Laboratory) 0.01 M in redistilled CH_2Cl_2 or $CHCl_3$ was added iodine in equimolar quantity. Six solutions thus prepared were illuminated through 10 cm. of water by high efficiency incandescent lamps. After three hours, 90% of the iodine disappeared in CH_2Cl_2 at -60 to $-90°$ and 98% disappered in $CHCl_3$ at -60 to $-70°$. Iodine did not react photochemically with either solvent. Three such solutions of photoiodinated butene were illuminated in quartz by a spark between high tungsten steel electrodes. Each evolved its iodine quantitatively within an hour as proved by titration with 0.01 N thiosulfate in three stages. Another kept at 20° for one hundred hours had already evolved 84% of the original iodine, also in stages. The cycle involving visible and ultraviolet light was repeated several times in a fresh sample. Six preliminary photolyses of less pure material had yielded similar results; one reverse thermal reaction was practically complete in four days. Experiments on quantum yields at various temperatures and wave-lengths, and the thermal reaction rates (Polissar, THIS JOURNAL, **52,** 956 (1930)) are being undertaken, using butene-1 and related compounds.

HARVARD UNIVERSITY GEORGE S. FORBES
CAMBRIDGE, MASSACHUSETTS ARTHUR F. NELSON
RECEIVED DECEMBER 21, 1935

ε-CAPROLACTONE

Sir:

Stoll and Rouvé [*Helv. Chim. Acta* **18,** 1087 (1935)] have recently reported the isolation of pure ε-hydroxycaproic acid and its conversion to the corresponding lactone by application of the high dilution technique. The same lactone was obtained by us previously [Van Natta, Hill and Carothers, THIS JOURNAL, **56,** 455 (1934)] from a sample of the acid admitted to be impure. Under the conditions described we concluded that the lactone is the principal primary product of the self-esterification of ε-hydroxycaproic acid. Stoll and Rouvé conclude that heating of the pure acid yields chiefly polyesters and very little if any of the monomeric lactone.

We do not doubt the correctness of their conclusions, but we maintain also that the results of our own experiments need not be doubted either. Stoll and Rouvé state (footnote, p. 1091) that since we obtained the lactone by an impossible method its identity may be doubted in spite of the correct physical constants, especially since it was not identified by means of known derivatives. As to the impossibility of the method used, our description is an exact record of an actual experiment and it is incomplete in only one point of detail that seems of any likely significance. Our acid was obtained after ether-extraction mixed with water and acetic acid. To remove these impurities under the mildest possible conditions the mixture was allowed to stand for four days (time not given in the text of our paper) at room temperature while connected directly to an evacuated receiver kept at $-80°$. It is quite likely that a large portion of the observed lactonization occurred under these conditions (titration indicated 20% lactone assuming that *all* the acetic acid had been removed). In any event it is evident that our conditions of temperature, catalysis and possible dilution were such that they might easily yield a result different from that reported by Stoll and Rouvé. We have already (*loc. cit.*) pointed out that the ε-hydroxy acid is a transition case where the choice between intra- and intermolecular reaction may be expected to be especially sensitive to experimental conditions.

As to the identity of our lactone, we *did* prepare and describe a derivative. The same crystalline hydrazide was obtained from: (1) the crude acid, (2) the monomeric lactone, (3) the dimeric lactone, (4) the polyester and (5) the (crude) ethyl ester of the hydroxy acid made by an independent method. Mixed melting points showed no depressions.

EXPERIMENTAL STATION F. J. VAN NATTA
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