product. The combined ether and ethanol extracts were dried over anhydrous potassium carbonate and then evaporated under reduced pressure until the nearly colorless product began to crystallize. The crude product was recrystallized from benzene to give 41.3 g. (68%), m.p. 135–138°.

Anal. Calcd. for $C_8H_{13}N_3$: C, 63.53; H, 8.66; N, 27.79. Found: C, 63.45; H, 8.71; N, 27.54.

Acylation of 2-Amino-3-aminomethyl-4,6-dimethylpyridine (IV).—These derivatives were prepared by conventional methods.

The action of acetic anhydride on IV gave 2-amino-3-acetamidomethyl-4,6-dimethylpyridine in 92% yield, m.p. 185–187°.

Anal. Calcd. for $C_{10}H_{15}N_3O$: C, 62.15; H, 7.82; N, 21.71. Found: C, 62.36; H, 7.89; N, 21.68.

2-Amino-3-benzamidomethyl-4,6-dimethylpyridine was obtained from IV by the action of benzoyl chloride in pyridine solution; m.p. 156–158°. The yield was 83%.

Anal. Calcd. for $C_{15}H_{17}N_3O$: C, 70.56; H, 6.71; N, 16.46. Found: C, 70.69; H, 7.03; N, 16.40.

The formate salt of IV (from equivalent amounts of 99% formic acid and IV), m.p. 172° dec., gave crude 2-amino-3-formamidomethyl-4,6-dimethylpyridine when heated in toluene solution until the calculated amount of water had been eliminated. Recrystallization from ethanol gave the analytically pure amide melting at 182–184° dec.

Anal. Calcd. for $C_9H_{13}N_3O$: C, 60.32; H, 7.31; N, 23.45. Found: C, 60.46; H, 7.41; N, 23.20.

2-Amino-3-carbethoxyaminomethyl-4,6-dimethylpyridine was obtained in 86% yield when IV and ethyl chlorocarbonate were allowed to stand at room temperature for 1 hour; m.p. 116–118°.

Anal. Calcd. for $C_{11}H_{17}N_3O_2$: C, 59.16; H, 7.68; N, 18.82. Found: C, 59.30; H, 7.82; N, 18.82.

2,5,7-Trimethyl-3,4-dihydropyrido[2,3-d]pyrimidine.—A mixture of 2-amino-3-acetamidomethyl-4,6-dimethylpyridine (14.5 g., 0.075 mole) and phosphorus oxychloride (100 ml.) was allowed to stand at room temperature for 1 hour. The solid dissolved with warming during this time. The excess phosphorus oxychloride was removed under reduced pressure and the residue poured onto ice. The solution was rendered basic by the addition of dilute ammonium hydroxide and the white solid which separated was collected by filtration. The crude material was recrystallized from benzene to give 9.0 g. (69%), m.p. 203° dec.

Anal. Calcd. for $C_{10}H_{13}N_3$: C, 68.54; H, 7.48; N, 23.98. Found: C, 68.49; H, 7.48; N, 23.83.

5,7-Dimethyl-2-phenyl-3,4-dihydropyrido[2,3-d]pyrimidine.—To a mixture of phosphorus pentachloride (5.0 g.) and phosphorus oxychloride (25 ml.) was added 2-amino-3-benzamidomethyl-4,6-dimethylpyridine (4.0 g., 0.0157 mole). The solution which formed was heated under reflux for 30 minutes, then cooled and poured slowly onto ice. The acidic solution was treated with activated carbon, filtered and then made basic by the addition of excess sodium carbonate solution. The crude yellow solid which separated was removed by filtration, dried and recrystallized from 95% ethanol to give 2 g. (61%) of pale yellow product melting at 190–193° dec.

Anal. Calcd. for $C_{15}H_{15}N_3$: C, 75.92; H, 6.37; N, 17.71. Found: C, 75.86; H, 6.50; N, 17.66.

