The rate of solution was estimated by the color development, while completeness of solution and stability were determined by analysis for mercury (2).

The most satisfactory method of preparation consists in first forming the ephedrine salts of the mercurial and of the fatty acid and then stirring these into the oil.

The concentration of mercurial which may be obtained is limited by the requirement that on an average four mols of the ephedrine salt are required for each mol. of the mercurial amine salt. Stable preparations containing 1.0% of the latter have been made.

LITERATURE CITED.

(1) A. L. Wilson, *Ind. Eng. Chem.*, 22 (1930), 143.
(2) Moore and Shelberg, *Ind. Eng. Chem., Analyt. Edit.*, 4 (1933), 224.

A COMPARISON OF THE EFFECT OF PHENYL ETHANOLAMINE AND EPHEDRINE ON NASAL MEMBRANES.*

BY T. B. GRAVE AND W. G. CHRISTIANSEN.

Ephedrine solutions are widely used for application to nasal membranes, and Tainter (*J. Pharmacol. Exper. Therap.*, 36 (1929), 52) described the satisfactory behavior of phenyl ethanolamine on nasal membranes. It was, therefore, of interest to prepare phenyl ethanolamine for comparison with ephedrine. The phenyl ethanolamine was prepared by methods described in detail below and was tested as the hydrochloride in 1, 2 and 4% aqueous solution and as the oleate in mineral oil solution using in both cases corresponding ephedrine solutions as controls. The solutions of the oleates were prepared by dissolving 5 Gm. of the base with a chemical equivalent quantity of oleic acid in mineral oil so that the total volume was 100 cc. The tests consisted of applying these solutions to the nasal membranes of both horses and human beings and observing the degree and duration of blanching and recording in the experiments on human beings the relief. As a result of these tests we conclude that there is little difference between the phenyl ethanolamine and ephedrine.

EXPERIMENTAL.

Preparation of ω-Aminoacetophenone.—$C_6H_5.CO.CH_2Cl + (CH_2)_6N_4 \longrightarrow C_6H_5.CO.CH_2.[(CH_2)_6.N_4].Cl.$

$C_6H_5.CO.CH_2[(CH_2)_6.N_4].Cl + 3HCl + 12C_2H_5OH \longrightarrow C_6H_5CO.CH_2.NH_2.HCl + 3NH_4Cl + 6CH_2.(OC_2H_5)_2$

The method of Mannich and Hahn, *Ber.*, 44 (1911), 1542, was used.

23 Gm. of chloracetophenone was dissolved in 140 cc. of chloroform and stirred with 21 Gm. of hexamethylene tetramine until complete solution took place (two hours). After standing over night the addition-reaction was complete; the mass of glistening crystals was filtered off and washed with cold chloroform. Yield 40 Gm. This product was "alcoholyzed" by a mixture of 320 cc. of absolute alcohol and 40 cc. of concentrated hydrochloric acid. After standing 72 hours at room temperature, the precipitated ammonium chloride was filtered off and the alcoholic solution concentrated *in vacuo*. The crude ω-aminoacetophenone hydrochloride which separated

* Section on Practical Pharmacy and Dispensing, Madison meeting, 1933.

was recrystallized from 10 cc. of hot water. Yield 6.75 Gm., melting at 190–192°, with decomposition.

Catalytic Hydrogenation to Phenyl Ethanolamine.—$C_6H_5.CO.CH_2.NH_2.HCl \xrightarrow{H_2} C_6H_5.CHOH.CH_2.NH_2.HCl$.

0.2 Gm. of platinum oxide (prepared by the method of Adams and Shriner, Org. Syn., VIII, 92) was reduced by shaking with hydrogen in aqueous suspension. The platinum black was filtered off and added to the solution of 6.75 Gm. of ω-aminoacetophenone hydrochloride in 225 cc. of redistilled alcohol and 12.5 cc. of conc. HCl. Absorption of hydrogen took place at the rate of 2 cc. per minute. When 970 cc. had been taken up, the reaction was stopped; theoretical for 1 mol., 963 cc. The filtrate from the platinum was evaporated to dryness *in vacuo*. The residue was twice recrystallized from absolute alcohol-ether; yield, 4.31 Gm., melting point 165–168°, with decomposition. The composition of the compound was verified by analysis of its chloroplatinate.

0.2 Gm. of the hydrochloride was dissolved in 2 cc. of absolute alcohol containing 0.25 cc. of HCl, and treated with 3 cc. of 10% aqueous chloroplatinic acid. The orange precipitate was washed with ice-cold alcohol and dried at 105°. Yield 0.29 Gm., melting point 203–204° with decomposition.

Analysis:

Found: Pt.: 28.45%.
Calcd. for $(C_8H_{11}ON)_2.H_2PtCl_6$: 28.53%.

Preparation of the Oleate.—In order to avoid all possibility of decomposition, the free base was prepared by the action of silver oxide and converted, without isolation, to the oleate. 4.0 Gm. of the hydrochloride was dissolved in alcohol and an excess of freshly precipitated silver oxide stirred in until the formation of silver chloride was complete. The filtrate was evaporated *in vacuo* with the theoretical quantity of oleic acid (6.54 Gm.) until free of alcohol.

The neutral oleate thus obtained was dissolved in liquid petrolatum for the physiological tests.

The biological tests on compounds reported herein were made in the Biological Research Laboratories of E. R. Squibb and Sons and we gratefully acknowledge their assistance.

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VARIATIONS IN HAND-MOLDED HYPODERMIC TABLETS.*

BY S. WALLEY BOWER.

During the past year the question of Variations in Hand-Molded Hypodermic Tablets arose, which suggested the following observations:

(*a*) What is the error in the manufacture of these tablets as based on the theoretical?

(*b*) What variations take place when the molding of the same lot of tablets extends over a period of several days?

(*c*) What is the relationship of the percentage error of the total count of the entire lot (as an average) with the error of tablets when weighed in small subdivisions?

* Section on Practical Pharmacy and Dispensing, A. PH. A., Madison meeting, 1933.