aldehyde. Benzene was obtained from both the iso- and terephthalic acids. Anisic acid gave anisole; *p*-toluic acid yielded toluene and the zinc salt of *p*-toluic acid; and diphenylacetic acid decomposed into diphenylmethane.

**Esters Decomposing without Evolution of Carbon Dioxide.**—Benzyl benzoate and benzyl butyrate decomposed into their respective acids, benzoic and butyric. The benzyl groups polymerized, forming a dark colored and tarry residue. Dimethyl phthalate gave phthalic anhydride, and *n*-amyl phthalate gave phthalic anhydride, amylene and its polymers. Phenyl acetate yielded phenol; and phenyl salicylate gave phenol at a temperature of 177° in presence of the catalyst, although a temperature of 280° is required to decompose it without a catalyst. Phenylacetic acid yielded acetic acid; *m*- and *p*-nitrobenzoic acid reacted with the catalyst to form the zinc salt of the acid with evolution of hydrogen chloride. The *p*-nitrobenzoic acid mixture blew up after fifteen minutes of heating, leaving a deposit of soot over the entire apparatus.

A new type of decomposition has been observed with the elimination of carbon monoxide instead of carbon dioxide from halogenated aliphatic acids. Monochloroacetic acid decomposed into carbon monoxide, formaldehyde and hydrogen chloride; and the trichloroacetic acid decomposed into carbon monoxide, carbonyl chloride and hydrogen chloride.

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## Reduction of Nitroguanidine. III. Synthesis of Aminoguanidine[1]

BY G. B. L. SMITH AND EDWARD ANZELMI

Aminoguanidine has been prepared by the reduction of nitroguanidine with zinc in a solution of acetic acid,[2] by the interaction of hydrazine hydrochloride and cyanamide,[3] and by the electrolytic reduction of nitroguanidine.[4] We have prepared aminoguanidine in high purity and excellent yield by the hydrazinolysis of methyl isothiourea sulfate. To 139 g. of methyl isothiourea sulfate in 200 ml. of water at 10° is added 119 ml. of a 42% solution of hydrazine hydrate[5] diluted with an equal volume of water. The methyl mercaptan liberated can be absorbed in a solution of sodium hydroxide.[6] Finally the aminoguanidine sulfate is precipitated by addition of an equal volume of 95% ethanol to the solution concentrated to 200 ml. A second crop of crystals separates on concentration of the filtered solution. The water of crystallization (1 molecule) may be removed by drying *in vacuo* or at 105°; yield 90%; m. p. 206° with decomposition (Thiele, 207–208°). Aminoguanidine sulfate was identified through conversion to the picrate and bicarbonate, by liberation in alkaline solution of one-half of the nitrogen as ammonia and by hydrolysis to hydrazine.[7] *Anal.* calcd. for $(CH_6N_4)_2H_2SO_4$: $SO_4$, 39.83; $NH_3$, 27.67; $N_2H_4$, 26.02. Found: $SO_4$, 39.3, 39.7; $NH_3$, 27.2, 27.5, 27.7; $N_2H_4$, 26.03, 25.93. The fact that aminoguanidine bicarbonate has the composition $CN_4H_6H_2CO_3$ has been established by analysis. A solution of aminoguanidine is a strong base $K_B$ estimated as $1.1 \times 10^{-3}$. Further work on aminoguanidine and alkylaminoguanidines is in progress.

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[1] For more full details see B.S. thesis by Edward Anzelmi, Polytechnic Institute of Brooklyn, 1933. Contribution No. 27 from the Department of Chemistry of the Polytechnic Institute of Brooklyn.

[2] Thiele, *Ann.*, **270**, 1 (1892).

[3] Pellizzarri and Cuneo, *Gazz. chim. ital.*, [2] **21**, 405 (1901).

[4] Boehringer, German Patent 167,637. We have as yet been unable to confirm this observation.

[5] From the Eastman Kodak Company.

[6] Phillips and Clarke, THIS JOURNAL, **45**, 1755 (1923).

[7] Jamieson, *Am. J. Sci.*, [4] **33**, 353 (1912).