5,7-Dimethyl-3,4-dihydropyrido[2,3-d]pyrimidine.—A mixture of 2-amino-3-aminomethyl-4,6-dimethylpyridine formate (5.0 g., 0.0254 mole) and xylene (75 ml.) was placed in a flask equipped with a water-trap and a condenser. The flask was heated by means of an oil-bath so that reflux was maintained. After 1.5 hours, the theoretical amount of water had been collected in the trap and the solid had dissolved to give a pale yellow solution. Cooling and addition of petroleum ether (30–60°) yielded 2.87 g. (70%) of slightly yellow product. Recrystallization from chloroform gave a product melting at 203° dec.

Anal. Calcd. for $C_9H_{11}N_3$: C, 67.05; H, 6.88; N, 26.07. Found: C, 66.99; H, 6.92; N, 25.98.

5,7-Dimethyl-3,4-dihydropyrido[2,3-d]pyrimid-2(1)-one.—A solution of 2-amino-3-carbethoxyaminomethyl-4,6-dimethylpyridine (28.7 g., 0.128 mole) in diphenyl ether (200 ml.) was heated under reflux with brisk stirring for 70 minutes. The solution was cooled and, after the addition of 200 ml. of petroleum ether (30–60°), the tan solid which had separated was removed by filtration. The product (21.3 g., 94%) was purified by recrystallization from cellosolve to give white crystals, m.p. 271–274° dec.

Anal. Calcd. for $C_9H_{11}N_3O$: C, 61.00; H, 6.26; N, 23.71. Found: C, 61.05; H, 6.51; N, 23.79.

LINCOLN 8, NEBRASKA

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY OF WAYNE UNIVERSITY]

Nitrogen Analogs of Ketenes. A New Method of Preparation[1]

BY CALVIN L. STEVENS AND JAMES C. FRENCH[2]

RECEIVED SEPTEMBER 19, 1952

A novel synthesis of a nitrogen analog of a ketene (I) from the reaction of an α-choloroimino chloride (II) with sodium iodide in 84% yield is described. Some chemical and physical properties of I are also included.

The present paper is an initial report of an investigation of the chemistry of the nitrogen analogs of ketenes. The investigation resulted in the discovery of a novel method for the preparation of a ketenimine (I) in good yield by the reaction of an α-chloroimino chloride (II) with sodium iodide. The ketenimine (I) reacted smoothly with water, methanol and chlorine to give the amide (III), iminoester (IV) and α-chloroimino chloride (II) in good yield.

In 1921, Staudinger[3] prepared several ketenimines in unspecified yields by the interaction of a phosphinimine (VIII) with a ketene. The phosphinimines were prepared from the unstable phosphazides (VII) which were made from azides (VI) and phosphines (V). Staudinger characterized the ketenimines by reaction with water to give amides. In an attempt to prepare ketenimines, Staudinger[3,4] reported that zinc failed to dehalogenate an α-chloroimino chloride.

$$\underset{\text{V}}{R_3P} + \underset{\text{VI}}{R'N_3} \longrightarrow \underset{\text{VII}}{R_3P{=}N{-}N{=}N{-}R'} \xrightarrow[-N_2]{} \underset{\text{VIII}}{R_3P{=}N{-}R'}$$

$$\text{VIII} + R''_2C{=}C{=}O \longrightarrow R''_2C{=}C{=}N{-}R' + R_3PO$$

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[1] Presented before the Organic Division at the 121st Meeting of the American Chemical Society, Buffalo, N. Y., March, 1952.

