A solution of this oxime (451 g.) in a mixture of 1080 cc. of glacial acetic acid and 455 cc. of acetic anhydride was cooled in ice and saturated with gaseous hydrogen chloride. The solution was then allowed to stand at 40° for sixty to seventy hours, cooled in ice and filtered (sintered glass!) with suction, using a rubber dam to squeeze out as much of the liquid as possible. The filter cake was immediately mixed with 820 cc. of concentrated hydrochloric acid and refluxed until a homogeneous solution was obtained (two hours). The 3,4-dimethylaniline hydrochloride could be obtained merely by crystallization from the cooled reaction mixture. However, to obtain the free base, the reaction mixture was made alkaline with aqueous sodium hydroxide, extracted with ether, dried and distilled. In this manner there was obtained 238 g. (25% yield from fenchone) of pure 3,4-dimethylaniline, b. p. 132–134° (45 mm.); m. p. 50–51°

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ABBOTT LABORATORIES
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NEW COMPOUNDS

p-Di-(*β*-chloro-*t*-butyl)-benzene

Although the preparation of (*β*-chloro-*t*-butyl)-benzene by the reaction of benzene with methallyl chloride in the presence of sulfuric acid[1] or hydrogen fluoride[2] has been described, the formation of a crystalline di-substituted compound analogous to *p*-di-*t*-butylbenzene has not hitherto been reported.

A solution of 300 g. (3.3 moles) of methallyl chloride in 300 g. (3.8 moles) of benzene was added during two hours to a well-stirred mixture of 500 g. (6.4 moles) of benzene and 400 g. of 96% sulfuric acid at 0–5°. After an additional one-half hour of stirring, the upper layer was separated from the 433 g. of lower layer, washed with water, dried, and distilled under reduced pressure. Neophyl chloride (*i. e.*, (*β*-chloro-*t*-butyl)-benzene) boiling at 90–91° at 9 mm. mercury pressure, $n^{20}D$ 1.5250, was obtained in 67% of the theoretical yield. The higher-boiling residue (55 g.) was a dark brown oil which became partly crystalline (long white needles) on standing. The mixture of oil and crystals was cooled in an ice–salt-bath and then filtered through a sintered glass plate. Recrystallization of the solid from alcohol yielded 11 g. (2.6% of the theoretical) of *p*-di-(*β*-chloro-*t*-butyl)-benzene, thick needles, m. p. 54.5–55°.

Anal. Calcd. for $C_{14}H_{20}Cl_2$: Cl, 27.36. Found: Cl, 27.36. No attempt was made to recover more of the compound from the filtrates.

The orientation of the chloroalkyl groups was proved by oxidizing 1 g. of the compound by refluxing it for twenty hours with a solution of 5 g. of sodium dichromate and 5 cc. of concentrated sulfuric acid in 100 cc. of water. Terephthalic acid was obtained; its identity was confirmed by means of its dimethyl ester, m. p. 140°.

Calcott, Tinker and Weinmayr,[2] using hydrogen fluoride as catalyst, obtained a 66% yield of neophyl chloride and a 20% yield of a liquid product, boiling at 140° at 4 mm., which they considered to be "di-(1'-chloro)-*t*-butylbenzene." Their product may have been a different isomer. More probably it was impure owing to loss of hydrogen chloride during distillation; the product contained only 25.6% chlorine.

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(1) F. C. Whitmore, C. A. Weisberger and A. C. Shabica, Jr., THIS JOURNAL, **65**, 1469 (1943).

(2) W. S. Calcott, J. M. Tinker and V. Weinmayr, *ibid.*, **61**, 1010 (1939).

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RECEIVED JULY 20, 1945

t-Butylphthalimide[1]

t-Butylurea, m. p., 175–180°, was prepared in 86% yield by the method of Harvey and Caplan.[2] An intimate mixture of *t*-butylurea (35 g., 0.3 mole) and phthalic anhydride (100 g., 0.67 mole) is placed in a large (1000 cc.) container which is then plunged into a bath at 200°. After the initial vigorous effervescence subsides (ten minutes) the temperature of the bath is raised to 240° and maintained there for five minutes. Rapid heating is essential; the reaction is usually over in fifteen minutes. The cooled product is partially dissolved in alcohol (100 cc.) and the mixture is made alkaline (litmus) by addition of aqueous sodium carbonate. The mixture is diluted with water to a volume of 1 liter, the solid is transferred to a Büchner funnel, pressed as dry as possible, and then warmed with petroleum ether (500 cc., b. p. 60–68°). The hot mixture is filtered and, after the filtrate is separated from the water layer (if any appears), it is cooled to room temperature. Any insoluble material is removed, and the clear filtrate is concentrated by distillation to about one-third of its original volume and set aside in a refrigerator. The crystalline material is filtered with suction and pressed as dry as possible. It weighs 43.5 g. and melts at 59–60° By concentration of the filtrate, a further 3 g. may be obtained; this also melts at 59–60°. The total yield, 46.5 g., is 76%.

Anal. Calcd. for $C_{12}H_{13}O_2N$: C, 70.93; H, 6.40 Found: C, 70.95; H, 6.26.

This procedure is based upon the general procedure of Manske for conversion of alkyl ureas to phthalimides.[3] When an alcoholic solution of the phthalimide is hydrolyzed by action of hydrazine hydrate[4] and then acidified, *t*-butylamine hydrochloride can be obtained in 89% yield. The combination of the three steps from *t*-butyl alcohol to *t*-butylamine *via* the urea and phthalimide constitutes the best method for preparation of *t*-butylamine.

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(1) The work described herein was done under contract with the Office of Scientific Research and Development, which assumes no responsibility for the accuracy of the statements herein.

(2) Harvey and Caplan, U. S. Patent 2,247,495, July 1, 1941; *Chem. Abs.*, **35**, 6267 (1941).

(3) Manske, THIS JOURNAL, **51**, 1202 (1929); see also Tingle and Brenton, *ibid.*, **32**, 116 (1910).

(4) Ing and Manske, *J. Chem. Soc.*, 2348 (1926).

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p-Aminobenzanilide and Derivatives

In the course of other investigations a considerable amount of *p*-aminobenzanilide was needed. Its preparation was previously described by several authors.[1,2,3] Rivier and Kunz[3] reported a yield of 70% of *p*-aminobenzanilide by reduction of *p*-nitrobenzanilide with stannous chloride and hydrochloric acid. Their method has been repeated, resulting in a yield of 90%. Other reducing agents, such as tin and hydrochloric acid, zinc and acetic acid, zinc and hydrochloric acid, or Raney nickel in alcohol,

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(1) *Friedlander*, **4**, 752 (1895).

(2) H. Kupferberg, *J. prakt. Chem.*, [2] **16**, 444 (1877).

(3) H. Rivier and S. Kunz, *Helv. Chim. Acta*, **15**, 376 (1932).