with lithium aluminum hydride produced the corresponding amine, 5-benzyloxytryptamine, which was isolated as the hydrochloride, m.p. 265° (*Anal.* Calcd. for $C_{17}H_{18}N_2O\cdot HCl$: C, 67.43; H, 6.33; N, 9.26. Found: C, 67.39; H, 6.22; N, 9.32). Catalytic debenzylation of this amine hydrochloride afforded the desired 5-hydroxytryptamine hydrochloride, a light-sensitive hygroscopic salt, m.p. 167–168° (*Anal.* Calcd. for $C_{10}H_{20}N_2O\cdot HCl$: C, 56.47; H, 6.16; N, 13.18. Found: C, 56.07; H, 6.20; N, 12.94). The picrate, formed in water from the hydrochloride, melted (Fischer–Johns apparatus) from 103–111°, resolidified at 124–134° and remelted from 185–189° (*Anal.* Calcd. for $C_{10}H_{12}N_2O\cdot C_6H_3N_3O_7\cdot H_2O$: C, 45.40; H, 4.05; N, 16.55. Found: C, 45.20; H, 3.94; N, 16.62). The absorption spectrum of 5-hydroxytryptamine in aqueous solution at *p*H 5.4 has a maximum at 2750 Å., a shoulder with a point of inflection at 2990 Å., and a minimum at 2500 Å. At *p*H 11.6, the position of the maximum at 2750 Å. is essentially unchanged while the second peak shifts from 2990 to 3220 Å. The data on the picrate and the absorption data on the hydrochloride are in excellent agreement with that published by Rapport[2] for the vasoconstrictor principle, serotonin.

Preliminary pharmacological investigation has shown 5-hydroxytryptamine to have vasoconstrictive properties.

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RECEIVED AUGUST 16, 1951

NINE OR MORE LIQUID PHASES

Sir:

The discovery of an ever-increasing number of incompletely miscible liquid phases has furnished an interesting challenge to physical chemists, not without value in that it has served to direct attention to the variety of factors which can contribute to immiscibility. In 1934 I exhibited a system of five stable liquid phases; in 1940 a sixth was added, and in 1949 a seventh.[1] In 1950 Kittsley and Goeden[2] added to the former set an eighth, a silicone oil; this owes its low solubility in the other liquids to its large cross-linked molecules. I wish now to point out that the "incompatibility" of different high polymers may be invoked to split a liquid in which they are soluble into two or even more liquid layers. Dobry and Boyer-Kawenoki[3] have made an experimental study of a number of such systems and Stockmayer[4] and Scott[5] have given their theoretical interpretation.

For example, the water layer of the previous set can be split into two by using two incompatibles reported by Dobry and Boyer-Kawenoki, methyl cellulose and polyvinyl alcohol, yielding nine layers and there seems no reason to doubt that the same principle could be applied to yield almost unlimited further splitting of any layer for which sufficiently soluble high polymers of different molecular weights and configurations can be found.

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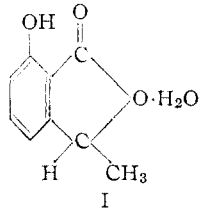
A DEGRADATION PRODUCT OF TERRAMYCIN

Sir:

The hydrolysis of the antibiotic terramycin, $C_{22}H_{24-26}N_2O_9$, in hot 20% sodium hydroxide in the presence of zinc has been previously reported[1] to yield terracinoic acid ($C_{13}H_{12}O_6$), ammonia, dimethylamine, acetic acid, carbon dioxide, and a phenolic lactone, $C_9H_8O_3\cdot H_2O$, m.p. 110–112°. *Anal.* Calcd. for $C_9H_8O_3\cdot H_2O$: C, 59.33; H, 5.54; H_2O, 9.89. Found: C, 59.32; H, 5.79; H_2O (K.F.) 9.30. The structure of the phenolic lactone has been shown by degradation and by synthesis to be 7-hydroxy-3-methylphthalide (I).



I

This phthalide is insoluble in bicarbonate and slowly soluble in cold aqueous sodium hydroxide. It gives a purple color with ferric chloride and a positive aminoantipyrine test. Titration of anhydrous I at room temperature shows it to be a monobasic acid with *pK* 8.5 and equivalent weight 162 (calcd. 164). A drop in *p*H occurs when the titrated solution is heated to 100° for one hour indicating the presence of a lactone. In hot sodium ethoxide, I yields an alcohol insoluble crystalline disodium salt of the free acid. *Anal.* Calcd. for $C_9H_8O_4Na_2\cdot H_2O$: C, 44.30; H, 4.12; Na, 18.79. Found: C, 44.00; H, 4.34; Na, 18.45.

Methylation with diazomethane yields a monomethyl ether, m.p. 73–74°, which is very slowly soluble in cold alkali. *Anal.* Calcd. for $C_{10}H_{10}O_3$: C, 67.46; H, 5.76; methoxyl, 17.41. Found: C, 67.45; H, 5.65; methoxyl, 18.0. This methyl ether forms a crystalline alcohol soluble monosodium salt.

Oxidation of the methyl ether of I by potassium permanganate in strongly alkaline solution yields a small amount of 3-methoxyphthalic acid, which has been identified as its anhydride. Fusion of 7-hydroxy-3-methylphthalide (I) with alkali yields salicylic acid, m.p. 159–60°, and acetic acid, identified through its *p*-nitrobenzyl ester, m.p. 77–78°. Cleavage of the aromatic ring-to-carbon bond to yield benzoic acids and aliphatic acids is characteristic of 3-monoalkylated phthalides. This cleavage suggests that the phenolic hydroxyl of I is in the 7 position. This assignment was also favored

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