found that comparable yields could be obtained in a much shorter time if ethanol or water was used as the solvent.

A mixture of O,O-diethyl chlorothiophosphate (37.7 g.), anhydrous sodium *p*-nitrophenoxide (32.2 g.), and ethanol (200 cc.) was refluxed for one hour, cooled to 20°, filtered, and the filtrate concentrated *in vacuo*. The residue was heated (oil-bath at 100–110°) with stirring at 0.5 mm. for a short period to remove any unreacted O,O-diethyl chlorothiophosphate. The crude parathion was dissolved in toluene (100 cc.) and washed with 5% sodium carbonate, then with water. After drying over Drierite and removal of the toluene at reduced pressure, the product weighed 43.5 g. (75% yield), n^{25}_D 1.5361. Distillation of a 200-g. sample prepared in another experiment gave 182 g. of pale yellow oil, b. p. 157–162° (0.6 mm.), n^{25}_D 1.5370.

Anal. Calcd. for $C_{10}H_{14}NO_5PS$: C, 41.23; H, 4.84; N, 4.81; P, 10.65; S, 11.01. Found: C, 41.38; H, 4.93; N, 4.60; P, 10.66; S, 11.06.

O,O-Diethyl chlorothiophosphate (41.6 g.) was added during one-half hour to sodium *p*-nitrophenoxide (32.2 g.) in water (100 cc.) at 95–100°, and stirring was continued at this temperature for two hours. After cooling to 20° the lower layer was separated, washed three times with water, and dried over sodium sulfate to give 37 g. (64% yield) of brown oil, n^{25}_D 1.5374.

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Streptomyces Antibiotics. XX. Conversion of Streptamine into Streptidine

BY FREDERICK W. HOLLY, RALPH MOZINGO AND KARL FOLKERS

Conversion of streptamine into streptidine with S-methyl isothiourea has been reported.[1] Another of the general methods for the preparation of guanidines from amines is also applicable for this conversion. Streptidine is formed when streptamine hydrochloride is heated with aqueous cyanamide. A 6% yield of streptidine, as the sulfate, was obtained by heating the mixture at 100° for forty-eight hours and a 17% yield by heating at 155° for two hours under hydrogen pressure.

Experimental

Streptidine Sulfate.—A solution prepared from 753 mg. of streptamine hydrochloride and 252 mg. of cyanamide in 2.5 ml. of water was heated for two hours at 155° under hydrogen pressure. The reaction mixture was cooled and a dark amorphous solid was removed. To the clear solution 1.0 ml. of concentrated sulfuric acid was added, and the solution was adjusted to *p*H 9 with ammonium hydroxide. The solution was concentrated under reduced pressure to a mixture of oil and crystals, and 4 ml. of 6 *N* ammonium hydroxide was added. The mixture was cooled, the crystals were collected on a filter, washed successively with water, alcohol and ether, and dried to give 180 mg. (17%) of streptidine sulfate.

Anal. Calcd. for $C_8H_{18}N_6O_4 \cdot H_2SO_4 \cdot H_2O$: C, 25.39; H, 5.86; N, 22.21. Found: C, 25.41; H, 5.60; N, 21.68.

A picrate prepared from the sulfate melted at 280–282°; when mixed with streptidine picrate, m. p. 280–282° (dec., microblock), the melting point was unchanged.

A solution containing 200 mg. of streptamine hydrochloride and 400 mg. of cyanamide in 10 ml. of water was refluxed for forty-eight hours. From the reaction mixture 14 mg. (6%) of streptidine sulfate was isolated by the procedure described above.

Concentration of the filtrate after removal of streptidine sulfate gave crystalline streptamine sulfate.

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(1) Wolfrom and Polglase, THIS JOURNAL, **70**, 1672 (1948).

The Ultraviolet Absorption Spectrum of Gossypol[1]

BY VERNON L. FRAMPTON, JOSEPH D. EDWARDS, JR., AND HENRY R. HENZE

In the formulation of a structure for gossypol, significance was attached to the absorption maximum at approximately 250 millimicrons.[2] We find no evidence for the existence of this maximum in the absorption spectrum of gossypol.

A preparation of gossypol[3] was repeatedly recrystallized from diethyl ether–petroleum ether (30–40°) and finally from chloroform, m. p. sharp at 199°. The absorption spectrum (triangles, Fig. 1) in 95% ethyl alcohol was then determined with a Beckman quartz spectrophotometer. After additional recrystallization from diethyl ether–petroleum ether, from petroleum ether (45–90°) and finally from chloroform, m. p. sharp at 199°, the absorption spectrum (dots, Fig. 1) was again determined.

Nine different gossypol preparations,[4] each from

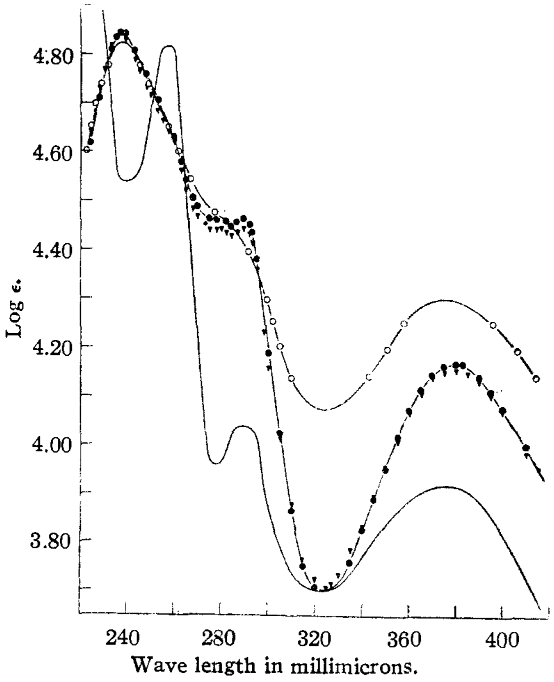

Fig. 1.—● ● ● and ▼ ▼ ▼, gossypol, authors; ○ ○ ○, gossypol-acetic acid, Grunbaumowna and Marchlewski ref. 4; ——, gossypol, Adams and Kirkpatrick, ref. 1.

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(1) These several data were drawn from a thesis presented by Joseph Daniel Edwards, Jr. to the Faculty of the Graduate School of the University of Texas in partial fulfillment of the requirements for the Master of Arts degree, January, 1948.

(2) Adams and Kirkpatrick, THIS JOURNAL, **60**, 2180 (1938).

(3) Campbell, Morris and Adams, *ibid.*, **59**, 1723 (1937).

(4) Dr. Boatner kindly supplied two preparations.