Some Derivatives of 3-Bromobenzaldehyde

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IN CONNECTION with the work of this laboratory it was necessary to prepare a large series of derivatives of 3-bromobenzaldehyde. The new compounds which were prepared are reported here. They are included in Table I.

EXPERIMENTAL

Condensation with Ethyl Cyanoacetate. Equimolar amounts of the 3-bromobenzaldehyde and ethyl cyanoacetate in dioxane were teated with piperidine as previously described (*1*) to give the product listed in Table I.

Table I. Derivatives of 3-Bromobenzaldehyde

RH$_2$	Yield %	M.P., °C.	Formula	Analysis Calcd. C	H	N	Found C	H	N
Cyclopropanecarboxyhydrazide	73	144-146	$C_{11}H_{11}N_2BrO$	49.46	4.15	10.49	49.79	4.03	10.59
p-Bromophenylhydrazine	86	133-134	$C_{13}H_{10}N_2Br_2$	44.10	2.85	7.91	44.17	2.72	8.17
1-Amine-3-nitroguanidine	56	211-212	$C_8H_8N_5BrO_2$	33.58	2.82	24.48	33.88	2.93	24.72
Thiosemicarbazide	92	207-208	$C_8H_8N_3BrS$	37.22	3.12	16.28	36.80	3.65	16.05
p-Fluoroaniline	93	41-42	$C_{13}H_9NBrF$	56.14	3.26	5.04	56.17	3.10	5.11
Ethyl cyanoacetate	64	98-99	$C_{12}H_{10}NBrO_2$	51.43	3.60	5.00	51.81	3.67	4.76

Reaction with Thiosemicarbazide. A hot solution of 0.91 gram (0.01 mole) of thiosemicarbazide in 30 ml. of water and 2 ml. of glacial acetic acid was added to 1.85 grams (0.01 mole) of 3-bromobenzaldehyde in 25 ml. of ethanol and the mixture was heated for 15 minutes. Cooling, filtration, and recrystallization from ethanol gave the thiosemicarbazone (Table I).

Other Derivatives. Mixtures of 0.01 mole of 3-bromobenzaldehyde and 0.01 mole of the appropriate reagent were heated in ethanol for 20 min., cooled, filtered, and recrystallized from ethanol to give the materials listed in Table I.

LITERATURE CITED

(1) Popp, F.D., *J. Org. Chem.* **25**, 646 (1960).

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