δ 3.4–3.8 (4 H, $-CH_2-N$) and 1.5–2.4 (4 H, aliphatic C–H). A variety of efforts to convert this sample to the lactone **17** were unsuccessful.

Preparation of the Lactone 17.—To a solution containing 1.0 mmole of *t*-butyllithium in 58 ml of pentane was added, dropwise and with stirring over a 10-min period, a solution of 1.786 g (1.09 mmoles) of the amino alcohol **4** in 25 ml of pentane. Then a solution of 2.02 g (1.0 mmole) of bromoacetyl bromide in 25 ml of pentane was added, dropwise and with stirring over a 10-min period, to the suspension of the lithium alkoxide. The resulting mixture was diluted with pentane to 200 ml and 1.0 g of solid sodium bicarbonate was added. The reaction mixture was stirred for an additional 5 min and then filtered and the residue was washed with ether. After the combined pentane solutions had been dried and concentrated, the crude residue was heated in 200 ml of refluxing acetonitrile for 15 hr. The resulting mixture was diluted with ether to precipitate 105 mg (3.6%) of the lactone **17**, mp 235–238° dec. Recrystallization from ethanol afforded the pure **lactone 17** as white prisms, mp 239–240° dec, with infrared absorption[18,28] at 1740 cm^{-1} (δ-lactone C=O) but no absorption in the 3-μ region attributable to an O–H function. The material has nmr[30] singlets at δ 4.87 (2 H, $N-CH_2-CO$), 3.25 (3 H, $N-CH_3$), and 0.98 [9 H, $(CH_3)_3C-$] with triplets (J = 7 cps) centered at δ 3.85 (4 H, $-CH_2-N$) and 2.36 (4 H, $-CH_2-\overset{|}{C}-O$). Solutions of the lactone **17** in deuterium oxide were obtained only when this solvent was heated; the nmr spectra of these solutions corresponded to the spectrum described above except that the peak at lowest field (δ 4.87) was lacking. Consequently, the hydrogen–deuterium exchange, $N-CH_2-CO- \rightarrow N-CD_2-CO-$, must occur relatively rapidly.

(30) Determined as a solution in perdeuteriodimethyl sulfoxide.

Anal. Calcd for $C_{12}H_{22}BrNO_2$: C, 49.32; H, 7.53; Br, 27.40; N, 4.79. Found: C, 49.30; H, 7.48; Br, 27.60; N, 4.50.

Because of the low yield of the lactone **17**, the nature of the remaining products of this reaction was examined briefly. Concentration of the acetonitrile–ether mother liquors remaining after precipitation of the lactone left 920 mg of gummy solid which exhibited no infrared absorption[22] in the 1700–1800-cm^{-1} region attributable to ester or lactone functions. The water-insoluble material (678 mg) obtained from the residue left after filtration of the original reaction mixture exhibited infrared absorption[22] of medium intensity at 1740 cm^{-1} and appeared to be a mixture of a lactone or ester and some other component.

A solution of 42.3 mg (0.185 mmole) of the lactone **17** in 10 ml of methanol containing several milligrams of sodium methoxide was refluxed for 15 hr and then cooled and acidified by the addition of 1 drop of aqueous 48% hydrobromic acid. After the resulting solution had been concentrated and then diluted with ether, the gummy precipitate which separated was collected and found to contain[31] the hydroxy ester **15** but not the lactone **17**. Recrystallization of the crude product from ethanol afforded 24 mg (51%) of the hydroxy ester **15**, mp 148–150° dec, which was identified with the previously described sample by a mixture melting point determination and by comparison of infrared spectra. An appropriate control experiment demonstrated that the stereoisomeric hydroxy ester **14** (mp 215–218°) was not isomerized to ester **15** but rather recovered unchanged (70% recovery) after treatment with refluxing methanol containing several milligrams of sodium methoxide.

(31) A thin layer chromatographic plate coated with silicic acid was employed for this analysis. The eluent was a mixture of chloroform–methanol–concentrated hydrochloric acid (12:12:1 v/v).

