

rigorous purification the m. p. can be raised to 88–89°. On catalytic hydrogenation the alcohol absorbs 0.9–1.0 mole of hydrogen.⁶

Three grams of a non-alcoholic liquid were recovered from the volatile fraction obtained during the above ethyl borate treatment. The substance polymerized very rapidly on heating and this property, together with the rotation, refractive index, and method of preparation, indicates that it was probably impure carvopinone.³

(6) Joshei, Hall and Palkin, *Ind. Eng. Chem., Anal. Ed.*, **13**, 447 (1941).

BUREAU OF AGRICULTURAL CHEMISTRY AND ENGINEERING
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The Separation of Ytterbium from Accompanying Rare Earths by Means of its Amalgam

By HERBERT N. MCCOY AND R. PHILIP HAMMOND

The preparation of ytterbium amalgam by the electrolytic method described earlier¹ has been repeated, using a larger amount (4 g. of oxide) of the same starting material. The electrolyses were carried to the point where no further amalgam formation took place. The rare earths from amalgams and from residual electrolytes were then extracted as chlorides called I and II, respectively. Chloride I formed much the larger part of the material; chloride II amounted to about 0.1 g.

From chlorides I and II spectrograms were made using a replica grating of 15,000 lines per inch (5906 lines per cm.) and a graphite arc source. These spectrograms for the region 2500 to 4600 Å., accompanied by that of iron, were enlarged photographically to give prints with a dispersion of one Å. per mm. The enlarged prints were examined with the aid of Massachusetts Institute of Technology wave length tables² and more than 250 of the strong lines identified.

The spectrogram of chloride I was that of ytterbium containing a much smaller proportion of europium. No lines of other rare earths could be found on this spectrogram. That of chloride II showed in addition to some remaining ytterbium and europium, the presence of gadolinium, terbium, dysprosium, yttrium, erbium, thulium and lutecium. The presence of holmium was probable but could not be established with certainty because its strong lines were masked by those of other elements present.

(1) McCoy, *THIS JOURNAL*, **68**, 1622, 3432 (1941).

(2) "Massachusetts Institute of Technology Wave Length Tables," George W. Harrison, John Wiley and Sons, Inc., New York, N. Y., 1939.

This work shows that of the rare earths contained in the starting material only ytterbium and europium form amalgams by the method used. If the small proportion of europium present had first been removed, as might easily have been done by fractional crystallization as dimethyl phosphates,³ bromates⁴ or ethyl sulfates⁵ the separation of ytterbium from the rare earths that usually accompany it would have been complete.

(3) Morgan and James, *THIS JOURNAL*, **36**, 10 (1914).

(4) James and Bissel, *ibid.*, **36**, 2060 (1914).

(5) Urbain, *Compt. rend.*, **126**, 835 (1898).

PRIVATE LABORATORY
1226 WESTCHESTER PL.
LOS ANGELES, CALIF.

LABORATORY OF
LINDSAY LIGHT & CHEMICAL CO.
WEST CHICAGO, ILLINOIS
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The Purification of Thorium Chloride Octahydrate

By CHESTER B. KREMER

In connection with previous researches on thorium compounds as well as in recent work in the production of thorium dioxide catalysts, the necessity of obtaining pure thorium chloride octahydrate arose. Thorium chloride "c. p." as purchased on the market contains appreciable amounts of iron and silica. Purification by simple recrystallization is difficult because of the tenacity with which iron compounds are retained by the salt and the formation of concentrated solutions, sirupy in nature, which will not deposit crystals. However, it is possible to obtain pure $\text{ThCl}_4 \cdot 8\text{H}_2\text{O}$ by a method which in part is based upon a slight modification of the Gooch and Havens method for the determination of aluminum.

Specially prepared asbestos for the following operations is obtained by boiling a high grade variety repeatedly with concentrated hydrochloric acid until free of detectable iron and then washing thoroughly with distilled water.

Two hundred grams of the "c. p." salt is dissolved in just enough 6 *M* hydrochloric acid for complete solution, filtered through asbestos and then extracted twice with ethyl ether,¹ this operation removing practically all of the iron.² The solution is then evaporated to small volume on a hot plate. Excess silica precipitates out and is filtered off through asbestos. The filtrate is cooled to 0° and dry hydrogen chloride bubbled in until the solution is saturated with the gas. An equal volume of ethyl ether is

(1) Isopropyl ether may be employed; see Dodson, Forney and Swift, *THIS JOURNAL*, **68**, 2573 (1936).

(2) Langmuir, *ibid.*, **22**, 102 (1900).