butyl selenocyanate, although sealed in ampules and kept in the dark, turned black after a month; the yellow *n*-hexyl compound slowly turned pink but got no darker; the colorless *n*-decyl compound remained colorless although exposed to light and air.

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1,5-Endomethylene-3,7-di-*p*-toluenesulfonyl-1,3,5,7-tetrazacycloöctane

A variation in reaction conditions has produced this new compound instead of the 1,3,5-tri-*p*-toluenesulfonylcyclotrimethylenetriamine reported by McMaster.[1] To 0.05 mole (7 g.) of hexamethylenetetramine in 80 cc. of water was added 0.199 mole (37.8 g.) of *p*-toluenesulfonyl chloride. To this stirred solution, covered with 60 cc. ether, was added 53 cc. of a 15% aqueous sodium hydroxide solution (0.199 mole, 7.95 g. sodium hydroxide) at such a rate that the alkalinity was held at *p*H 8.5-10. The product was a sticky mass which was filtered after trituration with ethanol to weigh 10 g. and melt about 220°. Two crystallizations from nitromethane increased this melting point to 236°. Yield of crude product is 46% of theoretical, and is almost insoluble in acetone, benzene, chloroform, ether and water but dissolves in pyridine.

Anal. Calcd. for $C_{19}H_{24}O_4N_4S_2$: C, 52.3; H, 5.42; N, 12.8; S, 14.6. Found: C, 52.5; H, 5.50; N, 12.6; S, 15.4.

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[1] McMaster, THIS JOURNAL, **56**, 204 (1934).

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1-(1-Diethylamino-3-propyl)-6-methoxy-3-methyl-1,2-dihydro-2-keto-quinoxaline

Twenty-three grams (0.091 mole) of 3-amino-4-(1-diethylamino-3-propylamino)-anisole[1] and 9.7 g. (0.11 mole) of pyruvic acid were allowed to react in 200 cc. of *p*-cymene according to the procedure of Kipnis, Weiner and Spoerri.[2] After refluxing for two hours, the solution was allowed to cool and the base removed from the cymene by extraction with dilute hydrochloric acid. The aqueous layer was made alkaline (ammonium hydroxide), extracted with carbon tetrachloride, and the organic layer was steam-distilled until about 3 liters of distillate were collected. The residual oil was again taken up in carbon tetrachloride, dried over potassium carbonate and distilled. The first distillation took place with much decomposition, the product being collected at 210-230° (4 mm.); however, on redistillation in a 15-cc. Claisen flask with a heated Vigreux column, the product boiled without decomposition at 195-197° (3 mm.), giving 9.5 g. (34.5% of the theoretical) of the product, a light yellow oil.

Anal. Calcd. for $C_{17}H_{25}N_3O_2$: N, 13.85. Found: 14.00.

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[1] Prepared by the method of Izmail'skiĭ and Simonov, *J. Gen. Chem. U. S. S. R.*, **10**, 1588 (1940); through *C. A.*, **35**, 2870 (1941). A substantial increase in yield (93%) was obtained by reducing the corresponding 3-nitro compound with hydrogen and Raney nickel at room temperature and an initial pressure of 2 atmospheres.

[2] Kipnis, Weiner and Spoerri, THIS JOURNAL, **66**, 1989 (1944).

[3] The Wm. S. Merrell Co., Postdoctoral Fellow.

A New Derivative of Kojic Acid

Four and twenty-five hundredths grams (0.03 mole) of kojic acid (assay better than 90%[1]) was dissolved in 50 ml. of ethanol. To this mixture was added 1.2 g. of sodium hydroxide dissolved in 5 ml. of water, and after thoroughly mixing the two solutions 5.97 g. (0.03 mole) phenacyl bromide was added. The resulting solution was shaken in a stoppered 125-ml. Erlenmeyer flask for fifteen minutes, during which time the phenacyl derivative had begun to precipitate out. The flask was then heated, under reflux, over a steam-bath for thirty minutes. The material was cooled and filtered, and repeatedly washed with water; the yield was 7.7 g. The compound was recrystallized twice from 90% alcohol producing long white needles, m. p. 156-157°.

Elementary analysis at this point gave inconclusive results. The compound was recrystallized twice from absolute ethanol, dried in the oven at 110° for twenty-four hours, and finally allowed to cool in the vacuum desiccator over sulfuric acid; m. p. 157.5-158°. The compound gave no test with ferric chloride and reduced Fehling solution only on prolonged heating.

Anal.[2] Calcd. for $C_{14}H_{12}O_6$: C, 60.88; H, 4.35. Found: C, 60.78; H, 4.40.

The analysis has shown that the expected 2-hydroxymethyl-5-phenacyl oxy-α-pyrone was not formed.

A small amount of the compound was acetylated in the usual way[3] recrystallized once from absolute ethanol and dried in the vacuum desiccator over sulfuric acid; m. p. 129-130°.

Anal. Calcd. for $C_{16}H_{14}O_7$: C, 60.38; H, 4.43. Found: C, 60.5; H, 4.6.

No attempt is made in this note to postulate the complete structure of these derivatives of kojic acid.

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RECEIVED MARCH 29, 1946

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[1] H. N. Barham, *Ind. Eng. Chem., Anal. Ed.*, **11**, 31 (1939).

[2] Analyses by Dr. Carl Tiedcke.

[3] Shriner and Fuson, "Identification of Organic Compounds," 2nd ed. John Wiley and Sons, New York, N. Y., p. 137.