Synthesis and Thermal Stability of 1,2-Diazetidinones. Reaction of Diphenylketene with Substituted Azobenzenes[1a]

J. HERBERT HALL[1b] AND RICHARD KELLOGG[1c]

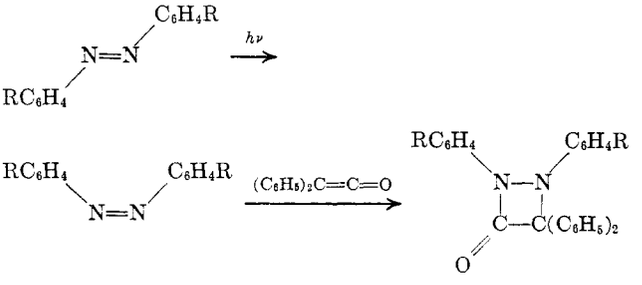
Southern Illinois University, Carbondale, Illinois, and Kansas State Teachers College, Emporia, Kansas

Received August 31, 1965

Several *cis-p,p'*-disubstituted azobenzenes have been generated *in situ* and reacted with diphenylketene to give tetraaryl-1,2-diazetidinones. Some of the problems encountered in the condensation are discussed. The decomposition of tetraphenyl-1,2-diazetidinone into phenyl isocyanate and benzophenone anil is promoted by placing strong electron-donating groups into the *para* positions of the N-1 and N-2 phenyl groups. This effect is interpreted to mean that the decomposition proceeds by initial homolytic cleavage of the N–N bond.

The formation of a 1,2-diazetidinone by reaction of a ketene with an azo compound was first reported by Ingold and Weaver.[2] They treated ethyl phenylazocarboxylate with diphenylketene and obtained a compound which was later identified by Bird[3] as ethyl 2,4,4-triphenyl-1,2-diazetidinone-1-carboxylate. Since the initial report several other workers[4–7] have studied this reaction. Cook and Jones[4] studied the reaction of diphenylketene with azobenzene and discovered that *cis*-azobenzene reacted rapidly with diphenylketene at room temperature to give tetraphenyl-1,2-diazetidinone. In contrast, *trans*-azobenzene did not react with diphenylketene at room temperature and only very slowly at 125–130°.

In order to determine what effect substituents have on the reaction of azobenzene with ketenes, a series of symmetrically substituted azobenzenes have been prepared and then allowed to react with diphenylketene. The technique used in carrying out these reactions was that developed by Cook and Jones[4] in which the *trans*-azobenzene and diphenylketene are dissolved in a suitable solvent and irradiated with ultraviolet light. In this way, the *trans*-azobenzene is converted to the *cis*-azobenzene, which then reacts with the diphenylketene. Ideally, the color of the azobenzene is discharged during the irradiation and



(1) (a) This work was supported in part by a grant from Research Corporation to Kansas State Teachers College. (b) To whom inquiries should be addressed: Southern Illinois University. (c) Undergraduate Research Assistant, 1960–1961, at Kansas State Teachers College.

(2) C. K. Ingold and S. D. Weaver, *J. Chem. Soc.*, **127**, 378 (1925).

(3) C. W. Bird, *ibid.*, 674 (1963).

(4) A. H. Cook and D. G. Jones, *ibid.*, 184 (1941).

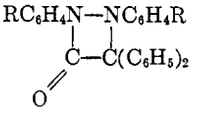
(5) L. Horner and A. Gross, *Ann.*, **573**, 17 (1951).

(6) G. O. Schenk and N. Engelhard, *Angew. Chem.*, **68**, 71 (1956).

(7) L. Horner and E. Spietschka, *Ber.*, **89**, 2765 (1956).

