The structures of the major *trans*-adduct, III, and the *cis*-adduct, IV, both formed by anti-Markovnikov addition to the acetylenic bond, are assigned by analogy to the similar addition with dimethylphosphorylsulfenyl chloride.[6] In general, *trans* addition of sulfenyl chlorides to acetylenes has been assumed.[11,12] The dependence of product orientation on solvent has recently been reported.[12] A small increase of the *cis*-adduct, IV, due to postisomerization on standing at ambient temperature was noted during the present study.

Experimental Section

Starting Unsaturates.—Propylene (99.7% purity), isobutylene (+99% purity), and methylacetylene (+98% purity) were obtained from Matheson Co. The 3,3-dimethylbutene and *t*-butylacetylene were products of the Columbia Organic Chemicals Co. and were distilled before use. The 3-methylbutene (99% purity) was obtained from Phillips Chemical Co. Norbornene from the Matheson Co. was sublimed prior to its use.

Method of Analyses.—Nmr spectra were recorded neat on a Varian Model A-60 resonance spectrometer using tetramethylsilane as an internal standard. The infrared spectra were obtained on a Beckman Model IR-10 infrared spectrophotometer.

Dimethylaminosulfenyl Chloride.—Dimethylamine (2 mol) was treated in an ethereal solution with 1 mol of freshly distilled sulfur dichloride according to a procedure described in a German patent.[8] The pure sulfenyl chloride, bp 55° (45 mm), was obtained in 55% yield. Its nmr spectrum showed a singlet at 3.12 ppm.

General Procedure for the Addition of Dimethylaminosulfenyl Chloride to Unsaturated Hydrocarbons.—An equimolar amount of the sulfenyl chloride was slowly added to a solution of the olefin in methylene chloride containing a small amount of suspended $CaCO_3$. In the case of acetylenes, a two- to fourfold excess of the unsaturate was used to avoid diadduct formation. The reactions were carried out under anhydrous conditions (nitrogen blanket) and at various reaction temperatures depending on the reactivity of the olefin, its boiling point being the limiting factor. Magnetic stirring was applied during all reactions. The progress of the addition was followed by nmr spectroscopy. Upon near completion of the reaction, the excess unsaturate and solvent were removed at 0° *in vacuo* (*ca.* 5 mm). The products were then again sampled for semiquantitative nmr analysis. In general, the crude products were found to be ~90% pure. For purposes of elemental analysis part of each product was further purified by fractional distillation *in vacuo*.

To Propylene.—To 9.7 g (0.23 mol) of propylene condensed into 50 ml of CH_2Cl_2 at −70°, 22.3 g (0.2 mol) of dimethylaminosulfenyl chloride was slowly added. The reaction mixture was kept at −70° for 2 hr and then for 1 additional hr at −50°. The solution was then slowly warmed to 0°. Removal of the solvent afforded 29.5 g (96%) of a pale yellow oil. An analytical sample, bp 77° (30 mm), was obtained on fractional distillation.

Anal. Calcd for $C_5H_{12}NSCl$: C, 39.06; H, 7.87; S, 20.85. Found: C, 39.08 H, 7.98; S, 20.85.

To Isobutylene.—The addition of 8 g (0.072 mol) of sulfenyl chloride to a solution of 4.1 g (0.073 mol) of isobutylene in 30 ml of CH_2Cl_2 proceeded slightly exothermically at −20°. The solvent was removed after 10 min at −20° and afforded 11.4 g (94%) of a pale yellow liquid which distilled at 73° (19 mm).

Anal. Calcd for $C_6H_{14}NSCl$: C, 42.97; H, 8.41; S, 19.12. Found: C, 42.87; H, 8.72; S, 19.03.

To 3-Methylbutene.—The sulfenyl chloride (8 g, 0.072 mol) was added to 5.1 g (0.073 mol) of 3-methylbutene in 30 ml of CH_2Cl_2 at −20°. After completion of addition the solution was slowly warmed to 0° and kept at this temperature for 30 min. Removal of the solvent afforded 12 g (92%) of crude product. The adduct distilled at 73–74° (9 mm).