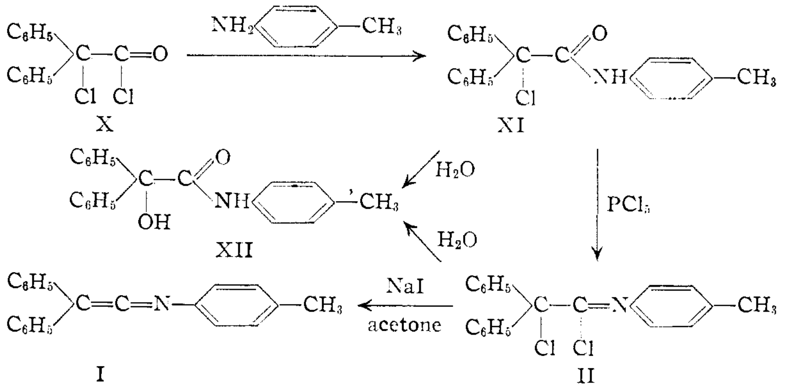
[2] Parke, Davis and Co. Fellow, 1951–1952.

[3] H. Staudinger and E. Hauser, *Helv. Chim. Acta*, **4**, 887 (1921); see also H. Staudinger and J. Meyer, *Ber.*, **53**, 72 (1920).

[4] H. Staudinger, *Ann.*, **356**, 55 (1907).

Bergmann[5] in 1930, and Dilthey[6] in 1940 reported the isolation of ketenimine-type compounds. However, later work has shown that the structures assigned to the compounds were incorrect.

The ketenimine, diphenylketene-*p*-tolylimine (I), was prepared by the sequence of reactions X → XI → II → I.

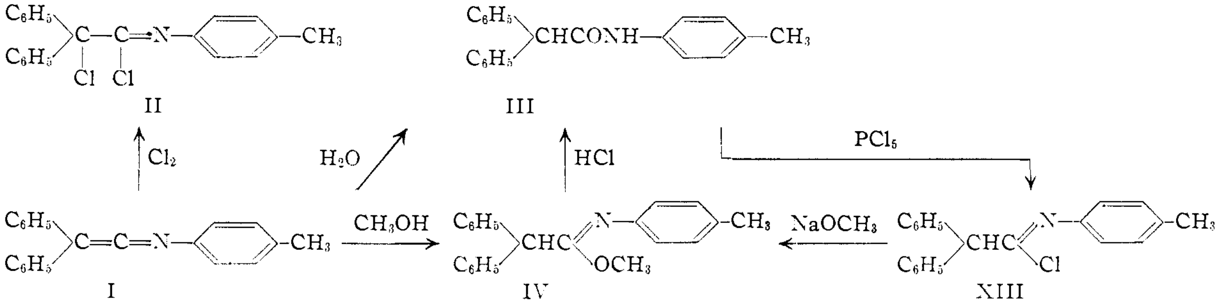


α-Chlorodiphenylacetyl chloride (X) was prepared from benzilic acid and phosphorus pentachloride in 87% yield by a modification of a previously described procedure.[7] Treatment of α-chlorodiphenylacetyl chloride (X) with two equivalents of *p*-toluidine gave pure N-(*p*-tolyl)-α-chlorodiphenylacetamide (XI) in 81% yield. This α-chloroamide (XI) could be hydrolyzed to the α-hydroxyamide (XII) in 94% yield. Further, Billman[8] has proved that X reacts with amines to give α-chloroamides and not α-aminoacid chlorides. When the α-chloroamide (XI) was allowed to react with phosphorus pentachloride, hydrogen chloride was smoothly evolved and an 87% yield of crystalline α-chlorodiphenyl-N-(*p*-tolyl)-iminoacetyl chloride (II) was obtained. This iminochloride (II) was characterized by hydrolysis to the α-hydroxyamide (XII) in 84% yield.

The α-chloroimino chloride (II) could be easily dehalogenated to the yellow crystalline ketenimine (I) when treated with an acetone solution of sodium iodide. The ketenimine with a two-degree melting point range was formed in this dehalogenation step in 83.5% yield. From the readily available benzilic acid the over-all yield was 50%. This method of introducing a double bond into a reactive molecule may be applicable to the preparation of the parent ketene as well as other ketenimines.

