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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 distils 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 of impure gallium chloride at first in a stream of chlorine and afterwards in a vacuum. [1] The apparatus was a complex affair, in which gallium could be burned in pure dry chlorine and then subjected to distillation 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 con-

[1] The principle of this method has meanwhile 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. THIS JOURNAL, 40, 1540 (1918). We agree entirely with their conclusions.

nections 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°, three more at about 175° (the melting point of gallium dichloride) three in nitrogen at 90 to 110°, and 5 sublimations *in vacuo* at 65 to 80°—14 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 for 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 g. of gallium chloride (weighed *in vacuo* in a sealed glass tube) yielded 1.07087 g. of silver chloride, having required 0.80587 g. of silver for complete precipitation. The atomic weights 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 meager data as those thus far secured furnish no worthy evidence concerning it; they are as merely preliminary as the data of Lecoq de Boisbaudran. The joint investigation was temporarily suspended by the departure of W. M. Craig into the Chemical Warfare Service; but he has now returned to the University and the work is being continued. This investigation and that described in the following brief paper were generously subsidized by the Carnegie Institution of Washington.

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