

TABLE I

No.	Guanyl- urea sulfate, mg.	Urea added, mg.	HCl, ml. cor.	Urea found, mg.
1	100	0	-0.03	..
2	0	30.0	10.68	29.8
3	100	30.0	10.62	29.7
4	100	30.0	10.36	28.9
5	100	30.0	10.39	29.0

chloric acid required for an end-point when guanylurea sulfate and urease were incubated together. Similar results were obtained when the incubation time for the mixture was increased to thirty minutes. In experiment 3 the urease was added to a mixture of guanylurea sulfate and urea. To determine whether the action of urease on urea would be influenced by previous treatment with guanylurea sulfate, the urease was added to the guanylurea sulfate in Expts. 4 and 5 and kept in contact with it at room temperature for five and for fifteen minutes, respectively, before the urea was added.

Results.—The experiments indicate that urease does not catalyze the hydrolysis of guanylurea sulfate. A solution of urease which has first been treated with guanylurea sulfate gives somewhat low values in the determination of urea; this effect is, however, very slight.

TEMPLE UNIVERSITY
SCHOOL OF MEDICINE
PHILADELPHIA 40, PA.

RECEIVED APRIL 25, 1946

Picrolonates of Bufotenine, Bufotenidine and Dehydrobufotenine

BY VENANCIO DEULOFEU AND BLANCA BERINZAGHI

Following our work¹ on the basic constituents of the venom of some South American toads, we have prepared the picrolonates of the indolic bases present in toad venom.

The picrolonates of bufotenidine and dehydrobufotenine can be prepared and purified more easily than bufotenine picrolonate. Dehydrobufotenine can be isolated more readily as the picrolonate than as its picrate, from the final residual mother liquors of the toad venom when the bases are extracted according to our procedure.¹ Dehydrobufotenine picrolonate, however, has too high a melting point to be of ready use in identification.

Bufotenine Picrolonate.—Amorphous bufotenine from *B. arenarum* was dissolved in ethanol and treated with a small excess of picrolonic acid. By heating the acid dissolves and when the solution was cooled crystals were obtained melting not sharply at 110°. By recrystallizing from ethanol, small, yellow prisms, melting 120–121° were obtained.

Anal. Calcd. for C₁₂H₁₆ON₂·C₁₀H₈O₅N₄: N, 17.94. Found: N, 18.34.

Bufotenidine Picrolonate.—Bufotenidine iodide was obtained from amorphous bufotenine and methyl iodide according to Wieland, Konz and Mittasch,² and melted at 210°. The iodide was dissolved in a little amount of water and treated with a slight excess of picrolonic acid. This was dissolved by heating, and on cooling yellow

prisms melting 253–255° were obtained. Recrystallized from 50% ethanol, the fine yellow needles melted 255°.

Anal. Calcd. for C₁₃H₁₈ON₂·C₁₀H₈O₅N₄: N, 17.42. Found: N, 17.14.

Dehydrobufotenine picrolonate was obtained by treating a water solution of dehydrobufotenine hydrochloride with an excess of picrolonic acid, heating to dissolution and cooling. The picrolonate precipitates and after crystallization from ethanol (50%) yellow prisms melting above 300° and darkening from 275° (quick heating) were obtained.

Anal. Calcd. for C₁₂H₁₄ON₂·C₁₀H₈O₅N₄: N, 18.02. Found: N, 17.99.

A similar picrolonate was obtained from the solution of crude bases of *B. arenarum* after separation of bufotenine.

FACULTAD DE CIENCIAS EXACTAS F. Y. N.
LABORATORIO DE QUÍMICA ORGÁNICA

BUENOS AIRES, ARGENTINA RECEIVED MARCH 20, 1946

The Use of Liquid Phase Oxidation for the Preparation of Nuclearily Substituted Styrenes. II. *p*-Vinylphenyl Acetate

BY WILLIAM S. EMERSON, JOSEF W. HEYD, VICTOR E. LUCAS, WILLIAM B. COOK, GRAFTON R. OWENS AND ROBERT W. SHORTRIDGE

In a previous paper¹ we have shown that methyl *p*-ethylbenzoate is smoothly oxidized to methyl *p*-acetylbenzoate by air in the presence of chromium oxide and calcium carbonate. While the oxidation of *p*-ethylphenyl acetate to *p*-acetylphenyl acetate is a great deal more difficult, we have accomplished it successfully (24% conversion and 79% yield) by means of oxygen in the presence of a chromium oxide-cobalt hydrate-calcium carbonate catalyst. Any free phenol in the reaction mixture inhibits the oxidation altogether, so that its presence must be rigorously avoided.

The *p*-acetylphenyl acetate was smoothly hydrogenated in the presence of copper chromite to *p*-(α -hydroxyethyl)-phenyl acetate. Distillation of the latter compound from potassium bisulfate yielded 48% of *p*-vinylphenyl acetate based on this carbinol.

The authors are grateful to Dr. G. F. Deebel and Messrs. C. E. Wheelock, E. L. Ringwald and R. P. Haase for the preparation of considerable quantities of *p*-ethylphenol.

Experimental

***p*-Ethylphenol** was prepared essentially according to the method of Hartman.² One hundred fifty-two grams (58%) was obtained from the fusion of 450 g. of sodium *p*-ethylbenzenesulfonate with 300 g. of potassium hydroxide and 750 g. of sodium hydroxide. It boiled at 95–101° (10 mm.) (218.5–219.5°).³

***p*-Ethylphenyl Acetate.**—*p*-Ethylphenyl acetate was prepared by refluxing for six hours 713 g. of *p*-ethylphenol with 1 liter of acetic anhydride containing 100 g. of sodium acetate. The reaction mixture was diluted with water and benzene, the layers separated and the benzene layer dis-

(1) Emerson, Heyd, Lucas, Chapin, Owens and Shortridge, *THIS JOURNAL*, **68**, 674 (1946).

(2) Hartman, "Org. Syntheses," Coll. Vol. I, p. 175.

(3) Béhal and Choay, *Bull. soc. chim.*, [3] **11**, 209 (1894).

(1) V. Deulofeu and E. Duprat, *J. Biol. Chem.*, **153**, 459 (1944).

(2) H. Wieland, W. Konz and H. Mittasch, *Ann.*, **513**, 1 (1934).