distilled to dryness under a vacuum. The residue was dissolved in 1500 cc. of water and treated with 50 g. of Norite at the boiling point. The filtrate was carefully acidified with glacial acetic acid. The 2-mercaptopyridine formed in bright yellow crystals which were collected, after chilling to 5°, and washed with 2 liters of ice water. The dry product weighed about 400 g. and melted at 121–124°. A further 67–77 g. (m. p. 125–128°) was obtained by extraction of the filtrate and washings by chloroform, removing the latter by distillation and crystallizing the residue from benzene. The total yield was 461–483 g. (83–87%).

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NEW COMPOUNDS

Derivatives of Mesitylene

Methyl 3,5-Dinitromesitoate.—Two grams of methyl mesitoate was added dropwise to an ice-cold mixture of 25 ml. of concentrated sulfuric acid and 25 ml. of fuming nitric acid at such a rate that the temperature did not rise above 10°. It was necessary to cool and stir continuously. The solution was kept cold for fifteen minutes and poured on cracked ice. The dinitro ester was recrystallized from methanol: yield 2.3 g. (m. p. 136–138.5°). The pure compound melted at 138.5–139.5°.

Anal. Calcd. for $C_{11}H_{12}O_6N_2$: N, 10.4. Found: N, 10.6.

α,α-Diphenylacetomesitylene. A. From the Acetone Derivative of Mesitylglycolic Acid.—A solution of 50 g. of the acetone derivative of mesitylglycolic acid[1] in 100 ml. of ether was added slowly to a solution of phenylmagnesium bromide containing approximately four times the theoretical amount of active reagent. The mixture was heated overnight under reflux and decomposed in the usual way. The product, presumably 1,1-diphenyl-2-mesitylethylene glycol, was an oil boiling at 190° (4 mm.). A 5-g. portion of the oil was heated under reflux for one hour with a mixture of 65 ml. of glacial acetic acid and 15 ml. of hydrochloric acid. When the mixture was poured on ice the α,α-diphenylacetomesitylene separated as a solid. It was recrystallized from ethanol; m. p. 152–153°; yield 4.5 g.

Anal. Calcd. for $C_{23}H_{22}O$: C, 87.86; H, 7.05. Found: C, 87.70; H, 7.04.

B. From Diphenylacetyl Chloride.—Twenty-seven grams of anhydrous aluminum chloride was added portionwise over a period of twenty minutes to a mixture of 29.5 g. of diphenylacetyl chloride, 15.1 g. of mesitylene and 200 ml. of carbon disulfide. The mixture was kept in an ice-bath and was stirred continuously throughout the period of addition and for seventy minutes afterward. By pouring the reaction mixture on ice the α,α-diphenylacetomesitylene was precipitated as a solid melting at 148–150°; yield 83%. It was purified by repeated recrystallization from ethanol; m. p. 152–153°.

Anal. Calcd. for $C_{23}H_{22}O$: C, 87.86; H, 7.05. Found: C, 87.50; H, 7.20.

Mesityl *p*-Phenylphenyl Ketone.—A solution of 9 g. of *p*-phenylbenzoyl chloride in 75 ml. of carbon disulfide was added slowly to a mixture of 5.5 g. of mesitylene, 6.5 g. of anhydrous aluminum chloride and 20 ml. of carbon disulfide. After the addition was completed the reaction mixture was stirred at room temperature for two hours. A portion of the solvent was evaporated and the residual mixture was poured into a mixture of ice and concentrated hydrochloric acid. The ketone, after one recrystallization from ethanol, melted at 108–110°; yield 80% of the theoretical. The melting point of the pure compound was 111–112°.

Anal. Calcd. for $C_{22}H_{20}O$: C, 87.96; H, 6.71. Found: C, 87.75; H, 6.77.

3,5-Dinitromesityl 4-Methyl-3-nitrophenyl Ketone.—Two grams of *p*-toluylmesitylene was dissolved in 8 ml. of concentrated nitric acid, and the mixture heated at 45° for ten minutes. The trinitro derivative, precipitated by pouring the reaction mixture on cracked ice, was recrystallized from 95% ethanol; m. p. 168–169°.

Anal. Calcd. for $C_{17}H_{15}N_3O_7$: C, 54.71; H, 4.02; N, 11.26. Found: C, 54.33; H, 4.09; N, 11.14.

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RECEIVED NOVEMBER 2, 1945

Di-*o*-tolylglycolic Acid

A solution of the binary mixture, Mg–MgI_2,[1] was made from 6 g. of magnesium, 30.6 g. of iodine, 120 ml. of ether and 240 ml. of benzene. To this reagent was added a solution of 20 g. of *o*-toluyl chloride in 20 ml. of ether. The mixture was heated under reflux, with stirring, for eighteen hours and treated with an ice cold solution of 20 ml. of water in 200 ml. of acetic acid. The organic layer was washed successively with 5% sodium thiosulfate solution, 10% potassium bicarbonate solution and water. After the solution had been dried the solvent was evaporated. The residual oil was mixed with a solution of 35 g. of hydrated copper sulfate in 25 ml. of water and 20 ml. of pyridine. The mixture was heated under reflux, with stirring, for four hours. The yellow *o*-tolil was distilled at 140–170° (2 mm.). It solidified and was recrystallized from ethanol; m. p. 92–94° (cor.)[2]; yield 34%.

Anal. Calcd. for $C_{16}H_{14}O_2$: C, 80.65; H, 5.92. Found: C, 80.98; H, 6.25.

o-Tolil was found to undergo the benzilic acid rearrangement. To a solution of 10 g. of *o*-tolil in 200 ml. of ether was added a solution of 5 g. of sodium ethoxide in 40 ml. of 95% ethanol. The container was stoppered tightly and allowed to stand for twenty-four hours. The solution was extracted with 200 ml. of water. Acidification of the aqueous solution precipitated 7 g. of the **di-*o*-tolylglycolic acid.** It was recrystallized from benzene; m. p. 162–163°; yield 65%. It gave a purple color with concentrated sulfuric acid.

Anal. Calcd. for $C_{16}H_{16}O_3$: C, 74.98; H, 6.29; neut. equiv., 256. Found: C, 74.85; H, 6.49; neut. equiv., 251.

(1) Gomberg and Bachmann, THIS JOURNAL, **49**, 236 (1927).

(2) Since this work was done, *o*-tolil has been described by Kharasch, Nudenberg and Simons [THIS JOURNAL, **65**, 495 (1943)].

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X-[N-(β-Acetamidoethyl)-N-methyl]-aminoazobenzene

Aniline (0.98 ml., 0.0108 mole) was dissolved in a mixture of 2.50 ml. (0.030 mole) of concentrated hydrochloric acid and about 8 ml. of water. The temperature was brought to 0° by adding ice, and the solution was diazotized at 0–5° by adding in the usual manner a solution of 0.77 g. (0.0108 mole) of 97% sodium nitrite in 1.5 ml. of water.

(1) Fuson and Rachlin, THIS JOURNAL, **64**, 1567 (1942).