The ketenimine was characterized by carbon and hydrogen analysis and a molecular weight determination indicated that the material was not dimeric. An infrared spectrum (Fig. 1) of the ketenimine (I) showed a strong absorption band at 5.0 which corresponds to an absorption at 5.0–5.2 μ which has been associated with the C=C=C group in allenic compounds.[9] The ultraviolet absorption spectrum (Fig. 2) has maximum absorption at 268.5 mμ (log ε 4.48) and 357 mμ (log ε 3.14).

In addition to the analytical and physical evidence, chemical evidence for the structure of the ketenimine was obtained by reactions with water, alcohol and chlorine.



Although an acetone solution of diphenylketene-*p*-tolylimine (I) reacted only slowly with water, hydrolysis was complete in a few seconds by the addition of a small amount of hydrochloric acid to give N-(*p*-tolyl)-diphenylacetamide (III) in 86% yield. The amide (III) was prepared independently from diphenylacetyl chloride and *p*-toluidine. The completion of the hydrolysis was indicated by the discharge of the yellow color of the solution.

A 45-minute reflux period was necessary for the complete decolorization of the yellow solution of the ketenimine (I) in anhydrous methanol containing a small amount of sodium methoxide. From this reaction, methyl diphenyl-N-(*p*-tolyl)-iminoacetate (IV) was isolated in 88% yield. Evidence for the structure assigned to IV was provided by the facile conversion of IV to the amide (III) in

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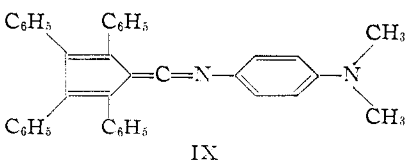
(5) E. Bergmann and H. Weiss (*Ber.*, **63**, 1176 (1930)) reported the formation of ketene-N-triphenylmethylimine as a product from the condensation of triphenylcarbinol and cyanoacetic acid but the following year Fosse (*Bull. soc. chim.*, [4] **49**, 171 (1931)) showed that the compound was N-triphenylmethylacetamide.

(6) W. Dilthey and P. Huchtemann (*J. prakt. Chem.*, **154**, 238 (1940)) reported the formation of *p*-dimethylaminophenylimino-2,3,4,5-tetraphenylfulvene (IX) by the condensation of *p*-nitrosodimethylaniline and 2,3,4,5-tetraphenylfulvene.



IX

A recent communication from E. I. Becker, Polytechnic Institute of Brooklyn, indicated that the structure assigned (IX) is in error and that the compound is the Schiff base of *p*-dimethylaminoaniline and tetraphenylcyclone.

(7) J. H. Billman and P. H. Hidy, THIS JOURNAL, **65**, 760 (1943).

(8) J. H. Billman, *et al.*, *ibid.*, **67**, 130 (1945).

(9) J. H. Wotiz, *ibid.*, **73**, 693 (1951); A. T. Blomquist, *et al.*, *ibid.*, **74**, 3636 (1952).

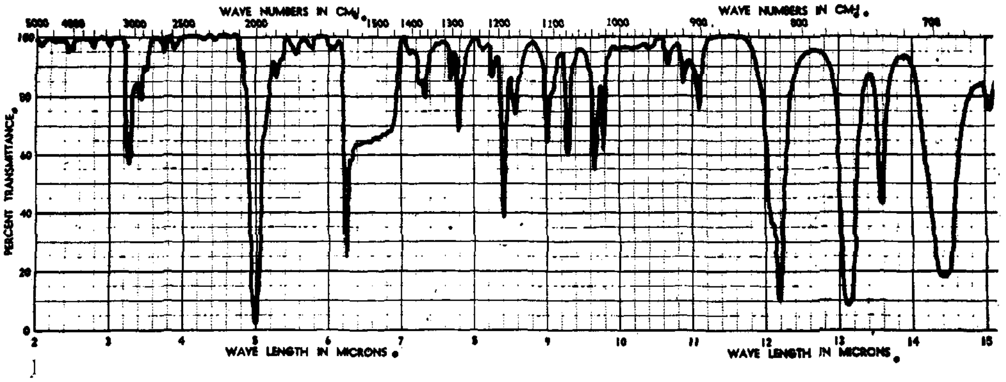


Fig. 1.—Infrared absorption spectrum of diphenylketene-*p*-tolylimine (I) in carbon disulfide (compensated).