TABLE I

SUBSTITUTED TETRAPHENYL-1,2-DIAZETIDINONES



R	Irradiation time, hr	Solvent[a]	Yield, %	$\nu_{C=O}$, cm^{-1}[b]	Mp, °C	Decompn temp, °C[c]	Formula	—Calcd, %— C	H	—Found, %— C	H
o-Cl	4	E	91	1775	170–170.3[d]	139	$C_{26}H_{18}Cl_2N_2O$	70.12	4.07	70.08	4.03
m-Cl	7	E	55	1755	166.5–167.0[d]	177	$C_{26}H_{18}Cl_2N_2O$	70.12	4.07	70.20	4.21
p-Cl	16	B	17	1785	154–155[e]	...	$C_{26}H_{18}Cl_2N_2O$	70.12	4.07	70.13	4.20
o-NO_2	14	B	20	1790	197.0–197.5[f]	...	$C_{26}H_{18}N_4O_5$	66.95	3.89	67.42	4.05
m-NO_2	7	B	37	1800	171–172[f]	186	$C_{26}H_{18}N_4O_5$	66.95	3.89	67.40	3.98
p-NO_2	28	B	0	...	...	...	...	...	...	...	...
m-Br	6	B	76	1760	180–181[e]	162	$C_{26}H_{18}Br_2N_2O$	58.45	3.40	58.27	3.53
p-Br	5	B	14	1775	185–186[f]	...	$C_{26}H_{18}Br_2N_2O$	58.45	3.40	58.54	3.67
o-OCH_3	1.5	B	19	1770	158[e]	133	$C_{28}H_{24}N_2O_3$	77.04	5.54	76.82	5.55
m-OCH_3	0.3	B	69	1780	103–106[g]	160	$C_{28}H_{24}N_2O_3$	77.04	5.54	77.00	5.69
p-OCH_3	8	E	71	1745	145	133	$C_{28}H_{24}N_2O_3$	77.04	5.54	77.04	5.54
p-$CO_2C_2H_5$	1.7	B	33	1785	143–145[h]	186	$C_{32}H_{28}N_2O_5$	73.83	5.38	73.88	5.40
p-$N(CH_3)_2$	16	B	0	...	...	78	...	...	...		
H	1.5	E	71	1785	175–176[i]	165	...	...	...		

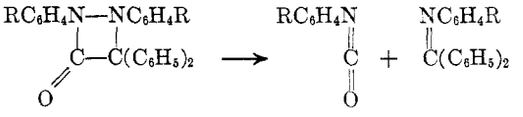
[a] Ether (E) or benzene (B). [b] Nujol mulls. This data was obtained using an Infracord and is reliable to ±10 cm^{-1}. [c] The temperature at which the isocyanate peak at 2270 cm^{-1} first appeared in the infrared spectrum of the heated sample (see Experimental Section). [d] Recrystallized from aqueous acetone. [e] Recrystallized from ethanol. [f] Recrystallized from benzene–ethanol. [g] Recrystallized from methanol. Prolonged heating in methanol or ethanol tends to decompose this compound. [h] Recrystallized from benzene. [i] Lit.[4] mp 175°.

signals completion of the reaction. The yields of 1,2-diazetidinones obtained with various substituted azobenzenes are given in Table I.

With *o,o'*-dichloro-, *m,m'*-dichloro-, *m,m'*-dibromo-, *m,m'*-dimethoxy-, and *p,p'*-dimethoxyazobenzenes the reaction proceeded as expected and gave good yields (55–91%) of the 1,2-diazetidinones. When nitro groups were present on the azobenzene, the yields were much lower, particularly with *o*-nitro and *p*-nitro substituents. When a very dilute ether solution of *p,p'*-dinitroazobenzene was irradiated for 17 hr, the 330-mμ absorption band of the *trans* isomer decreased in intensity and a new band appeared at 240 mμ, presumably owing to the *cis* isomer. However, the rate of isomerization is too slow to allow a practical rate of formation of the 1,2-diazetidinone. This is in agreement with the report of Cook and Jones[8] in which they failed to isolate the *cis* isomers of *o,o'*- and *p,p'*-dinitroazobenzenes from their irradiated solutions.