Anal. Calcd for $C_7H_{16}NSCl$: C, 46.26; H, 8.87; S, 17.64. Found: C, 46.18; H, 9.03; S, 17.59.

To 3,3-Dimethylbutene.—The sulfenyl chloride (8 g, 0.072 mol) was added to 6.1 g (0.073 mol) of 3,3-dimethylbutene in 30 ml of CH_2Cl_2 at −20°. The solution was then allowed to warm to 0° and then kept at this temperature for 30 min. After an additional 30 min at ambient temperature, the solvent was removed at this temperature and 3 mm. A tan liquid, 11.8 g (84%), was obtained.

Anal. Calcd for $C_8H_{18}NSCl$: C, 49.08; H, 9.27; S, 16.38. Found: C, 48.99; H, 9.27; S, 16.70.

Attempted distillation at 80° (bath temperature) and 55 mm resulted in strong foaming and reversal of the adduct to its starting materials. Approximately 85% of the sulfenyl chloride and 3,3-dimethylbutene were collected in separate traps.

To Norbornene.—A slightly exothermic reaction at −20° was observed upon addition of 11.15 g (0.1 mol) of the sulfenyl chloride to 9.4 g (0.1 mol) of norbornene in 15 ml of CH_2Cl_2. After 15 min at −20° the reaction mixture was allowed to warm to −10°. Removal of the solvent at this temperature left 19.2 g (93%) of an orange oil. Distillation *in vacuo* afforded a tan liquid, bp 52–53.5° (0.001 mm).

Anal. Calcd for $C_9H_{16}NSCl$: C, 52.54; H, 7.84; S, 15.58. Found: C, 52.15; H, 8.05; S, 15.39.

To Methylacetylene.—The sulfenyl chloride (11.15 g, 0.1 mol) was slowly added to 20 g (0.5 mol) of methylacetylene condensed into 30 ml of CH_2Cl_2 at −30°. After 3 hr at this temperature, nmr analysis indicated only 20% conversion of the sulfenyl chloride. Therefore, 140 mg of $AlCl_3$ was added. Within 20 hr at −30° the reaction mixture had gradually darkened and the conversion had reached 85%. The excess methylacetylene was then removed at −30° and under slight vacuum. The solution was then cooled to −80°, 100 ml of precooled CH_2Cl_2 added, and the cold solution washed with 20 ml of 5% aqueous $NaHCO_3$ solution followed by 20 ml of water. After drying with $MgSO_4$ and removal of the solvent at ambient temperature (60 mm), 9.5 g (67%) of a dark liquid was obtained. Distillation afforded a tan, malodorous liquid, bp 88–89° (48 mm).

Anal. Calcd for $C_5H_{10}NSCl$: C, 39.60; H, 6.65; S, 21.14. Found: C, 40.07; H, 6.90; S, 21.18.

To *t*-Butylacetylene.—The sulfenyl chloride (2.78 g, 0.025 mol) was added to a solution of 6.3 g (0.077 mol) of *t*-butylacetylene in 5 ml of CH_2Cl_2 at 0°. After 15 min, at this temperature, the reaction mixture was allowed to reach room temperature and was then kept at ambient temperature for 5 hr. Removal of the solvent and excess unsaturate afforded 3.9 g (81%) of a light brown liquid. Distillation resulted in a yellow, malodorous liquid of bp 29–33° (0.003 mm).

Anal. Calcd for $C_8H_{16}NSCl$: C, 49.59; H, 8.32; S, 16.55. Found: C, 49.29; H, 8.13; S, 16.48.

Registry No.—III (R = CH_3), 16133-70-3; III (R = *t*-Bu), 16133-71-4.

Acknowledgment.—The authors wish to thank Mr. W. C. Whitlock for excellent technical assistance.

(11) N. Kharasch and C. N. Yianios, *J. Org. Chem.*, **29**, 1190 (1964).

(12) V. Calò, G. Melloni, G. Modena, and G. Scorrano, *Tetrahedron Lett.*, **49**, 4399 (1965), and references therein.

