and the solid which separated was filtered. The filtrate was evaporated to dryness to give an oily residue which on triturating with a little ethyl acetate solidified. The solid was filtered and recrystallized from acetonitrile; total yield of ethyl dihydroxymandelate, 100 g. Recrystallization from acetonitrile gave 90 g. (52%) of pure product, m.p. 147–149°, (reported by Shaw, *et. al. loc. cit.*, m.p. 153–154° and by Barger and Ewins,[3] m.p. 152–153°).

3,4-Dihydroxymandelic acid, VI (*R* = *H*). Ethyl 3,4-dihydroxymandelate V (R = H), 80 g., was hydrolyzed as described for ethyl 3-methoxy-4-hydroxymandelate. The product was recrystallized from 100 ml. of acetonitrile to give 57 g. (81%) of pure dihydroxymandelic acid, m.p. 145–146° (reported by Shaw, *et al. loc. cit.*, m.p. 137°).

Anal. Calcd. for $C_8H_8O_5$: C, 52.18; H, 4.38. Found: C, 52.15; H, 4.45. The dicyclohexylammonium salt was prepared as described above and was recrystallized from isopropyl alcohol, m.p. 209–211°.

Anal. Calcd. for $C_{20}H_{31}O_5N$: C, 65.71; H, 8.55; N, 3.83. Found: C, 65.47; H, 8.19; N. 3.84.

Chromatography. Small scale chromatograms[4] on Schleicher and Schuell (609) paper were developed with three different solvent systems: (a) 1-butanol, acetic acid, water (8:2:2), (b) isopropyl alcohol, concd. ammonia, water (8:1:1), (c) methylbutynol, 2*N* ammonia (7:3), and furnished single spots which were visualized with diazotized sulfanilic acid. The Rf values for 3-methoxy-4-hydroxymandelic acid in the above solvent systems were: (a) 0.715 (5:1:4), (b) 0.52, (c) 0.42, and for 3,4-dihydroxymandelic acid: (a) 0.51, (b) 0.14, (c) 0.15.

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The Presence of Mescaline in *Opuntia cylindrica*

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In 1948 Guillermo Cruz-Sanchez[1] described the pharmacology of *Opuntia cylindrica* and an alkaloid derived therefrom. In two other papers with Dr. Carlos Gutierrez-Noriega[2,3] were reported the effects of oral administration of this alkaloid in doses varying from 5 to 11.5 g. per kilo to a total of thirty-four subjects, of whom two developed a brief psychotic state. The method of preparation of the alkaloid, some of its physical and chemical properties, the psychological changes, as well as the dosage of the alkaloid employed, suggested the possibility of the presence of mescaline. However, the material was never adequately purified and the laboratory facilities for satisfactory identification were unavailable. The only other reference to *Opuntia cylindrica* we have been able to find is in a review article by Buscaino.[4]

Through the courtesy of Dr. Vincente Zapata Ortiz of the University of San Marcos, Peru, an alcohol extract was sent us for examination. Further, through the courtesy of Dr. Leoncio Zapata, a physician on the staff of this hospital, we received several kilograms of the whole dried plant of *Opuntia cylindrica*. We have been able to identify the alkaloid present as mescaline, present in concentration of 0.9% of the whole dried plant. There is no more than a slight trace of additional alkaloids.

EXPERIMENTAL

Isolation and characterization of Mescaline. Three hundred grams of the powdered dried *Opuntia* was moistened with a mixture of methanol-aqueous ammonia (20:1). The mass was transferred to a glass chromatography column which was set up for continuous Soxhlet extraction with chloroform. After 24 hr. of extraction the residue left after evaporation of 10 ml. of final chloroform extract did not give a filter paper spot with ninhydrin. The extract was partly evaporated in a stream of air at room temperature, treated with an excess of 5% acetic acid, and extracted with water. The aqueous solution was extracted with benzene, which took up most of the lipids. This was brought to *p*H 7–7.6. Benzene extraction yielded a trace of material giving a ninhydrin test, but this could not be isolated. The *p*H of aqueous solution was raised to 10 with sodium hydroxide. Benzene extraction now resulted in quantitative transferral of the ninhydrin-positive material to the benzene. Benzene was washed twice with distilled water, evaporated to dryness in a stream of warm air, and finally over phosphorus pentoxide. Exactly 2 g. of one extraction at this point dissolved in 10 ml. of 95% ethanol and was titrated to *p*H 3.0 with 6.19 ml. of 1*N* sulfuric acid, brought to dryness, and the residue extracted with benzene. The white, semicrystalline residue weighed 1.31 g. which, on the basis of the titration in sulfuric acid, corresponds to a molecular weight of 211 for the raw base. The sulfate was recrystallized three times from water-ethanol and the melting point compared with authentic mescaline sulfate in a Fisher-Johns melting point apparatus. The crystals of the *Opuntia* sulfate melted sharply at 184° simultaneously with mescaline sulfate. The crystals of *Opuntia* and of mescaline sulfates were mixed and recrystallized in alcohol; they melted sharply at 184°. The chromatogram on Whatman No. 1 filter paper of authentic mescaline sulfate, *Opuntia* sulfate, and a mixture of the two gave a single ninhydrin-positive spot, Rf 0.48, when developed by ascending chromatography with butanol, acetic acid, water (4:1:1). The picrates of *Opuntia* and mescaline were prepared. After three recrystallizations they melted sharply and simultaneously on the cover of the slip at 224°. The maximum yield of crude base was 0.9%. The filtrates recovered after the recrystallizations of the mescaline were further worked up. Finally a crystalline, very bitter material was obtained in very small yield, which was chromatographed and compared with mescaline. When

