methyl-(and α,α-diethyl)-butyrate, and 3-hydroxy-4-ethyl-4-methyl- (and 4,4-diethyl)-5-ketotetrahydrofuroic acid, but circumstances will not permit a continuation of the present study to include these substances.

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Observations on the Rare Earths. L.[1] The Separation of Erbium from Yttrium by Fractional Precipitation of the Ferrocyanides[2]

BY THERALD MOELLER AND H. E. KREMERS

Yttrium and erbium oxide mixtures which accumulate during bromate fractionations of the yttrium earths are usually further separated by basicity methods, although the very favorable solubility difference between the ferrocyanides of these elements[3,4] renders ferrocyanide fractionation more rapid. In spite of the gelatinous nature of the precipitates, comparatively pure yttrium material has been prepared by this procedure,[5,6,7,8] and Prandtl,[9] using large quantities of nitric acid to promote crystallinity, has obtained erbium oxide of high purity. The lack of specificity in Prandtl's directions suggested a further study of the problem, and the resulting modified procedure gives consistently reproducible results, is rapid, and is free from undesirable oxidation of the ferrocyanide.

Experimental

Ferrocyanide Precipitation.—Erbium–yttrium chloride solutions were prepared in 3 *N* hydrochloric acid to contain 2% rare earth oxide and 10% ammonium chloride. To these solutions at room temperature, sufficient 20% potassium ferrocyanide solution (calculated as the trihydrate) to precipitate about one-half the rare earth material was added slowly with constant stirring. Experimentally, about 15 ml. precipitated the equivalent of 1 g. of rare earth oxide from material analyzing 85–100% Er_2O_3 and 20 ml. for lower erbium concentrations. Slow precipitation began only after complete addition of the ferrocyanide. After several hours, the precipitates were removed by suction filtration on qualitative paper.

Recovery of Rare Earth Materials.—Part of the iron was removed from the precipitates by boiling the solids with 50% sodium hydroxide, diluting, and filtering. After ignition of these hydroxides, the remainder of the iron was removed by oxalate precipitation of the rare earths from chloride solutions. The ferrocyanide mother liquors gave iron-free oxides after two precipitations with oxalic acid.

Fractionation of Erbium–Yttrium Mixtures.—Erbium–yttrium mixtures showing no absorption lines for holmium or thulium were fractionated by the above procedure. Each fractionation was followed by means of spectrophotometric analyses (based upon the 489 and 653 mμ erbium absorption bands)[10] of oxides recovered from both precipitates and mother liquors. Oxides of approximately the same composition were combined and refractionated.

The fractionation of 43 g. of a mixture (ER-18) containing 57% Er_2O_3 gave 4.8 g. of 89% Er_2O_3, 2.1 g. of 86% Er_2O_3, 5.9 g. of 80% Er_2O_3, 6.8 g. of 62% Er_2O_3, 6.7 g. of 38% Er_2O_3, and 15 g. of an oxide mixture low in erbium after twenty-eight fractional precipitations (five series with five or six fractions in each).

A series of fractions (ER-34) obtained from a urea separation[11] and a previous ferrocyanide fractionation and consisting of (1) 6.9 g. of 87% Er_2O_3, (2) 5.9 g. of 80% Er_2O_3, (3) 13.8 g. of 59% Er_2O_3, (4) 20.3 g. of 27% Er_2O_3, and (5) 45 g. of 17% Er_2O_3 yielded ultimately 3.14 g. of 98.5% Er_2O_3 (two precipitations of fraction (1)); 1.3 g. of 94% Er_2O_3, 2.35 g. of 93% Er_2O_3 and 2.8 g. of 80% Er_2O_3 (four precipitations of fractions (2), (3), and (4), respectively); and 5.6 g. of 48% Er_2O_3 (three precipitations of fraction (5)). In addition, 5.0 g. of 65% Er_2O_3, 17.6 g. of 44% Er_2O_3, and 9.5 g. of 27% Er_2O_3 were recovered from the final mother liquors.

Success of the ferrocyanide procedure is dependent upon the complete removal of iron, since even traces of iron yield gelatinous blue precipitates which nullify the effects of crystallinity in the rare earth ferrocyanides. Although some unavoidable loss is inherent in the oxalate precipitations, the speed and efficiency of the method recommend it.

Acknowledgment.—The authors wish to express their appreciation to Dr. C. J. Rodden of the National Bureau of Standards for the analysis of an erbium oxide sample used in the calibration of our spectrophotometer.

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(1) For the preceding communication in this series see Kleinberg, Taebel and Audrieth, *Ind. Eng. Chem., Anal. Ed.*, **11**, 368 (1939).

(2) Presented before the Division of Physical and Inorganic Chemistry at the 106th meeting of the American Chemical Society at Pittsburgh, Pa., September 6, 1943.

(3) Rowland, *Chem. News*, **70**, 68 (1894).

(4) Prandtl and Mohr, *Z. anorg. allgem. Chem.*, **236**, 243 (1938).

(5) Bettendorf, *Ann.*, **352**, 88 (1907).

(6) Meyer and Wuorinen, *Z. anorg. Chem.*, **80**, 7 (1913).

(7) Prandtl, *Z. anorg. allgem. Chem.*, **143**, 277 (1925).

(8) Canneri, *Atti accad. Lincei*, **8**, 164 (1928).

(9) Prandtl, *Z. anorg. allgem. Chem.*, **198**, 157 (1931).

(10) Rodden, *J. Research Natl. Bur. Standards*, **28**, 265 (1942).

(11) Fogg and Hess, THIS JOURNAL, **58**, 1751 (1936).

NOYES CHEMICAL LABORATORY
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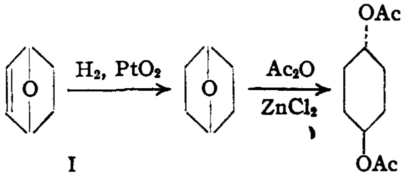
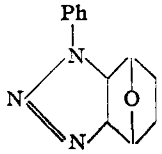
3,6-Epoxycyclohexene from Furan and Ethylene[1]

BY WALTER NUDENBERG AND LEWIS W. BUTZ

3,6-Epoxycyclohexene (I) has now been prepared by the procedure and with the apparatus previously employed in similar preparations.[2] It has been characterized as indicated below.





II

Into a mixture of 30–35 g. of furan and a few crystals of hydroquinone ethylene was introduced in two or three portions with intermittent shaking up to an initial pressure of 1100–1200 lb. per sq. in. at room temperature. The bomb was heated to 155° and kept at 150–155°, the pressure dropping and ultimately becoming constant after six-

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(2) Joshel and Butz, THIS JOURNAL, **63**, 3350 (1941).