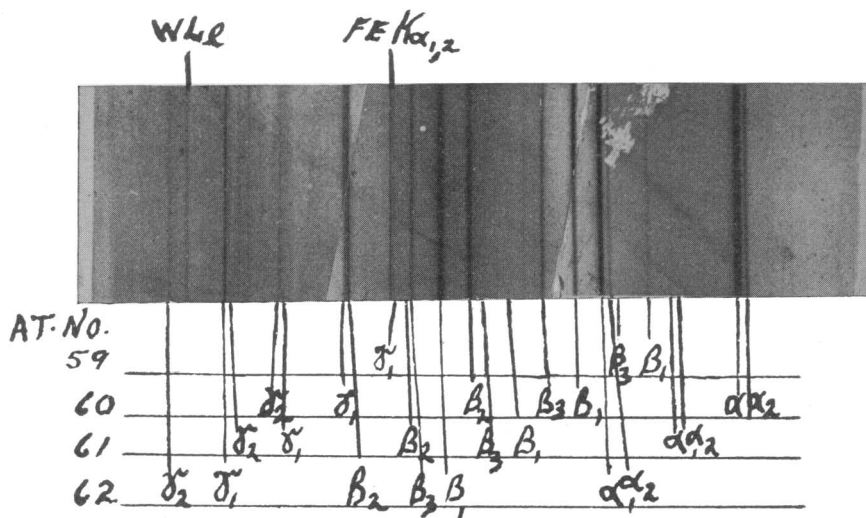
These conflicting results would indicate either that two elements giving similar absorption bands exist or that the solubility curves of the bromates of Nd and Tb cross at about ordinary temperature. The latter possibility could be easily investigated by a very careful determination of the solubility curves of these salts. Such work was undertaken and it was found that the curves of the above-mentioned compounds did not cross between zero and 45°. Neodymium bromate proved to be more soluble than terbium bromate.

A careful examination of the fractions of bromates more soluble than terbium obtained by Bissell and one of the authors, as previously mentioned, showed the complete absence of neodymium absorption bands. This would indicate that the faint absorption bands shown in addition to that of terbium in the terbium gadolinium fractions were due to element 61.

In order to confirm the presence of element 61, the minute amount of neodymium which accompanied the yttrium earths derived from a very large quantity of Brazilian monazite sand was roughly concentrated by fractionally crystallizing the bromates and lastly the double magnesium nitrates. This neodymium concentrate which was expected to carry the major portion of element 61 was then submitted to X-ray examination.

*X-Ray Identification.*—Figure 1 shows a reproduction of the X-ray *L* series lines obtained by means of a Siegbahn vacuum spectrograph. Three overlapping plates were taken to cover the complete region. The displacement of the lines with atomic number serves as a very positive method of identification. Besides obtaining lines in the position in which they would be expected, it is further necessary to show the impossibility of their existence due to impurities.

The target of the X-ray tube contained iron and copper, while tungsten



lines always appear due to depositions on the target from the heated filament. The powdered sample was rubbed lightly on the face of the target at the beginning of each exposure.

This sample contained the elements samarium (62), neodymium (60), praeodymium (59) and a slight amount of cerium (58) in addition to (61). The estimated amount of oxalate of element (61) was from 1 to 1.5 per cent.

While there are more than twenty *L* series lines for each element only about 7 of these are fairly strong, namely  $\alpha_1$ ,  $\alpha_2$ ,  $\beta_1$ ,  $\beta_2$ ,  $\beta_3$ ,  $\gamma_1$ ,  $\gamma_2$  (Siegbahn notation). All seven of these lines for element 61 are observable on the original plates although some of them are rather faint in the reproduction. The strongest line is  $\alpha_1$ . This lies approximately midway between the

corresponding lines for elements 60 and 62 as do all of the other lines for element 61. Using the values of Siegbahn for elements 60 and 62 the wave-lengths of the  $L$  series lines for element 61 are as follows:

$\alpha_2$	$\alpha_1$	$\beta_1$	$\beta_2$	$\beta_3$	$\gamma_1$	$\gamma_2$
2.289	2.279	2.078	2.038	1.952	1.799	1.725

That the  $\alpha_1$  line is not due to impurities may be shown. Some of the possibilities of a line in this neighborhood are V  $K_1^\beta$  (2.280), Cr  $K_1^\alpha$  (2.285), Ce  $L_6^\beta$  (2.277) and La  $L_9^\beta$  (2.277). If the lines were due to the  $K$  series of any element the most prominent lines would be the  $\alpha_1\alpha_2$  doublet similar to that for Fe shown in the figure. A test was made for the  $\alpha_1\alpha_2$  lines of vanadium and traces of it were found, but the intensity was such that the  $\beta_1$  line having an intensity much less would be difficult to observe. The  $L_6^\beta$  line of cerium (58) would have an intensity of about one-tenth that of the  $\beta_1$  line for cerium. The  $\beta_1$  line, however, is just barely discernible, therefore any of the  $\beta$  lines with subscripts greater than three would be too faint to appear. Further, along with the  $\alpha_1$  line appears the  $\alpha_2$  line in its proper place and intensity. While it is difficult to observe this in the reproduction, it was sufficiently strong to be measured by the comparator on the original plates. While the other lines attributed to 61 are less distinct, an analysis of the possibility of their being due to impurities, particularly in the case of  $\beta_1$ ,  $\beta_3$  and  $\gamma_2$ , confirms their assignment.

<sup>1</sup> *Physic. Rev.*, **25**, p. 197.

<sup>2</sup> *J. Amer. Chem. Soc.*, **36**, p. 2060.

## THE DENSITY OF OXYGEN AND ITS COMPRESSIBILITY BELOW ONE ATMOSPHERE. II

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Read before the Academy November 8, 1926

Experiments upon the normal density of oxygen have recently been reported.<sup>1</sup> The final result of these experiments for the weight of the normal liter, 1.42901, is subject to a correction of  $-0.00003$ , owing to the use in the calculations of an incorrect value for the force of gravity at the Coolidge Laboratory. If the true value, 980.399, is employed, the corrected result of all our earlier experiments is 1.42898, a value which is identical with the result of the last three and unquestionably the best series. This corrected value has already been used in computing the atomic weight of helium.<sup>2</sup>