trode at temperatures as high as 40° for even the most precise work.

EASTMAN KODAK COMPANY
ROCHESTER, N. Y.
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The Preparation of *m*-Bromobenzaldehyde

BY FLOYD T. TYSON

For work in progress in this Laboratory it was necessary to prepare *m*-bromobenzalaminoacetal by the interaction of *m*-bromobenzaldehyde with aminoacetal. The *m*-bromobenzaldehyde was prepared by the reduction of *m*-nitrobenzaldehyde by stannous chloride and hydrochloric acid, followed by diazotization and conversion to bromobenzaldehyde by the Sandmeyer method as originally described by Einhorn and Gernsheim[1] and modified by Buck and Ide.[2] In these procedures, no attempt is made to isolate the intermediate *m*-aminobenzaldehyde from admixture with stannic chloride and hydrochloric acid.

Analysis of the *m*-bromobenzalaminoacetal yielded uniformly low results for halogen calculated as bromine and very high results for carbon and hydrogen. These analytical values were in accord with the assumption that much of the chloro compound was present with the bromo compound.

The *m*-bromobenzaldehyde used for the preparation of the impure *m*-bromobenzalaminoacetal was analyzed for halogen and the halogen calculated as bromine. The analytical values obtained were in agreement with the theoretical values for bromobenzaldehyde. However, analyses of the *m*-bromobenzaldehyde preparations for both chlorine and bromine proved the presence of both chlorine and bromine. The amounts of chlorine indicated a percentage of chlorobenzaldehyde ranging from 20 to 72 depending upon details of procedure.

Since *m*-nitrobenzaldehyde should be a convenient source for the preparation of pure *m*-bromobenzaldehyde, a procedure has been devised which will be published elsewhere, in which the presence of chlorides in the reaction mixture was avoided.

Experimental

The following table summarizes data obtained in the analysis of products resulting from the attempted preparation of pure *m*-bromobenzaldehyde from *m*-nitrobenzaldehyde as described in the literature. The preparations analyzed were fractionated; b. p. 92–97° (6 mm.).

TABLE I

Prepn.	Analyses, % Cl	Analyses, % Br	Calcd. % chlorobenzaldehyde present
1	18.17	11.86[3]	72.0
	18.31	11.57[4]	
2	5.11	34.50[3]	20.3
3	6.78	31.58[3]	26.9

Preparation No. 1 was made as described by Buck and Ide.[2] Preparation No. 2 was made as No. 1 except that the hydrobromic acid was added to the cuprous bromide mixture before mixing with the diazotized solution. Preparation No. 3 was made as described by Einhorn and Gernsheim.[1]

The analytical results reported in the following table were calculated on the erroneous assumption that the mixtures of silver chloride and bromide actually obtained in the analyses of preparations No. 1, 2 and 3 (Table I) were pure silver bromide. The satisfactory agreement with the values for *m*-bromobenzaldehyde may explain the fact that the preparations as recorded in the literature[1,2] were reported as essentially pure.

TABLE II

Prepn.	% Br found as assumed	% Br calcd. for *m*-bromobenzaldehyde
1	43.12	43.19
2	43.18	
3	43.25	

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[1] Einhorn and Gernsheim, *Ann.*, **284**, 141 (1894).

[2] Buck and Ide, *Org. Syntheses*, **13**, 30 (1933).

[3] Analyses made by the method of displacement of bromine from weighed silver chloride and bromide by chlorine as described in "Quantitative Analysis of Inorganic Materials," by N. Hackney, J. B. Lippincott, Philadelphia.

[4] Analysis made by the method of Baubigny [*Compt. rend.*, **136**, 1197 (1903)] as described by Hackney.[4]

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The Optical Rotation of a Grignard Reagent

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Frequent use is made of the Grignard reaction in preparing optically active compounds from optically active halogen compounds, but not to our knowledge has the rotation of an optically active Grignard reagent been measured previously. Porter[1] observed a very small rotation in an ethereal solution of methyl-*n*-hexylmagnesium

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[1] Porter, THIS JOURNAL, **57**, 1436 (1935).