When *p,p'*-dibromo-, *p,p'*-dichloro-, and *p,p'*-dicarbethoxyazobenzenes were photolyzed with diphenylketene in refluxing benzene, the solutions turned bright blue, red, and green, respectively. The formation of these intensely colored solutions was accompanied by a reduced yield of the 1,2-diazetidinone. Considerable amounts of starting azo compounds were recovered from these reactions. Increasing the irradiation time did not increase the yield. The intensely colored materials apparently absorb most of the ultraviolet radiation and, in effect, stop the reaction. When these colored solutions were chromatographed on alumina, separation of colored fractions was possible, but the amount of material present in these fractions was too small for purification.

Irradiation of a benzene solution of *p,p'*-bis(dimethylamino)azobenzene and diphenylketene did not give the expected 1,2-diazetidinone. The infrared spectrum of the crude reaction mixture contained a very intense band at 2270 cm^{-1}, suggesting the presence of an isocyanate. This was confirmed by reaction of a portion of the mixture with aniline to give the known compound, *p*-dimethylamino-N,N'-diphenylurea. Careful chromatography of the reaction mixture on alumina gave a yellow solid, which was identified by its melting point and its hydrolysis to benzophenone as N-(*p*-dimethylaminophenyl)diphenylketimine. These results indicate that reaction of *p,p'*-bis(dimethylamino)azobenzene with diphenylketene does give the 1,2-diazetidinone, but that it undergoes decomposition in the refluxing benzene into the isocyanate and the ketimine. This is supported by the

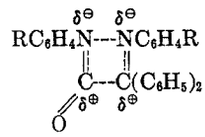


report[4] that tetraphenyl-1,2-diazetidinone decomposes at 190° to give benzophenone anil. Also, Schenk and Engelhard[6] reported that 1,2-diphenyl-1,2-diazetidinone decomposed into phenyl isocyanate and polymerized formaniline when it was refluxed in acetone.

The fact that the presence of *p*-dimethylamino groups on the N-1 and N-2 phenyl groups promoted the decomposition of tetraphenyl-1,2-diazetidinone into the isocyanate and ketimine prompted a qualitative study on the decomposition temperatures of some of the substituted tetraphenyl-1,2-diazetidinones. The results are given in Table I. It can be seen from this data that the presence of *o*- and *p*-methoxy groups on the N-1 and N-2 phenyls also promotes the decomposition, although apparently to a lesser degree than the *p*-dimethylamino groups.

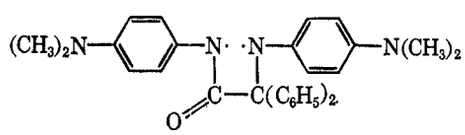
If the decomposition of tetraphenyl-1,2-diazetidinones proceeds by a concerted process, the transition state might be expected to resemble the products and therefore exhibit the following polarity. Such a

(8) A. H. Cook and D. G. Jones, *J. Chem. Soc.*, 1309 (1939).



transition state should be stabilized by electron-withdrawing groups and destabilized by electron-donating groups on the N-1 and N-2 phenyls. This leads to the prediction that the reaction should be promoted by electron-withdrawing groups. Since this is not the case, the concerted process seems unlikely.

A mechanism which seems more consistent with the observed substituent effect involves homolytic cleavage of the relatively weak N–N bond to give the diradical. It is well known that *p*-dimethylamino and *p*-methoxy groups stabilize imino radicals in the dissociation of tetraphenylhydrazines.[9] It seems reasonable to assume that the same type of stabilization should promote the homolytic cleavage of 1,2-diazetidinones.



Experimental Section

Preparation of 1,2-Diazetidinones.—The 1,2-diazetidinones listed in Table I were prepared by dissolving the appropriately substituted *trans*-azobenzene (0.0025 mole) and diphenylketene (0.0025 mole) in 20–100 ml of dry ether or benzene and irradiating the solution with ultraviolet light. The light source was a General Electric, Uviarc (U.A.-3), air-cooled mercury lamp. The irradiations were carried out in a Pyrex flask fitted with reflux condenser and drying tube. The lamp was located within 1 cm of the bottom of the flask. Sufficient heat was generated by the lamp to cause the solvent to reflux during the irradiation. The irradiation times and the solvents used are listed Table I. At the end of the irradiation, the solvent was distilled, the last traces being removed under vacuum. The residue remaining was then processed as described for each of the individual compounds listed below.

