tempts were made to recrystallize the material without success. Partial oxidation occurred when it was heated in various solvents.

9-aci-Nitrofluorene. This material was prepared by acidification of a water solution of potassium 9-fluorenenitronate with either sulfuric acid or trifluoroacetic anhydride. It is precipitated immediately from water as a yellow-green powder, m.p. 134.5–137° (lit.[2], m.p. 134–137°).

aci-d-9-Nitrofluorene. A solution of 0.05 g. of potassium 9-fluorenenitronate in 2.2 g. of deuterium oxide was prepared in a flask protected from atmospheric moisture. The flask was immersed in an ice-bath and two drops of trifluoroacetic anhydride were added. The yellow-green *aci*-compound precipitated immediately. It was collected and stored in a vacuum desiccator over phosphorus pentoxide.

1,2-Dinitro-1,2-bidiphenyleneëthane. This material was prepared by the method of Nenitzescu.[7] It was recrystallized from acetic acid, m.p. 183–184° (lit.[7] m.p. 184°).

Spectra. The infrared spectra were measured as Nujol mulls on a Perkin Elmer Model 21 infrared spectrophotometer with a rock salt prism. The ultraviolet spectrum was measured in absolute ethanol with a Beckman DK-1 ultraviolet spectrophotometer.

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Catalytic N-Alkylation of Aniline with 1-Propanol

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Rice and Kohn[1] recently reported that Raney nickel catalyzes the N-alkylation of aniline with alcohols but that U.O.P. nickel catalyst[2] is ineffective for this purpose. Evidently the batch of catalyst used by Rice and Kohn was poisoned or it was not properly reduced before use, as we have found the U.O.P. nickel catalyst to be at least as active as Raney nickel. An 80% yield of N-*n*-propylaniline was obtained in 6 hours whereas an equal amount of Raney nickel required 16 hours[1] to give the same yield.

Small amounts of acetaldehyde, propionaldehyde, and acetone were formed when ethanol, 1-propanol, and 2-propanol, respectively, were refluxed with U.O.P. nickel catalyst. This supports the reaction mechanism suggested by Rice and Kohn[1] for the catalytic N-alkylation of aniline with alcohols, namely that the alcohol is dehydrogenated to a carbonyl compound which then reacts with the amine to give an N-alkylideneamine (or an α-hydroxyamine) and that this intermediate is subsequently hydrogenated to the corresponding N-alkylaniline.

EXPERIMENTAL[3]

Materials. 1-Propanol, 2-propanol (both stored over Drierite), and aniline were freshly distilled. The U.O.P. nickel catalyst, obtained from the Harshaw Chemical Co., was prereduced at 425° in a stream of hydrogen, cooled under hydrogen, and pulverized under the appropriate alcohol before use.

N-n-Propylaniline. A mixture of 25 g. (0.28 mole) of aniline, 15 g. of prereduced U.O.P. nickel catalyst, and 80 g. (1.3 moles) of 1-propanol was refluxed with stirring for 6 hours. The cooled mixture was filtered and the catalyst was washed with 25 ml. of 1-propanol. The combined filtrate-wash was concentrated, and the residual liquid was distilled through a 10-cm. Vigreux column to give 29.3 g. (80% yield) of N-*n*-propylaniline, b.p. 110–111°/20 mm., n_{D}^{25} 1.5410.

The product was analyzed for secondary aromatic amine by electrometric titration.[4]

Anal. Calc'd for $C_9H_{13}N$: Secondary amine, 7.42 meq./g. Found: Secondary amine, 7.44 meq./g.

Its *phenylthiourea* derivative melted at 101–102°; its mixture melting point with an authentic sample was also 101–102°.[5]

Dehydrogenation of ethanol. A mixture of 79 g. of absolute ethanol and 15 g. of prereduced U.O.P. nickel catalyst was refluxed with stirring for 12 hours. The vapors which passed the water-cooled condenser were condensed in a Dry Ice-trap.

The condensate gave a *dimedone derivative* melting at 138–140° whose mixture melting point with an authentic sample of acetaldehyde dimedone derivative was also 138–140°.[6]

Dehydrogenation of 1-propanol. A mixture of 120 g. (2.0 moles) of 1-propanol and 10 g. of prereduced U.O.P. nickel catalyst was refluxed under a 30-cm. Vigreux column for 10 hours and 89 g. of distillate was collected.

Analysis of the distillate for carbonyl indicated the presence of 0.6 g. (0.01 mole) of propionaldehyde.

Anal. Calc'd for C_3H_6O: Carbonyl, 16.7 meq./g. Found: Carbonyl, 0.11 meq./g.

The distillate gave the *dimedone derivative of propionaldehyde*, m.p. 155–156°; its mixture m.p. with an authentic sample was also 155–156°.[6]

Dehydrogenation of 2-propanol. A mixture of 120 g. (2.0 moles) of 2-propanol and 10 g. of prereduced U.O.P. nickel catalyst was refluxed under a 30-cm. Vigreux column for 6 hours and 53.5 g. of distillate was collected.

Analysis of the distillate for carbonyl indicated the presence of 1.5 g. (0.03 mole) of acetone.

Anal. Calc'd for C_3H_6O: Carbonyl, 16.7 meq./g. Found: Carbonyl, 0.45 meq./g.

The distillate gave the 2,4-dinitrophenylhydrazone of acetone, m.p. 122–123°; its mixture m.p. with an authentic sample was also 122–123°.[6]

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