

common lead nitrate and 1.7991 for uraniolead nitrate. The outcome of this work, then, by finding no appreciable difference in the molal solubility of the different samples of lead nitrate containing isotopes of widely different atomic weights, confirms earlier work upon other properties of these interesting substances. Evidently weight (or mass) is the prime distinguishing feature of the two kinds of lead here studied, as it was in other cases. This work and that described in the preceding section thus afford further support for the hypothesis due to Russell, Fleck, Soddy and Fajans.

<sup>1</sup> This paper and the two following are taken from an abbreviated version of a report to the Carnegie Institution of Washington (which generously subsidized the investigations) presented in August, 1918.

<sup>2</sup> Richards and Yngve, *J. Amer. Chem. Soc.*, *Easton, Pa.*, **40**, 1918, (164).

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## THE PURIFICATION BY SUBLIMATION AND THE ANALYSIS OF GALLIUM CHLORIDE

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A new method for the purification of gallium salts was worked out in some detail. This rests upon the convenient fact that gallium trichloride sublimes and distills at a low temperature, whereas the other chlorides likely to be associated with it are much less volatile. The method rested, therefore, upon fractional distillation and sublimation, at first in a stream of chlorine and afterwards in a vacuum, of impure gallium chloride.<sup>1</sup> The apparatus was a complex affair, in which gallium could be first burned in pure dry chlorine and then subjected to distillation either first in pure chlorine, then in nitrogen, and finally in a vacuum, the whole apparatus being fused together without rubber connections and scrupulously dried. In order to avoid the use even of ground joints (with their attendant alternative danger of leakage or contamination from lubricant) the gas connections were opened by means of sealed magnetic hammers acting on enclosed capillaries, and closed by fusion of the glass connections. The gallium was provided through the great kindness of Mr. F. G. McCutcheon of the Bartlesville Zinc Company, Blackwell, Oklahoma, to whom grateful thanks are due. Three successive distillations of the trichloride of gallium were made in chlorine at 220° to 230°C., three more at about 175° (the melting point of gallium dichloride), three in nitrogen at 90° to 110°, and five sublimations in vacuo at 65° to 80°—fourteen in all. Distillation in nitrogen or in vacuo is needful to eliminate dissolved chlorine. The resulting product showed no trace of any other substance in its spark spectrum when examined with great care in a Hilger wave length spectrometer. Although this study did not reach a final stage, and much more

remains to be done in order that perfect certainty may be attained, nevertheless the outcome must be regarded as promising in pointing toward an adequate and convenient method of separating gallium from other metals.

Three fractional samples of the chloride discussed in the previous section were analyzed, using the usual methods employed in Harvard University in order to make a preliminary determination of the atomic weight. The samples were small and for various reasons the results cannot be considered as anything more than preliminary, but the outcome is, nevertheless, worth recounting. In the last and best determination 0.43947 gram of gallium chloride (weighed in vacuo in a sealed glass tube) yielded 1.07087 grams of silver chloride having required 0.80587 gram of silver for complete precipitation. The atomic weight of gallium computed from these two sets of data are respectively 70.09 and 70.11—concordant results indicating a value somewhat higher than that usually accepted for gallium, but near enough to show that the chloride was at least not far from being pure, and that the whole proceeding is capable, when employed with larger quantities of material and with the experience already gained, of affording an accurate evaluation of this atomic weight. Of course such meagre data as those thus far secured furnish no worthy evidence concerning it; they are as merely preliminary as the data of Lecoq de Boisbandrau. At the conclusion of the war it is hoped that the joint investigation (which was stopped by the departure of W. M. Craig into War Service) may be continued and completed.

<sup>1</sup> The principle of this method has since been published by Dennis and Bridgman, although the details of their treatment differed from ours. Their work was entirely independent of ours (which was brought to a close in March, 1918) and was entirely unknown to us. *J. Amer. Chem. Soc., Easton, Pa.*, 40, 1918, (1540).

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### THE PURIFICATION OF GALLIUM BY ELECTROLYSIS, AND THE COMPRESSIBILITY AND DENSITY OF GALLIUM

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The method of separating gallium from indium by means of the different solubilities of the hydroxides in caustic alkali, recommended by various authorities, was tested without success. The separation was found to be so incomplete that several per cent of indium remained in the gallium, at least under the conditions used in our work, and it seemed clear that this difference in solubility is not enough to effect a complete separation. Much more promising results were obtained by the electrolytic method. Gallium occupies a place in the electrolytic series between indium and zinc. It is far less easy to deposit than indium, but, on the other hand, much more easy to deposit than zinc. By carefully regulating the hydrogen-ion concentration