1,2-Di(*o*-chlorophenyl)-4,4-diphenyl-1,2-diazetidinone.—The residue remaining after evaporation of the solvent was dissolved in a small amount of acetone, and water was added until the solution was turbid. Chilling in ice gave 1.0 g (91%) of the diazetidinone.

1,2-Di(*m*-chlorophenyl)-4,4-diphenyl-1,2-diazetidinone.—After evaporation of the solvent, the remaining viscous orange liquid was extracted with hot ethanol to remove unreacted *m,m'*-dichloroazobenzene. The ethanol-insoluble solid was recrystallized from aqueous acetone to give 0.61 g (55%) of the 1,2-diazetidinone.

1,2-Di(*p*-chlorophenyl)-4,4-diphenyl-1,2-diazetidinone.—At the end of the irradiation, the solution was deep red. The residue remaining after removal of the solvent was warmed with 5 ml of absolute alcohol. After cooling, the insoluble solid was filtered off, giving 0.50 g of a mixture of the expected diazetidinone and the starting azo compound. The mixture was recrystallized from ethanol (charcoal) to give 0.20 g (17%) of the diazetidinone.

1,2-Di(*o*-nitrophenyl)-4,4-diphenyl-1,2-diazetidinone.—After the irradiation, the solution was concentrated to 5 ml and cooled. The yellow needles which separated were filtered off: yield 0.20 g. This solid gave no melting point depression with the starting *o,o'*-dinitroazobenzene. The filtrate was evaporated to dryness and the residue was extracted with six 50-ml portions of hot hexane. Evaporation of the hexane gave 0.4 g of solid, which consisted of yellow granules and red powder. Washing of the solid with carbon tetrachloride dissolved the red powder and left 0.24 g (20%) of the diazetidinone.

1,2-Di(*m*-nitrophenyl)-4,4-diphenyl-1,2-diazetidinone.—The residue remaining after removal of the solvent was refluxed with 30 ml of hexane for a few minutes. The hot hexane was decanted. Recrystallization of the hexane-insoluble solid from 1:2 benzene–ethanol gave 9.42 g (37%) of the diazetidinone.

1,2-Di(*m*-bromophenyl)-4,4-diphenyl-1,2-diazetidinone.—After removal of the solvent, 5 ml of ethanol was added to the oily residue. The oil solidified and was filtered off: yield 1.06 g (76%).

1,2-Di(*p*-bromophenyl)-4,4-diphenyl-1,2-diazetidinone.—At the end of the irradiation, the solution was intense blue, resembling blue ink. The dark residue remaining after removal of the solvent was warmed with 10 ml of ethanol and then allowed to cool. The crude solid was filtered off, washed with ethanol, and recrystallized from 1:2 benzene–ethanol. The product obtained was a mixture of the *p,p'*-dibromoazobenzene and the expected diazetidinone. The mixture was separated by treating it with 50 ml of ethanol, which dissolved the diazetidinone, but only a part of the *p,p'*-dibromoazobenzene. The azo compound was filtered off. The filtrate was treated with charcoal, concentrated to 8 ml, and cooled to 5°. Filtration of the solid which separated gave 0.20 g (14%) of the diazetidinone.

1,2-Di(*o*-methoxyphenyl)-4,4-diphenyl-1,2-diazetidinone.—After solvent removal, the sticky residue was extracted with three 50-ml portions of boiling hexane. Chilling of the hexane extracts to −10° gave a crude product, which was recrystallized from methanol to give 0.20 g (19%) of the diazetidinone.

1,2-Di(*m*-methoxyphenyl)-4,4-diphenyl-1,2-diazetidinone.—Removal of the solvent left a yellow glass. It was extracted thoroughly with four 50-ml portions of boiling hexane. The hexane extracts were evaporated. The residue was recrystallized from ethanol: yield 0.72 g (69%).

