in the condensation of dicyclohexylamine with ethylene oxide. When Gilman and Clark[7] could not condense isopropyllithium with tri-isopropylsilane, they attributed this to the sterically hindered nature of the isopropyl group.

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[7] Gilman and Clark, *ibid.*, **69**, 1499 (1947).

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4-*n*-Butyl-2,6-di-*t*-butylphenol

Following the method of Stevens,[1] isobutylene was bubbled into 9.2 g. of 4-*n*-butylphenol[2] containing 0.25 ml. of concentrated sulfuric acid until the gain in weight of the reaction mixture showed that slightly more than the theoretical amount (6.9 g.) had been added, then the excess isobutylene was swept out with natural gas. The reaction mixture was washed free from acid with successive 5% sodium carbonate washes, dried by adding benzene and distilling, and the product vacuum distilled. The main fraction of 11 g. (68%) boiled at 154–157° (10.5 mm.), and on refractionation gave a clear, colorless, rather viscous product, b. p. 144–144.5° (6 mm.), n^{20}_{D} 1.5019, d^{20}_{4} 0.920.

Anal. Calcd. for $C_{18}H_{30}O$: C, 82.38; H, 11.52. Found: C, 82.30; H, 11.52.

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[1] D. R. Stevens, *Ind. Eng. Chem.*, **35**, 655–660 (1943).

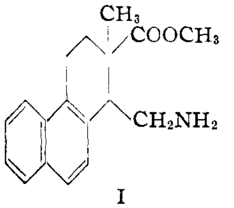
[2] R. V. Rice and W. C. Harden, *J. Am. Pharm. Assoc.*, **25**, 7–9 (1936).

COMMUNICATIONS TO THE EDITOR

THE LACTAMS OF *cis*- AND *trans*-1-AMINOMETHYL-2-CARBOMETHOXY-2-METHYL-1,2,3,4-TETRAHYDROPHENANTHRENE

Sir:

In order to secure more information concerning the configuration of the steroids at the C/D ring juncture, we have prepared the diastereoisomeric (*cis* and *trans*) amino esters (I) which correspond in configuration to desoxyequilenin and desoxyisoequilenin and have studied their tendency to form γ-lactams (II). It was hoped that their behavior in this respect would indicate which amino ester had the *cis* and which the *trans* configuration. It was found that both amino esters yielded lactams, one of which must be the *cis* lactam and the other the *trans* lactam. However, the lactam from the amino ester related to desoxyisoequilenin formed more rapidly than the lactam from the amino ester related to desoxyequilenin. Thus, when an aqueous solution of the amine ester hydrochloride corresponding to desoxyequilenin was treated with one equivalent of alkali and the liberated product was extracted immediately into ether (total time, ten minutes), only the free amino ester was formed. Under identical conditions the amino ester corresponding to desoxyisoequilenin gave a 60% yield of the γ-lactam (II) (m. p. 205–206°. *Anal.* Calcd. for $C_{17}H_{17}NO$: C, 81.24; H, 6.77; N, 5.58. Found: C, 81.11; H, 6.86; N, 5.46). The lactam (m. p. 234–236°. *Anal.* Found: C, 81.20; H, 6.83; N, 5.33) of the desoxyequilenin series was obtained when an excess of alkali was employed and the ether solution of the amino ester was allowed to stand for a longer period of time.



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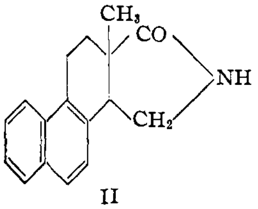

II

The more rapid formation of the lactam from the amino ester corresponding to desoxyisoequilenin may be indicative of the *cis* configuration which is currently assigned to desoxyisoequilenin. Further evidence is being sought in experiments in progress on the preparation of the corresponding 2-methyl-1,2,3,4-tetrahydrophenanthrene-1,2-dicarboxylic acids and a study of their ability to form anhydrides.

The amino esters were prepared by Curtius degradation of the acetic acid side chain of the two diastereoisomeric (*cis* and *trans*) 2-carbomethoxy-2-methyl-1,2,3,4-tetrahydrophenanthrene-1-acetic acids.[1] The degradation was accomplished by treatment of the acid chloride with sodium azide,[2] followed by rearrangement of the resulting azide to the isocyanate, which was hydrolyzed by concentrated hydrochloric acid to the amine ester hydrochloride in good yield; m. p.: normal (desoxyequilenin) form, 241–242°; iso form, 212–213°. *Anal.* Calcd. for $C_{18}H_{22}ClNO_2$:

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[1] Bachmann and Wilds, THIS JOURNAL, **62**, 2084 (1940). The α acid has been shown to have the configuration of desoxyisoequilenin; the β acid corresponds to desoxyequilenin. The results of these experiments will be published soon.

[2] After our work had been completed, Billeter and Miescher, *Helv. Chim. Acta*, **31**, 1302 (1948), reported that the acid chloride of the 7-methoxy derivative of the acid did not react with sodium azide.