

In order to determine whether or not an allylic transformation also results during the formation of the Grignard reagent, we have converted all of these bromide preparations containing variable quantities of  $\alpha$ -methylallyl bromide, into the magnesium compound under identical conditions. The resulting butene mixtures should be of constant composition regardless of the composition of the bromides if an allylic equilibrium is involved; if not, the butene mixtures should have compositions corresponding to those of the bromide preparations. The results of these experiments will be available in the near future.

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#### TESTS FOR ELEMENT 87 (VIRGINIUM) BY THE USE OF ALLISON'S MAGNETO-OPTIC APPARATUS

*Sir:*

In December, 1930, one of us (McGhee) handed out by number to Professor Allison twelve (to him) "unknowns" which were tested by him and checked by two assistants 100% correctly in three hours. The improved model of his machine which was later installed in our laboratory under Dr. Allison's direction has been proved to be reliable by numerous tests on known solutions of pure substances. No two, different, metallic salts of the same acid produce minima that coincide. Hence we read with interest the footnote to an article by Papish and Wainer [THIS JOURNAL, 53, 3818 (1931)] in which it was suggested that "solutions of  $\text{SnCl}_4$  and  $\text{ReCl}_2$  gave minima which were coincident with those obtained for element 87," possibly due to the formation of the complex ions,  $\text{SnCl}_3^+$  and  $\text{ReCl}^+$ .

To test this question, hydrochloric acid solutions of the minerals samarskite, pollucite and lepidolite, and of crude cesium chloride were made up. Element 87 (Virginium) had been reported in all of them. When observations were made on these solutions within the scale limits between which  $\text{ViCl}$  would fall, twelve minima were found. Similar observations on the sulfates of the minerals yielded only six minima. Since the same number of minima are always found for any one metal, no matter to which acid radical it might belong, it was thought that six of the minima appearing in the chloride region might indeed be due to  $\text{ReCl}^+$  and  $\text{SnCl}_3^+$ . With this in view, a solution of pure  $\text{SnCl}_4$  was made up and examined for minima. In addition to the minima of  $\text{SnCl}_4$  previously determined, four minima at 44.20, 44.40, 44.98, 46.06 were found. Likewise a solution of  $\text{ReCl}_2$  (obtained by adding hydrochloric acid to pure potassium perhenate) was examined for minima. Beside those previously attributed

to  $\text{ReCl}_2$ , minima at 45.93 and 47.54 were observed. The four minima of  $\text{SnCl}_4$  and the two of  $\text{ReCl}_2$  which occur at the scale readings given above, coincide with six of those observed in hydrochloric acid solutions of the minerals tested. The other six, which belong to Virginium, occur at 44.01, 44.62, 45.11, 46.22, 46.42, 47.80. *There are no corresponding minima for rhenium and tin in sulfate solutions of these minerals. None of the minima attributed to  $\text{ViCl}$  was found in the solutions of stannic chloride or rhenium chloride.*

A careful observer need not confuse the minima of Virginium with those of rhenium and tin since the minima of Virginium are in no case nearer than 2.5 cm. to the minima of these elements. In this work each of us has made ninety independent observations, sixty-three of which agree within 1 mm. each with the other; and twenty within 2 mm., while no other variation exceeds 3 mm.

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#### DRY DISTILLATION OF COPPER PHTHALATE DOES NOT YIELD FLUORANE

Sir:

In THIS JOURNAL, 52, 3003 (1930), Ekeley and Mattison published a paper entitled "Fluorane from the Dry Distillation of Copper Phthalate." In a recent letter to me (the senior author), L. P. Kyrides of the Monsanto Chemical Works stated that the results as described in the above paper could not be duplicated in his laboratory. I regret to say that I also am unable to duplicate them, the distillate evidently being an impure phthalic anhydride. In 1923 at my suggestion the junior author of the paper performed the experimental work. It never occurred to me to doubt the accuracy of the melting point determinations and the two sets of combustion data as reported by her, since the product she presented dissolved in concentrated sulfuric acid with yellow-green fluorescence. Publication of the results was withheld until similar experiments could be performed on copper tetrachlorophthalate which seemed to point to the formation of dodekachlorofluorane, though analyses by another experimenter no nearer than within 1% of the theoretical were ever obtained. The final responsibility for the publication of the 1923 results on copper phthalate rests upon me, since I should have verified the junior author's data before publication and thus have avoided the erroneous statement appearing in the literature.

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