1,2-Di(*p*-methoxyphenyl)-4,4-diphenyl-1,2-diazetidinone.—After removal of the solvent, the residue was extracted once with 50 ml of boiling hexane. The hot hexane was decanted. Treatment of the insoluble residue with 5 ml of ethanol, followed by filtration, gave 0.81 g (71%) of the diazetidone as colorless needles.

1,2-Di(*p*-carbethoxyphenyl)-4,4-diphenyl-1,2-diazetidinone.—The solution during the irradiation became very dark green in color. Removal of the benzene gave a dark sticky substance. This material was dissolved in 50 ml of hot hexane and the solution was decolorized with charcoal. The solvent was evaporated and the residue was recrystallized from ethanol to give 0.14 g of recovered *p,p'*-dicarbethoxyazobenzene. The ethanol filtrate was evaporated and the residue was dissolved in a minimum amount of benzene. This was placed on an alumina column. Elution of the column with 220 ml of benzene, followed by 70 ml of chloroform, gave a colorless oil, which after crystallization from benzene gave 0.36 g (33%) of the diazetidinone.

Reaction of Diphenylketene with *p,p'*-Bis(dimethylamino)azobenzene.—The oily residue remaining after the removal of the solvent was extracted with 10 ml of hexane.

The hexane-insoluble residue was dissolved in 5 ml of benzene and the solution was filtered to remove 0.06 g of the starting azo compound. The benzene filtrate was placed on an alumina column and eluted with benzene to give 0.12 g of a solid. This solid was dissolved in 3 ml of ether and the solution was filtered to give 0.02 g of starting azo compound. Evaporation of the ether gave 0.09 g (12%) of the benzophenone anil of *p*-dimethylaminoaniline. Recrystallization of a portion of the compound from a concentrated ether solution gave a pure sample, mp 86–88°, lit.[10] mp 86°). The remainder of the anil was treated with a few drops of concentrated hydrochloric acid. A colorless oil was formed, which solidified on cooling. This solid melted at 45–47°, and its infrared spectrum was identical with that of a sample of benzophenone.

A few drops of the hexane-soluble fraction were evaporated to give an oil. The infrared spectrum of this oil contained a strong band at 2270 cm^{-1}, indicating the presence of an isocyanate group. To the bulk of the hexane-soluble fraction was added 0.24 g of aniline. The solution was allowed to stand overnight. The solid which precipitated was filtered off, washed with hexane, and dried: yield 0.22 g (33%). Recrystallization from ethanol gave a pure sample of *p*-dimethylamino-N,N'-diphenylurea, mp 207–208° (lit.[11] mp 207–208°).

(9) (a) H. Wieland, *Ber.*, **48**, 1078 (1915); (b) *ibid.*, **48**, 1091 (1915); (c) G. N. Lewis and D. Lipkin, *J. Am. Chem. Soc.*, **63**, 3232 (1941).

(10) F. J. Moore, *Ber.*, **43**, 563 (1910).

(11) H. Staudinger and S. Jelagin, *ibid.*, **44**, 370 (1911).

Thermal Stability of 1,2-Diazetidinones.—In small test tubes were placed 0.03 g of each of the 1,2-diazetidinones together with 0.3 g of paraffin wax. These tubes were then immersed for 20 min in a preheated oil bath. A small portion of the wax was removed from each tube and pressed between salt plates, and the infrared spectrum was obtained. If there was no isocyanate peak at *ca.* 2270 cm^{-1}, the tube was placed back in a bath at a higher temperature and the process was repeated. The decomposition temperatures listed in Table I are the lowest temperature at which an isocyanate peak was observable.

