acetone and ethyl acetate to give 0.2 g. of solid melting at 205–208° after several recrystallizations from aqueous methanol. Fischer[23] gives 207° as the melting point of 2-amino-5-nitrobenzenesulfonamide. The yield was 4.2%, and 0.3 g. of unreacted sulfonamide was also recovered.

Cyclohexylamine and m-dinitrobenzene. Cyclohexylamine (3.0 g., 0.030 mole) and *m*-dinitrobenzene (5.0 g., 0.030 mole) were subjected to the same conditions, and the resulting aqueous concentrate was extracted with benzene. Evaporation of the extract to a tar and extraction with dilute sulfuric acid gave 0.02 g. (0.5%) of *m*-nitroaniline after neutralization; m.p. 104–109°. A mixed melting point with an authentic sample gave no depression. The acid-extracted tar was recrystallized from aqueous ethanol, and 4.7 g. of unreacted *m*-dinitrobenzene, m.p. 88–90°, was recovered.

SCHOOL OF PHARMACY
UNIVERSITY OF WISCONSIN
MADISON, WIS.

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Vinyl Derivatives of the Metals. III. Vinylmercuric Halides

DIETMAR SEYFERTH

Received November 19, 1956

Recent studies of vinyltin compounds[1,2] have shown that a vinyl group connected to a tin atom is quite labile, and its position in the Kharasch-type cleavage series for organotin compounds was shown to be:

$$C_6H_5 > CH_2{=}CH > n\text{-Alkyl}$$

Manulkin[3] reported the cleavage of tetraalkyltin compounds by mercuric halide in alcoholic solution to give a mixture of trialkyltin halide and dialkyltin dihalide with the corresponding alkylmercuric halide.

We have utilized the observed lability of the vinyl-tin bond to prepare the vinylmercuric halides, reported here for the first time, by preferential cleavage of the vinyl group from alkylvinyltin compounds with mercuric chloride, bromide, or iodide:

$$R_3SnCH{=}CH_2 + HgX_2 \xrightarrow{Et_2O} R_3SnX + CH_2{=}CH\cdot HgX$$

The method of Manulkin was modified as described in the experimental part of this study. Our Soxhlet method of carrying out this reaction has the advantage that this usually quite vigorous reaction is moderated, and that only small quantities of solvent are required. This method is particularly useful when the extremely insoluble mercuric iodide is used.

The three vinylmercuric halides, their melting points, and their analytical data are listed in Table I. Work utilizing these compounds in other organometallic syntheses is in progress.

TABLE I

VINYLMERCURIC HALIDES, $CH_2{=}CH\cdot HgX$

X	M.p., °C.	Analyses[a] Calcd., %	Analyses[a] Found, %
Cl	185–186	Hg: 76.24	75.92
Br	168–170	Hg: 65.22	65.12
I	150–151.5	C: 6.77	6.96
		H: 0.85	0.91
		I: 35.80	36.04

[a] Analyses were performed by the Schwarzkopf Microanalytical Laboratory, Woodside, N. Y.

EXPERIMENTAL

The preparation of vinylmercuric bromide is described as an example of the technique used.

A solution of 25.7 g. (0.1 mole) of *n*-butyltrivinyltin in 200 ml. of ether was prepared in the still pot of a Soxhlet extraction apparatus. A Soxhlet thimble was filled with 36.0 g. (0.1 mole) of mercuric bromide and placed in the apparatus. The cycling of ether was continued for about 20 hr. until all of the mercuric bromide had been brought to reaction. During the course of the reaction white platelets of vinylmercuric bromide crystallized in the still pot.

The solution was cooled and filtered, concentrated to about one third of its original volume and filtered again to give 20.0 g. of vinylmercuric bromide, a yield of 65%. Recrystallization of a small sample from ether gave analytically pure compound. Fractional distillation of the liquid residue gave 20.5 g. (66.1% yield) of the new compound, *n-butyldivinyltin bromide*, b.p. 72° at 0.75 mm. to 70° at 0.6 mm., d_4^{25} 1.529, n_D^{25} 1.5221.

Anal. Calcd. for $C_8H_{15}BrSn$: C, 31.01; H, 4.88; MR_D, 61.85. Found: C, 31.11; H, 5.03; MR_D, 61.80.

Vinylmercuric chloride was prepared in the same manner using di-*n*-butyldivinyltin and mercuric chloride. Vinylmercuric iodide was obtained from the reaction of *n*-butyltrivinyltin with red mercuric iodide. The reaction proceeded exceedingly slowly due to the insolubility of mercuric iodide. After the ether had been cycled for 2 days only 3 g. of vinylmercuric iodide had crystallized.

Under identical conditions no reaction was observed between tetravinylgermane, $(CH_2{=}CH)_4Ge$, and mercuric chloride.

The vinylmercuric halides are stable with respect to atmospheric oxygen and moisture; therefore no special precautions are required. Because of the known high toxicity of RHg*X* compounds,[4] all operations were carried out in a good hood.

Acknowledgment. The author is indebted to the United States Office of Naval Research for its support of this research[5] and to Professor E. G. Rochow for his interest in this work.

DEPARTMENT OF CHEMISTRY
HARVARD UNIVERSITY
CAMBRIDGE 38, MASS.

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