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the mescaline had Rf 0.48, and gave a single well-rounded spot, the residual material gave both a mescaline spot and a faint tail suggestive of presence of another alkaloid. This was not identified.

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Isolation of Evolitrine from *Cusparia macrocarpa*

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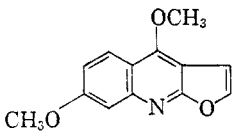
Cusparia macrocarpa is a rutaceous plant indigenous to Brazil where the leaves and stems are used in folk medicine. As part of an investigation of Brazilian flora, we have examined this plant for alkaloids.

The residue from an alcoholic extract of the leaves and stems[2] was distributed between an aqueous phase, at various *p*H's, and ether, essentially following the general scheme used with *Balfourodendron riedelianum*.[3] Further purification of the crude fractions was effected by chromatography on alumina.

Crystalline material resulted only from that fraction obtained by continuous extraction of the aqueous phase at *p*H 2. This very weakly basic compound had the composition $C_{13}H_{11}O_3N$. Its ultraviolet absorption (λ_{max} 246, 308, 319, 333 mμ) was practically identical with that reported for evolitrine[4] (I), and it formed a picrate with the



same melting point as reported[4] for evolitrine picrate (201–202°). Direct comparison[5] by ultraviolet and infrared absorption, and mixed melting point clearly established the identity of this alkaloid as evolitrine.

A paper chromatographic examination of the various fractions showed the absence of any other alkaloidal material. However, evidence was obtained for the presence of a polar, non-extractable form of evolitrine which was converted to evolitrine by the action of alkali. This evidence was the fact that the ether extract of the *p*H 7 aqueous phase showed the complete absence of evolitrine, but when this extraction was continued at *p*H 10, evolitrine was found in the ether phase. Efforts to isolate this polar form by addition of chloride ion and extraction with butanol failed.

EXPERIMENTAL

The *isolation scheme* was the same as that used previously.[3] From 2.3 kg. of plant material (leaves and stems) were isolated ether extracts from the aqueous phase at *p*H 2, 4, 7, and 10. These fractions were chromatographed on alumina using benzene, benzene-chloroform, and chloroform for elution. Recombination on the basis of ultraviolet absorption and crystallization from benzene-hexane led to 600 mg. of evolitrine from the *p*H 2 extract. This material, after sublimation, melted at 113–114° (reported[4] m.p. 114–115°). It formed a picrate with alcoholic picric acid, m.p. 201–202° (reported[4] 201–202°).

Paper chromatography of the various fractions and subfractions was carried out by the ascending method with 1-butanol–5% acetic acid as solvent and Dragendorff's reagent for detection.

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(5) We are grateful to Dr. R. G. Cooke, University of Melbourne, for this sample.

Some Reactions of Triphenylethoxysilane

HENRY GILMAN AND T. C. WU

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In connection with studies on the comparison of organosilicon compounds with their carbon analogs,[1] some reactions of triphenylethoxysilane have been examined.

Alkoxytriphenylsilanes resemble alkoxytriphenylmethanes in one respect; they react with potassium metal to give triphenylsilylpotassium[2] and triphenylmethylpotassium,[3] respectively. However, the reaction of triphenylethoxysilane with sodium does not give the silylsodium compound or hexaphenyldisilane. Under similar conditions, triphenylchlorosilane reacts with sodium to give high yields of hexaphenyldisilane.[4]

When triphenylmethyl ethyl ether is treated with phenyllithium or with *n*-butyllithium, 9-phenylfluorene is formed on hydrolysis.[5] How-

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