carbonate. The precipitated strontium carbonate was removed by filtration and the solution containing the sodium salt concentrated to a glass under reduced pressure. The glass was dissolved in 4 ml. of hot water, and 429 mg. of *p*-phenylphenacyl bromide and 8 ml. of ethanol were added. Although some precipitation occurred, this offered no difficulty as it was readily apparent that the character of the solid changed during the ensuing 2-hour refluxing. The mixture was allowed to cool to room temperature and filtered, yielding 0.5 g. of crude ester which after 2 recrystallizations from 40 parts of 1:1 ethanol–acetone melted at 145.5–147.5° with no depression on admixture with the ester prepared similarly by Stewart and Richtmyer from 1,6-anhydro-β-D-gulopyranose.[2]

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Decomposition of Quaternary Ammonium Salts. IV. Methyl Ketones

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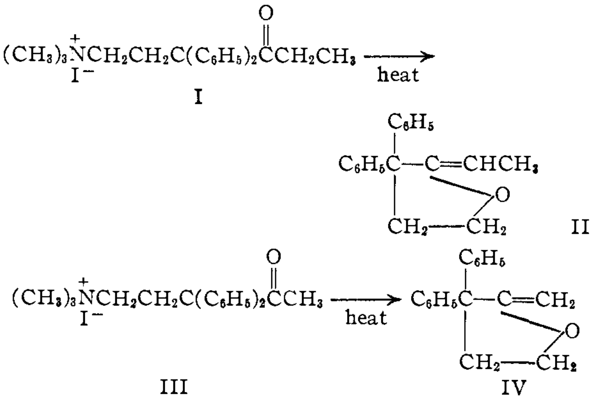
Although the decomposition of quaternary salts of the methadone (I) type to give ethylidenetetrahydrofurans[2,3] has been reported, only ethyl ketones were mentioned. This paper deals with the product obtained from the pyrolysis of the quaternary salt of a methyl ketone, 5-dimethylamino-3,3-diphenyl-2-pentanone (III). Since the product from this reaction was a liquid and all of those reported for the ethyl ketones were solids, it was felt that the reaction might not be analogous. Subsequent reactions, however, substantiate the similarity of these decompositions. The oil decolorizes bromine readily and forms adducts with hydroxylamine and 2,4-dinitrophenylhydrazine to give a product analogous to those reported[2,3] for the ethylidenetetrahydrofurans. The methylidenetetrahydrofuran (IV) gave benzophenone on chromic oxide oxidation.


I II III IV

Experimental

5-Dimethylamino-3,3-diphenyl-2-pentanone.—This compound was prepared according to procedures previously described.[4]

5-Dimethylamino-3,3-diphenyl-2-pentanone Oxalate.—This derivative was prepared by the addition of an excess of oxalic acid to a solution of the ketone in absolute alcohol. Upon the addition of ether the crude product, melting at 160–162°, precipitated. After several recrystallizations it melted at 163.0–163.5°.

Anal. Calcd. for $C_{19}H_{23}ON \cdot C_2H_2O_4$: C, 67.90; H, 6.79; N, 3.77. Found: C, 67.88; H, 6.92; N, 3.73, 3.76.

5-Dimethylamino-3,3-diphenyl-2-pentanonemethiodide.—An excess of methyl iodide was added to a solution of the ketone in absolute alcohol. Upon the addition of ether, an oil separated which crystallized with cooling and scratching. The solid melted at 144–148°, and after repeated crystallization from alcohol and ether melted at 151.5–152°.

Anal. Calcd. for $C_{20}H_{26}ONI$: C, 56.74; H, 6.19; N, 3.32. Found: C, 56.65; H, 6.36; N, 3.26.

Pyrolysis of 5-Dimethylamino-3,3-diphenyl-2-pentanonemethiodide.—In a 50-ml. distilling flask equipped with a receiver and thermometer there was placed 23 g. of the methiodide III. The system was evacuated to 13 mm. and the flask heated gently with a free flame. The solid melted, and in the course of about 15 minutes 8.7 g. of viscous liquid distilled at about 190°. A second fraction weighing 2.2 g. was distilled at 205–270° (13 mm.). A tarry residue remained in the flask. These distillates darkened upon standing.

The viscous liquids obtained above were treated with benzene and a small amount of white solid was filtered from each. The benzene filtrates were extracted with 5% hydrochloric acid, washed with water, and redistilled. The first fraction distilled at 188–192° (12 mm.), and weighed 7.4 g. The second fraction distilled at 195–203° (16 mm.).

Upon standing overnight in an ice-bath, a few tenths of a gram of a white solid separated from the second fraction. After recrystallization from petroleum ether and sublimation under vacuum it melted at 75–78°. It was not investigated further. The remainder of fraction 2 was shown to be the same as fraction one by the preparation of the derivative with 2,4-dinitrophenylhydrazine.

The oil obtained did not react with potassium permanganate in acetone except on long standing, but reacted rapidly with bromine in carbon tetrachloride at ice temperature, with the evolution of hydrogen bromide.

Anal. Calcd. for $C_{17}H_{16}O$: C, 86.40; H, 6.84. Found: C, 86.26; H, 6.90.

Reactions of Pyrolysis Product of III. (a) With Hydroxylamine.—This reaction was run in the usual manner.[2] After several recrystallizations from alcohol the product melted at 168–170.5°, and after drying in the Abderhalden apparatus at 56° for 2 hours, melted at 171–172°.

Anal. Calcd. for $C_{17}H_{19}O_2N$: C, 75.80; H, 7.11; N, 5.20. Found: C, 75.94, 75.83; H, 6.93, 7.03; N, 5.20, 5.24.

(b) **With 2,4-Dinitrophenylhydrazine.**—This reaction was run in the usual way. The product was a finely divided, bright yellow powder. After several recrystallizations from benzene and petroleum ether it melted at 194.0–194.5° dec. This derivative was unstable and difficult to purify.

Anal. Calcd. for $C_{23}H_{22}O_5N_4$: C, 63.58; H, 5.11; N, 12.90. Found: C, 63.96, 64.11; H, 5.12, 5.28; N, 12.72, 12.75.

(c) **Oxidation with Chromic Oxide.**—One gram of the oil was oxidized with 5 g. of chromic oxide in glacial acetic acid. From this, a neutral oil was obtained which formed a semicarbazone melting at 163–166°. Its melting point was not depressed by the addition of an authentic sample of benzophenone semicarbazone.

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