52% yield by the action of dry hydrogen chloride.[10] Further, the iminoester (IV) was prepared independently by first allowing N-(*p*-tolyl)-diphenylacetamide (III) to react with one equivalent of phosphorus pentachloride in benzene to give the corresponding imino chloride, diphenyl-N-(*p*-tolyl)-iminoacetyl chloride (XIII). This imino chloride (XIII) was not isolated but was allowed to react directly with sodium methoxide in methanol to give a 19% yield of the crystalline imino ester, methyl diphenyl-N-(*p*-tolyl)-iminoacetate (IV), in addition to 37% of starting amide (III) recovered. Braun[11] reports that imino chlorides which have a hydrogen on the α-carbon atom are unstable and the low yield may be attributed to the decomposition of XIII.

Chlorine gas immediately decolorized a petroleum ether solution of the ketenimine to form α-chlorodiphenyl-N-(*p*-tolyl)-iminoacetyl chloride (II) in 86% yield. This α-chloroimino chloride (II) was identical with the sample prepared from the α-chloroamide (XI).

The reactions of I with water, alcohol and chlorine are analogous to the reactions of ketenes with these reagents, although compound I also can be considered a nitrogen analog of an allene as well as an N-substituted derivative of the "enol" form of a nitrile.[12]

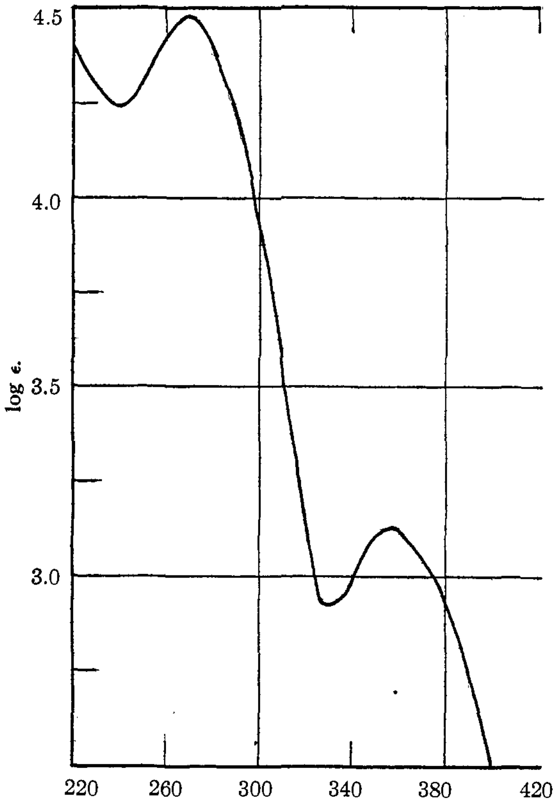


Fig. 2.—Ultraviolet absorption spectrum of diphenylketene-*p*-tolylimine (I) in ethanol. Courtesy of J. M. Vandenbelt, Parke, Davis and Co.

Experimental

α-Chlorodiphenylacetyl Chloride (X).—After the initial vigorous interaction of 40 g. (0.192 mole) of phosphorus pentachloride and 20 g. (0.088 mole) of benzilic acid had subsided, the resulting oil, protected from moisture by a calcium chloride tube, was heated for one hour on a steam-bath. Distillation under reduced pressure removed phosphorus oxychloride and gave 20.55 g. (87%) of α-chlorodiphenylacetyl chloride (X), b.p. 125–138° (0.5 mm.), m.p. 45–49°.

