was washed with dilute sodium carbonate solution, then with dilute sodium sulfate solution, dried over sodium sulfate, and fractionated.

Method B.—Monoisobutyrate, monostearate, diacetate, diisobutyrate, dicaproate, distearate, and dibenzoate: The reaction vessel was a three-necked flask fitted with a thermometer, dropping funnel, and mercury-sealed stirrer. For monoester formation, 0.50 mole of the acid chloride was added dropwise, over a period of one hour at a reaction temperature of 25–35°, to a stirred, cooled mixture of 0.50 mole of 2-methyl-2,4-pentanediol (10% excess for the monoisobutyrate, and 100% excess for the monostearate) and 0.50 mole of pyridine. For diester formation the quantities of reactants, in the same procedure, were 0.50 mole of 2-methyl-2,4-pentanediol and 1.05–1.10 moles of both the acid chloride and pyridine. After the acid chloride addition, the mixture was allowed to react further for either eighteen hours at room-temperature or one hour at 60–70°. The final reaction mixture was treated with 100 ml. of water, and the two layers were separated. The ester layer was washed with dilute sodium carbonate or sodium hydroxide solution, then with dilute sodium sulfate solution, dried over sodium sulfate, and fractionated.

With the mono- and distearate, ether and some alcohol were added to the ester layer to facilitate the difficult phase-separation after washing. The ether was evaporated from the dried solution, and the ester was heated up to 110° at 2 mm. before analysis. In attempted distillations of the mono- and distearate, a portion was heated in a Claisen flask to 200° at 2 mm. Decomposition was evident with both esters; it then was found that the acid no. of the samples had risen to about 54.

Unsuccessful Esterification Reactions with 2-Methyl-2,4-pentanediol.—In several unsuccessful attempts at esterification, evidence was obtained of preferential dehydration of the 2-methyl-2,4-pentanediol, which presumably was catalyzed by the acidic nature of the reaction mixtures. Thus a mixture of 0.75 mole of the diol and 0.50 mole of stearic acid, which was heated for four hours at an increasing temperature of 180–245° under a fractionating column fitted for azeotropic removal of water with benzene, gave 23 ml. of water layer instead of the 9 ml. calculated for esterification. A mixture of 0.50 mole of the diol and 1.00 mole of benzoyl chloride was heated for four hours at an increasing temperature of 30–90°. The crystalline product which separated on cooling the reaction mixture was identified as benzoic acid; the crude acid amounted to an 80% yield. In attempted esterifications with anhydrides of dibasic acids, mixtures of 0.25 mole of the diol and 0.50 mole of either phthalic, succinic, or maleic anhydride were heated at 100–120° for about an hour. The respective dibasic acids were isolated from the reaction mixtures in yields of 30–40%; the other products were not examined.

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The Preparation of *p*-Amino-tetraphenylmethane

By Benjamin Witten and E. Emmet Reid

A systematic investigation of the preparation of *p*-aminotetraphenylmethane by the method of Ullmann and Munzhuber[1] has shown that the best method is to reflux for three hours triphenylcarbinol in two times its weight of glacial acetic acid with two equivalents of aniline hydrochloride. The yield is 70–80%, about the same as was obtained by Ullmann and Munzhuber with four equivalents of aniline hydrochloride and five hours of refluxing. In one hour the yield is only 48%. The aniline hydrochloride must be thoroughly dry. If triphenylmethyl chloride is substituted for the carbinol, the time of reflux can be cut to one hour without detriment to the yield. The product obtained in any case is the hydrochloride, from which the base is liberated by boiling with water containing alkali.

It has been reported[2] that triphenylmethyl chloride and aniline, refluxed in a mixture of alcohol and benzene, give N-triphenylmethylaniline, $(C_6H_5)_3CNHC_6H_5$. We have confirmed this, but have found, however, that, if acetic acid is used as the reaction medium, para substitution takes place and the product is *p*-triphenylmethylacetanilide, $(C_6H_5)_3CC_6H_4NHCOCH_3$. This has been compared with compound obtained from action of acetyl chloride on 4-aminotetraphenylmethane and found to be identical.

Thus three different compounds can be obtained from triphenylmethyl chloride and aniline, depending on the solvent and the presence or absence of hydrochloric acid.

Experimental

***p*-Triphenylmethyl Acetanilide.**—A solution of 2 g. of *p*-aminotetraphenylmethane and 10 g. of acetyl chloride in 100 ml. of benzene was refluxed for one hour. The benzene was boiled off at reduced pressure and the residue recrystallized from benzene. The yield was 2.5 g. (93%) of colorless needles of *p*-triphenylmethylacetanilide melting at 229°.

A mixture of 14 g. of triphenylmethyl chloride, 10 g. of aniline and 50 ml. of acetic acid was refluxed for three hours and poured into 200 ml. of water. The precipitate was filtered off, washed with water, dried at 110°, and recrystallized from toluene. The yield was 16 g. (84%) of colorless crystals melting at 229°.

Anal. Calcd. for $C_{27}H_{27}NO$: C, 85.9; H, 6.1; N, 3.72. Found: C, 86.2; H, 6.1; N, 3.69.

A mixture of this preparation and the former melted at the same temperature.

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[1] Ullmann and Munzhuber, *Ber.*, **36**, 407 (1903).

[2] Elbs, *Ber.*, **17**, 704 (1884); Hemilian, *ibid.*, **17**, 746 (1884); Gomberg, *ibid.*, **35**, 1829 (1902); Van Alphen, *Rec. trav. chim.*, **46**, 500 (1927).

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NEW COMPOUNDS

Esters of *p*-Cyclohexylphenol[1]

***p*-Cyclohexylphenyl propionate** (m. p. 48–50°) was prepared in 53% yield as follows: 20 g. of *p*-cyclohexylphenol, 44 ml. of propionic anhydride and 2 ml. of anhydrous pyridine were refluxed for sixty-five minutes. The volatile portion was distilled off, and the residue was

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[1] Abstracted from the thesis presented by Albert J. Byer to DePaul University in partial fulfillment of the requirements for the M.S. degree in 1943.