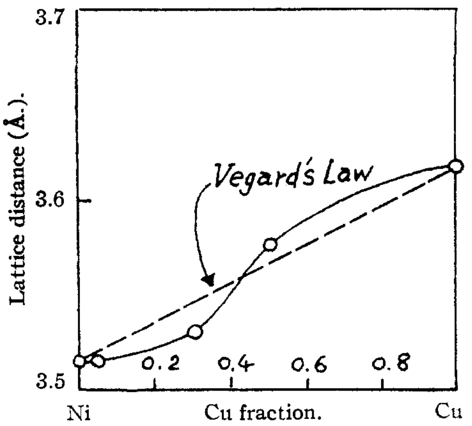

Fig. 1.

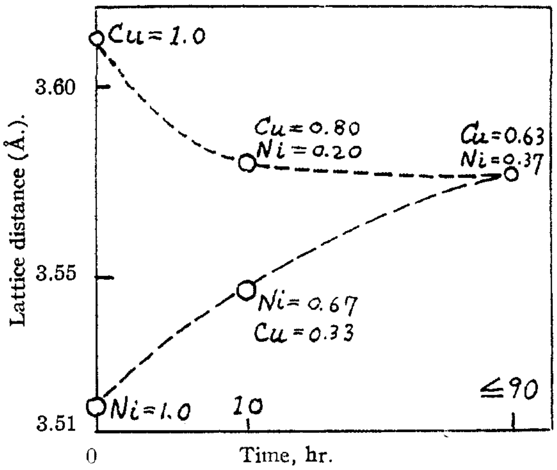

Fig. 2.

clusion that a non-equilibrium state of the alloy phase is formed on the catalyst surface, as stated by Morris and Selwood in their paper. It remains unstable on the surface to an extent depending upon the composition of the catalyst. The authors have come to the same conclusion from an adsorption study of these catalysts.[4]

Acknowledgment.—The interest of Prof. S. Kodama and the effective advice of Prof. S. Tawara in this work are gratefully acknowledged.

(4) S. Kodama and N. Kadota, unpublished results, 1949.

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The Dehydrogenation of Coumaran

BY BODO KLARMANN

Coumaran (2,3-dihydrobenzofuran) is readily available by the elegant synthesis of Bennett and Hafez[1] who effected ring closure by treating the diazotized *o*-amino derivative of *β*-phenylethyl alcohol with base.

The dehydrogenation of coumaran to coumarone (benzofuran) has not been reported, although the reverse reaction has been accomplished by Alexander[2] by reduction with sodium in absolute alcohol and by Weger[3] who employed hydrogenation.

Our experiments have shown that this dehydrogenation can be achieved satisfactorily by the use of sulfur; selenium is less satisfactory. With sulfur, and taking recovered starting material into account, a 45–50% yield was obtained. Since coumarone boils at 169°, and coumaran at 188°, separation by fractional distillation is readily achieved.

Experimental

Three hundred and sixty grams of pure coumaran, m.p. −21.5°, and 96 g. of sulfur are refluxed for 15 hours on an oil-bath (220°) during which time the sulfur dissolves and hydrogen sulfide is evolved. The reaction product was steam distilled, leaving behind a black residue. Addition of sodium chloride to the distillate caused a colorless oil to separate which after drying over calcium chloride was slowly fractionated at 8 mm. through a Widmer column (spiral 70 cm. long). The crude steam distillate (290 g.) yielded as a fore-run approximately 65 g. of pure coumarone boiling constantly at 168–169° (747 mm.), melting at −27°, and resinifying with concd. sulfuric acid in characteristic fashion. Coumarone isolated from high-boiling coal tar fractions by picric acid, purified by recrystallization and regenerated with steam gave an identical melting point.

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(1) Bennett and Hafez, *J. Chem. Soc.*, 287 (1941).

(2) Alexander, *Ber.*, **25**, 2409 (1892).

(3) Weger, *Z. angew. Chem.*, **22**, 391 (1909).

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The Preparation of Ethylidene Iodide

BY R. L. LETSINGER AND C. W. KAMMEYER

Reported methods for the preparation of ethylidene iodide either give poor yields or involve reagents such as aluminum iodide which are not readily available. It has been found that ethylidene iodide can conveniently be prepared in reasonable yields (60%) by the action of aluminum chloride on a mixture of ethylidene chloride and ethyl iodide.

Experimental

A mixture of 39.6 g. (0.4 mole) of ethylidene chloride and 187 g. (1.2 moles) of ethyl iodide was heated with 2.0 g. of aluminum chloride on a steam-bath for three hours. During this time ethyl chloride, which was formed in the reaction, bubbled out of the solution. The mixture was then poured into water, washed with a sodium bisulfite solution, and dried over magnesium sulfate. On distillation 67.3 g. (60%) of ethylidene iodide was obtained; b.p. 75–76° (25 mm.); d^{25}_{4} 2.794.

In a similar reaction where the mole ratio of ethyl iodide to ethylidene chloride was two to one, the yield of the iodide was 42%.

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The Hydrogenolysis of Sulfilimines and its Application to the Purification of Sulfides

BY M. A. MCCALL, D. S. TARBELL AND MARY ANN HAVILL

Several types of solid derivatives have been proposed for the characterization of liquid sulfides,[1]

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(1) (a) Faragher, Morrell and Comay, THIS JOURNAL, **51**, 2774 (1929); (b) Ipatieff, Pines and Friedman, *ibid.*, **60**, 2731 (1938); (c) Ipatieff and Friedman, *ibid.*, **61**, 684 (1939); (d) Mann and Pope, *J. Chem. Soc.*, **121**, 1052 (1922). Other types of derivatives, including sulfonium salts, are discussed by Connor in Gilman, "Organic Chemistry," John Wiley and Sons, Inc., New York, N. Y., 1943, pp. 858–861, 867–868.