N-(*p*-Tolyl)-α-chlorodiphenylacetamide (XI).—To 8.1 g. (0.0756 mole) of *p*-toluidine in 125 ml. of ether was added over a period of five minutes 10.0 g. (0.0377 mole) of α-chlorodiphenylacetyl chloride in 100 ml. of ether. The reaction mixture was allowed to stand three hours at room temperature and then filtered. The precipitate was washed with ether, and the filtrate and combined ether washings were concentrated until a thick mass of crystals remained. The solid was filtered and washed with petroleum ether (b.p. 30–60°) to give 10.3 g. (81%) of the α-chloroamide (XI), m.p. 104–106°. A recrystallization from a petroleum ether–ether mixture gave needles, m.p. 105–106°.

Anal. Calcd. for $C_{21}H_{18}ClNO$: C, 75.10; H, 5.40; Cl, 10.56. Found: C, 74.84; H, 5.70; Cl, 10.46.

Hydrolysis of N-(*p*-tolyl)-α-chlorodiphenylacetamide (XI).—This amide (0.44 g.) was refluxed for 14 hours in 10 ml. of acetone and 5 ml. of water. To the hot, clear solution was then added 2 ml. of water and after the solution had crystallized, 0.394 g. (94%) of the crystalline N-(*p*-tolyl)-α-hydroxydiphenylacetamide (XII)[13] was obtained, m.p. 188–189°.

α-Chlorodiphenyl-N-(*p*-tolyl)-iminoacetyl Chloride (II).—To a solution of 15.0 g. (44.8 millimoles) of N-(*p*-tolyl)-α-chlorodiphenylacetamide in 15 ml. of dry benzene was added 9.3 g. (44.6 millimoles) of phosphorus pentachloride

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(10) S. M. McElvain and C. L. Stevens (THIS JOURNAL, **69**, 2667 (1947)) found that N-alkyl iminoesters were converted to the corresponding amides and alkyl chlorides by the action of dry hydrogen chloride.

(11) J. v. Braun, *et al.*, *Ann.*, **453**, 113 (1927).

(12) An interesting example of a reaction in which a nitrile reacted to give an "enol" derivative was reported in a private communication from Prof. H. J. Backer, University of Groningen, Holland, who found that certain negatively substituted acetonitriles gave N-methylketenimines when treated with diazomethane.

(13) H. Klinger (*Ann.*, **389**, 261 (1912)) reports 189–190°.

and the mixture refluxed for 90 minutes. The volatile components of the resulting clear yellow reaction mixture were removed *in vacuo* (0.3 mm.) and the last traces of remaining phosphorus oxychloride were removed by dissolving the oily residue in 50 ml. of dry benzene and again evaporating to dryness. The solid residue was transferred to a filter and washed with 50 ml. of dry petroleum ether (b.p. 30–60°) and recrystallized from 180 ml. of dry petroleum ether and 45 ml. of dry ether. Standing for two days at 5° gave 8.85 g. (56%) of hexagonal crystals, m.p. 88.5–90°. The material (1.4 g., m.p. 83–86°) obtained from concentrating and cooling the petroleum ether washings was combined with the mother liquor from the above recrystallization. The resulting solution was treated with charcoal, concentrated and allowed to stand three days at 5°, after which 4.92 g. (31%) of crystalline material was obtained which melted at 84–86°. Material of this purity could be used directly in the reaction with sodium iodide. The total yield was 87%.

An analytical sample was obtained from material melting at 88.5–90° by two recrystallizations from an ether–petroleum ether mixture, m.p. 92.5–94°.

Anal. Calcd. for $C_{21}H_{17}Cl_2N$: C, 71.19; H, 4.84; Cl, 20.0. Found: C, 71.31; H, 4.95; Cl, 20.0.

Hydrolysis of α-Chlorodiphenyl-N-(*p*-tolyl)-iminoacetyl Chloride (II).—A solution was prepared by dissolving 0.197 g. of the imino chloride (II) in 10 ml. of acetone and 2 ml. of water. After 30 hours, additional water was added and 0.145 g. (82%) of N-(*p*-tolyl)-α-hydroxydiphenylacetamide (XII) was precipitated, m.p. 188–190°. A mixed melting point with the product obtained from the hydrolysis of N-(*p*-tolyl)-α-chlorodiphenylacetamide (XI) was not depressed.

