

ate and the *o*-hydroxybenzoate are quite soluble in water. The benzoate and the *o*-bromo, *o*-chloro and *o*-hydroxy salts are very soluble in water. All of the salts are fairly soluble in chloroform and in ethyl alcohol, and slightly soluble in ether.

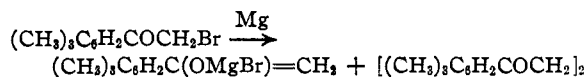
CHEMISTRY DEPARTMENT
UNIVERSITY OF COLORADO
BOULDER, COLORADO

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Some New Amidine Hydrochlorides

BY JOHN B. EKELEY, DIRK V. TIESZEN AND ANTHONY RONZIO

We have prepared a number, as given below, of required amidine hydrochlorides not yet noted in the literature, by the usual method from the corresponding cyanides. They were recrystallized from water and dried in a vacuum over concentrated sulfuric acid. They are colorless crystals and exhibit the reactions of amidines. The



Approximately 60% of the theoretical amount of magnesium required to give the enolate was consumed in the reaction. It is believed that the organic magnesium compound is the enolate because of its solubility behavior in ether and ether-benzene solutions, which is similar to that of the bromomagnesium enolate¹ formed in the reaction of ethylmagnesium bromide with acetomesitylene.

Refluxing a mixture of 4.82 g. of bromoacetylmesitylene, 0.54 g. of magnesium and 40 cc. of ether for one-half hour caused the appearance of a white, milky precipitate, which dissolved after the addition of 60 cc. of hot benzene. The mixture was heated for one hour longer, and filtered from unreacted magnesium (0.2 g.). Addition of water caused a precipitate to appear which dissolved when hydrochloric acid was added. The ether-benzene solution was dried and distilled. On vacuum distillation of the remaining oil there was obtained 1.45 g. of acetomesitylene and 0.88 g. of α -bromoacetylmesitylene. The residue, after several

TABLE I
R AMIDINE HYDROCHLORIDES

R	M. p., °C.	Formula	Analyses					
			Calcd.	N, %	Found	Calcd.	Cl, %	Found
Isocapro-	113.5	C ₈ H ₁₄ N ₂ ·HCl	23.50	23.77	23.53	18.59	18.70	18.75
Phenoxybutyro-	150	C ₁₀ H ₁₄ ON ₂ ·HCl	16.52	16.68	16.52	13.06	13.12	13.05
<i>m</i> -Tolenyl-	185.5	C ₈ H ₁₀ N ₂ ·HCl	20.78	20.75	20.95	16.41	16.24	16.47
<i>p</i> -Chlorobenz-	241-242	C ₇ H ₆ ClN ₂ ·HCl	18.57	18.73	18.74	14.91	14.91	14.80

melting points were made using a short-stem Anschütz thermometer.

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The Reaction of Magnesium with α -Halo Ketones

BY C. HAROLD FISHER

In the preparation of organic magnesium compounds by the interaction of magnesium and α -halo ketones it should be possible to avoid complications arising from reactions of the carbonyl group by working with hindered halo ketones. An exploratory examination, which cannot be continued, has shown that this is true in the case of α -bromoacetylmesitylene. After decomposition of the magnesium derivative with dilute acid the products were acetomesitylene (45%), 1,2-di-(2,4,6-trimethylbenzoyl)-ethane (10%), and starting material (18%).

crystallizations from alcohol, melted at 136-137.5°. When mixed with a sample of di-(2,4,6-trimethylbenzoyl)-ethane prepared by Conant and Lutz² the melting point was 137-138.5°. The acetomesitylene was identified by conversion into 3,5-dinitroacetylmesitylene³ (m. p. 138-139.5°), which was compared with an authentic specimen.

Under similar conditions magnesium reacted also with α -bromo- β -phenylbenzalacetophenone. However, the reaction mixture gave a negative test⁴ for the Grignard reagent; and treatment with dilute acid gave a sticky yellow solid which was not identified. After partial purification had been accomplished by washing with hot alcohol the product was halogen-free and failed to melt under 250°. Efforts to effect crystallization from benzene and ligroin were unsuccessful, and its study was abandoned.

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- (1) Kohler and Tishler, *THIS JOURNAL*, **57**, 221 (1935).
- (2) Conant and Lutz, *ibid.*, **45**, 1306 (1923).
- (3) Walker and Fuson, *ibid.*, **52**, 3273 (1930).
- (4) Gilman and Schulze, *ibid.*, **47**, 2002 (1925).