tassium iodide solution. The orange-red, liquid product of the decomposition was purified by distillation. One fraction, amounting to a yield of about 20%, had b.p. 80° (17 mm.), $n^{20}\text{D}$ 1.610 and d^{20}_4 1.23. These properties when compared with those previously reported[3] identify the compound as benzenesulfenyl chloride.

Hydrolysis of Sulfur Trichlorides.—When added to a cold sodium bicarbonate solution ethylsulfur trichloride formed sodium ethanesulfinate which reacted on boiling with benzyl chloride to form benzyl ethyl sulfone. The latter melted at 84° and the melting point was unchanged when the sample was mixed with benzyl ethyl sulfone prepared by the oxidation of benzyl ethyl sulfide.

Unsuccessful Attempts to Prepare Sulfur Trichlorides.—Benzyl disulfide (5 g.) was chlorinated at −40°, but no precipitate formed. The reaction mixture was distilled under reduced pressure and there was obtained 2 g. of liquid with the properties: b.p. 89° (37 mm.), $n^{20}\text{D}$ 1.540, d^{20}_4 1.11, which identify the product as benzyl chloride.

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The Structure of Isoamidone. I

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While carrying out some work on 4,4-diphenyl-6-dimethylamino-3-hexanone we had occasion to prepare several derivatives. The melting points of these compounds agreed very closely with those reported[2] for Isoamidone I, and the melting points of the hydrochloride of this substance, when taken in the same melting point apparatus was the same as that of the hydrochloride of 4,4-diphenyl-6-dimethylamino-3-hexanone. No depression in melting points was observed on mixing.

These data prove that Isoamidone I is 4,4-diphenyl-6-dimethylamino-3-hexanone. A logical explanation for the finding of this compound in the mother liquors from the synthesis of Amidone is that the propylene chlorohydrin, which was used in the preparation of the dimethylaminochloropropane, was contaminated with ethylene chlorohydrin.

Experimental

The 6-dimethylamino-4,4-diphenyl-3-hexanone was prepared by the procedure[3] of Dupre, *et al.*, in which 4-bromo-2,2-diphenylbutanenitrile is aminated with dimethylamine and then the ketone is prepared by the usual Grignard reaction. The ketone was usually isolated as the hydrobromide and then converted to the desired derivative.

Preparation of Derivatives of 6-Dimethylamino-4,4-diphenyl-3-hexanone.—The hydrobromide was dissolved in warm water and the solution was made basic with sodium hydroxide. The oil was extracted with ether and the ether was dried over magnesium sulfate and the solvent distilled. Concentrated hydrochloric acid and ethanol were added. The mixture was concentrated to dryness and crystallized on scratching. It was recrystallized from acetone and melted at 173.5–175°,[4] not depressed on an admixture of Isoamidone I hydrochloride of m.p. 173.5–175°.

Anal. Calcd. for $C_{20}H_{26}NOCl$: Cl, 10.68. Found: Cl, 10.92.

Methiodide (from absolute ethanol), m.p. 199.5–200.5°; reported for Isoamidone I methiodide[2] 195–196°.

Anal. Calcd. for $C_{21}H_{28}NOI$: N, 3.20. Found: N, 3.17, 3.10.

Oxalate (from absolute ethanol), m.p. 161.5–163°; reported for Isoamidone I oxalate,[2] 158–160°.

Anal. Calcd. for $C_{20}H_{25}NO \cdot C_2H_2O_4$: N, 3.65. Found: N, 3.60.

Picrate (from ethanol) m.p. 134–136°; reported for Isoamidone I picrate,[2] 131–133°.

Anal. Calcd. for $C_{26}H_{28}N_4O_8$: N, 10.68. Found: N, 10.48, 10.52.

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[1] Abstracted in part from the thesis submitted in partial fulfillment of the requirements for the degree of doctor of philosophy.

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The Rate of the Reaction between Thallium(III) and Iron(II) in Perchloric Acid Solution

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While preparing a paper describing our work[2] on the kinetics of the Fe(II)–Tl(III) reaction, the article by Johnson[3] on the same subject appeared. We are therefore limiting our report to results which amplify or supplement those reported by him. Measurements made under the same conditions agree quantitatively.

Experimental

The preparation of the thallous, thallic, ferrous and ferric perchlorates has been previously described.[4] The perchloric acid was G. F. Smith Chemical Company's 70% "purified." Spot tests as recommended by Feigl[5] showed that this acid had a ferric ion content of less than $3 \times 10^{-6}F$ and a chloride ion concentration of less than 3×10^{-5}. No chlorate could be detected. The ferrous perchlorate stock solution contained traces of chloride ion (for quantitative information see reference 4), but not in sufficient amounts to affect the results of this investigation. Sodium and lithium perchlorates were prepared by adding the reagent grade carbonate to perchloric acid. Basic magnesium carbonate was used in the same manner to prepare a magnesium perchlorate solution. Manganese perchlorate solutions were prepared by dissolving the reagent grade metal in perchloric acid. These solutions all contained less than the above limits of iron and chloride ion. All other chemicals used were Baker and Adamson reagent grade.

The reactants, in separate glass-stoppered bottles, were allowed to come to uniform temperature in a constant temperature bath. The order of addition did not affect the results. All experiments were carried out at 25 ± 0.1°. The time of starting a run was taken at the time when one half of the second reagent had been added to the first. Samples for analysis were taken with a pipet and discharged into an acidic chromate solution which stopped the reaction by oxidizing iron(II) and precipitating thallium(I). The acidic precipitate was metathesized into the stoichiometric thallous chromate by a previously described method.[4]

Although chloride ion has been mentioned as a catalyst for the dichromate oxidation of thallium(I),[6] tests showed

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