Preparation of Diphenylketene-*p*-tolylimine (I).—Ten grams (0.067 mole) of dry sodium iodide and 2.5 g. (0.0071 mole) of α-chlorodiphenyl-N-(*p*-tolyl)-iminoacetyl chloride (II), m.p. 88–90°, were dissolved in 50 ml. of dry acetone and the solution refluxed for 90 minutes. The deep red reaction mixture then was evaporated to dryness *in vacuo* and the residue transferred to a filter and washed with petroleum ether (b.p. 30–60°) until the washing was colorless (*ca.* 200 ml.). Further washing was detrimental since free iodine began to appear in the filtrate. The filtrate was shaken with cold, dilute sodium sulfite solution, dried over sodium sulfate and then evaporated to dryness *in vacuo*. The residue consisted of 1.67 g. (83.5%) of bright yellow solid, diphenylketene-*p*-tolylimine (I), m.p. 81–83°. Two recrystallizations from petroleum ether gave an analytical sample, m.p. 83–83.5°.

Anal. Calcd. for $C_{21}H_{17}N$: C, 89.01; H, 6.05; mol. wt., 283. Found: C, 89.32; H, 6.29; mol. wt., 290 (in benzene).

The reaction mixture obtained by refluxing 3.47 g. of less pure imino chloride (II), m.p. 84–86°, with 16 g. of sodium iodide in 80 ml. of dry acetone for 90 minutes was isolated in the same manner as described above to give 1.98 g. (71.5%) of diphenylketene-*p*-tolylimine (I), m.p. 75–79°. Recrystallization of this material from petroleum ether gave 1.43 g. of the yellow solid, m.p. 79–81° (51%).

Hydrolysis of Diphenylketene-*p*-tolylimine (I).—To a solution of 0.251 g. of N-(*p*-tolyl)-diphenylketenimine in 10 ml. of acetone were added three drops of water and two drops of concentrated hydrochloric acid. The yellow color was discharged within a few seconds and the clear solution was heated and water added dropwise until the solution became turbid. After the solution had cooled, 0.299 g. (86%) of N-(*p*-tolyl)-diphenylacetamide (III) was deposited as well-formed needles, m.p. 179.5–180.5°.

N-(*p*-Tolyl)-diphenylacetamide (III).—A mixture of 12 g. (0.056 mole) of diphenylacetic acid and 7.5 ml. (0.10 mole) of thionyl chloride was heated on a steam-bath for two hours. After removing the excess thionyl chloride under reduced pressure, an ether solution of the resulting partially crystalline mass was added to an ether solution of 6.5 g. (0.0608 mole) of *p*-toluidine and 8.5 ml. (0.064 mole) of collidine. The reaction mixture was allowed to stand overnight and then filtered. The ether filtrate was washed with dilute hydrochloric acid and evaporated to give only a very small amount of material. The precipitate was washed well with ether and then digested with 200 ml. of hot ethyl acetate. The insoluble collidine hydrochloride (6.3 g., 97%) was filtered and the filtrate concentrated until a white solid began to precipitate. After a few hours the solid was filtered and washed with petroleum ether (b.p. 30–60°). The yield of white crystalline material was 14.55 g. (85.5%), m.p. 171–173°. One recrystallization from aqueous acetone gave needles, m.p. 180–181°. N-(*p*-Tolyl)-diphenylacetamide was reported[14] to melt at 173°. For this reason, the material melting at 180° was analyzed. Calcd. for $C_{21}H_{19}NO$: C, 83.69; H, 6.36. Found: C, 83.57; H, 6.43. A mixed melting point of this material with the product obtained from the hydrolysis of N-(*p*-tolyl)-diphenylketenimine (V) gave no depression.