The Solvolysis of Alkyl Diazotates. I. Partition between Carbonium Ions and Diazoalkanes in Aqueous Base

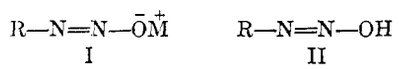
Robert A. Moss

Wright Laboratory, School of Chemistry, Rutgers, The State University, New Brunswick, New Jersey

Received December 1, 1965

A series of alkyl diazotates, obtained by basic cleavage of N-alkyl-N-nitrosourethans, afforded, upon hydrolysis, carbonium ion products and/or diazoalkanes. Partition was dependent on alkyl-group structure. This dependence is discussed in some detail. Alkyl diazotates appear to be a source of carbonium ions in aqueous base.

N-Nitrosourethans have long been employed as precursors of diazoalkanes.[1] Intermediacy of diazotate salts, I, in the preparation of diazomethane and phenyldiazomethane was demonstrated by Hantzsch.[2,3]

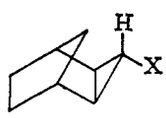


I II

Much later, Huisgen suggested their occurrence as an intermediate in the basic decomposition of nitrosocaprolactam.[4] Under conditions similar to those of Hantzsch,[2] we decomposed a series of N-alkyl-N-nitrosourethans, observing facile formation of ether-insoluble, proton-sensitive substances, presumably alkyl diazotates. Aqueous quenching of these substances very rapidly affords diazoalkanes, and/or nitrogen, and products commonly associated with carbonium ions. The relation between diazo and ionic decomposition pathways, the common origin of these pathways in another intermediate, possibly II, and the relation between alkyl-group structure and decomposition pathway will be discussed.

Results

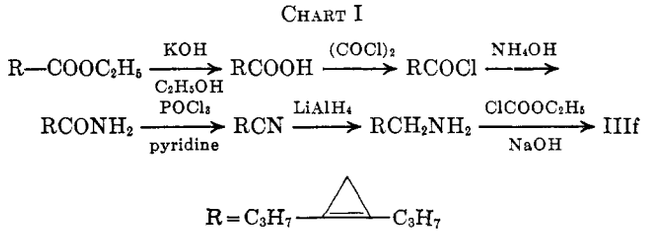
Preparation of Starting Materials.—Urethans IIIa–f, (X = $NHCOOC_2H_5$) were selected for nitrosation. Urethans IIIa,[5] b,[6] c,[7] and d[8] were known. The last compound was prepared by addition of ethanol to the commercially available allyl isocyanate, the first three *via* reaction of ethylchloroformate with the requisite amine.[9] Urethans IIIe and f were unknown. The former was obtained from Curtius rearrangement in refluxing ethanol of acid azide IVc, itself obtained, *via* acid chloride IVb, from the known acid IVa.[10] The



IVa, X = COOH
b, X = COCl
c, X = CON_3

latter was obtained from the requisite ester[11] (Chart I). Some apparent shortcuts failed, for example, ammonolysis of starting ester directly to amide. Also lithium aluminum hydride reduction of amide to amine was effected only with simultaneous double-bond reduction.[12] Urethans were characterized by infrared and nmr spectra.[13] Satisfactory elemental analyses were obtained for the new compounds.

Chart I



Nitrosation of urethans IIIa–f was accomplished with ethereal nitrogen tetroxide,[14] care being taken, in the case of IIIf, to keep the reaction temperature below −55° (above this temperature, destruction of the double bond was dominant[15]). Nitrosourethans IIIe

(1) H. v. Pechmann, *Ber.*, **28**, 855 (1895), and references therein.
(2) A. Hantzsch and M. Lehmann, *ibid.*, **35**, 897 (1902).
(3) See also H. Zollinger, "Azo and Diazo Chemistry," Interscience Publishers, Inc., New York, N. Y., 1961, p 44 ff.
(4) R. Huisgen and J. Reinertshofer, *Ann.*, **575**, 174 (1952). See also G. Nischk and E. Müller, *ibid.*, **576**, 232 (1952).
(5) A. L. Wilds and A. L. Meader, Jr., *J. Org. Chem.*, **13**, 763 (1948).
(6) D. W. Adamson and J. Kenner, *J. Chem. Soc.*, 181 (1939).
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