Methanolysis of Diphenylketene-*p*-tolylimine (I).—To a solution of 0.60 g. of this ketenimine in 6 ml. of dry ether was added 10 ml. of dry methanol in which was dissolved 0.07 g. of sodium metal. After refluxing for 45 minutes the reaction mixture was evaporated to dryness *in vacuo*. The nearly white residue was digested well with dry petroleum ether (b.p. 30–60°), treated with charcoal and filtered. The filtrate was concentrated to 15 ml. and allowed to stand overnight to give 0.59 g. (88%) of white needles, m.p. 93.5–95°. Two recrystallizations from petroleum ether gave an analytical sample of methyl diphenyl-N-(*p*-tolyl)-iminoacetate, m.p. 94–95°.

Anal. Calcd. for $C_{22}H_{21}NO$: C, 83.77; H, 6.41; OCH_3, 9.84. Found: C, 84.15; H, 6.51; OCH_3, 10.15.

Action of Dry Hydrogen Chloride on Methyl Diphenyl-N-(*p*-tolyl)-iminoacetate (IV).—Dry hydrogen chloride was passed into a solution of 0.305 g. of this imino ester in 25 ml. of dry ether for 3.5 hours. After the reaction mixture had remained at room temperature overnight, the volume of ether was reduced by one-half and an equal volume of petroleum ether (b.p. 30–60°) added. Scratching the resulting supersaturated solution with a glass rod precipitated a white solid (0.152 g., 52%), m.p. 173–180°. One recrystallization from aqueous acetone gave 0.122 g. of material, m.p. 180–181°, mixed m.p. with III, 180–181°.

Independent Preparation of Methyl Diphenyl-N-(*p*-tolyl)-iminoacetate (IV).—A mixture of 2.0 g. (6.65 millimoles) of N-(*p*-tolyl)-diphenylacetamide (III) and 1.50 g. (7.20 millimoles) of phosphorus pentachloride was heated at 50–60° for 40 minutes. The reaction mixture was then subjected to reduced pressure (0.3 mm.) to remove the phosphorus oxychloride and the solid residue was quickly digested with 20 ml. of dry ether. The resulting solution, which was decanted from a small amount of insoluble material, was added dropwise to a cold solution of sodium methoxide prepared from approximately 0.5 g. of sodium in 10 ml. of dry methanol. Sodium chloride separated immediately but the reaction mixture was allowed to stand for four hours at room temperature after which time the suspension was shaken twice with water and the ether layer dried over sodium sulfate. Removal of the ether left an oily residue with an unpleasant odor which was dissolved in petroleum ether (b.p. 30–60°) to yield a clear yellow solution. After standing overnight, 0.75 g. (37%) of crystalline material deposited, which was proven to be the starting material (III) by melting point and mixed melting point determination. The filtrate, however, when chilled on Dry Ice deposited a mass of impure material which was recrystallized from 10 ml. of petroleum ether (cooling on Dry Ice was necessary) to give 0.4 g. (19%) of a white solid, m.p. 93–94°. A mixed melting point of this material with the product (IV) obtained from the methanolysis of diphenylketene-*p*-tolylimine was 93–94.5°.

Chlorination of Diphenylketene-*p*-tolylimine (I).—A small amount of chlorine gas was bubbled into a solution of 0.290 g. of diphenylketene-*p*-tolylimine dissolved in 10 ml. of petroleum ether (b.p. 30–60°). Within a few seconds the solution became colorless. The reaction mixture was allowed to stand overnight to give well-formed prisms (0.22 g.) melting at 92–93.5°. A mixed melting point with α-chlorodiphenyl-N-(*p*-tolyl)-iminoacetyl chloride (m.p. 92–93°) was undepressed. Concentration of the mother liquor gave an additional 0.09 g. of material, m.p. 89.5–92°. The total yield was 0.31 g. (86%).

DETROIT, MICHIGAN

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[14] H. Staudinger, *Ann.*, **356**, 81 (1907).