The Chemistry of Sulfoacetic Acid Derivatives. I. Reactions of Chlorosulfonylacetyl Chloride with Nucleophiles

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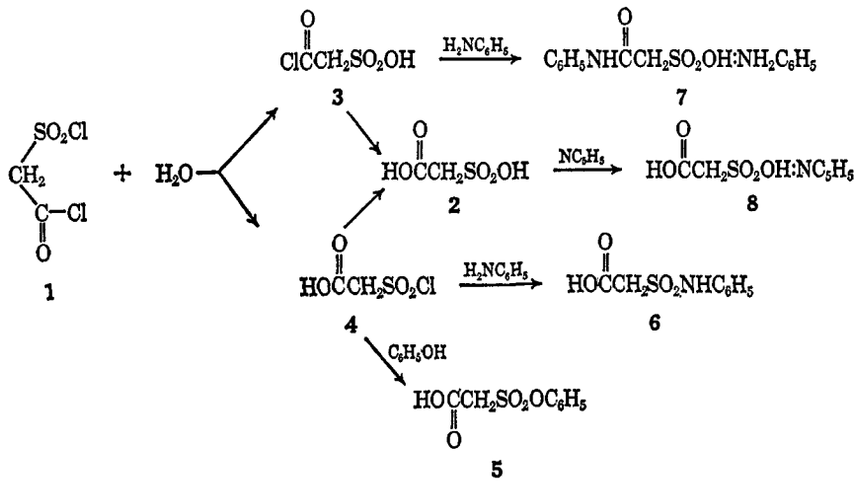
Vieillesfosse,[1] Bodendorf and Senger,[2] and Hinman and Locatell[3] have all investigated the course of the

(1) R. Vieillefosse, *Compte. Rend.*, **208**, 1406, 1515 (1939); *Bull. Soc. Chim. Fr.*, 351 (1947); *ibid.*, **6**, 34 (1939).

(2) K. Bodendorf and N. Senger, *Ber.*, **72B**, 571 (1939).

(3) (a) R. L. Hinman and L. Locatell, Jr., *J. Amer. Chem. Soc.*, **81**, 5655 (1959); (b) B. E. Hoogenboom, R. Abbott, L. Locatell, Jr., and R. L. Hinman, *J. Org. Chem.*, **24**, 1983 (1959); (c) R. L. Hinman and B. E. Hoogenboom, *ibid.*, **26**, 3461 (1961).

SCHEME I



partial hydrolysis of the diacid chloride of sulfoacetic acid in the presence of a limited amount of water. Bodendorf and Senger concluded that partial hydrolysis of the diacid chloride **1** yields, in addition to sulfoacetic acid (**2**), mainly the carboxylic acid chloride **3**; Vieillefosse and Hinman and Locatell, however, suggested that the monoacid chloride produced in the partial hydrolysis of the diacid chloride is the sulfonyl chloride (**4**) and that, whereas both ends of the diacid chloride are easily hydrolyzed, the sulfonyl chloride is slightly more stable to hydrolysis than the carboxylic acid chloride.

We have repeated the partial hydrolysis of the diacid chloride **1** and have isolated only low purified yields (<10%) of a monoacid chloride. This product has been identified as the sulfonyl chloride (**4**) by its conversion into the corresponding phenyl ester, phenyl carboxymethanesulfonate (**5**),[3a] and anilide, carboxymethanesulfonanilide (**6**).[4] When a portion of the crude partial hydrolysis mixture was treated with excess aniline, the main isolated product (25%) was identified as the aniline salt (**7**) of N-phenylcarbamylmethanesulfonic acid, as reported by Vieillefosse[1] and Bodendorf and Senger.[2] In addition, small yields of carboxymethanesulfonanilide (**6**, 2.3%) and sulfoacetic acid (**2**, 6.6%), isolated as the pyridine salt **8**, were obtained.

This series of experiments indicates that the partial hydrolysis of the diacid chloride (**1**) of sulfoacetic acid occurs more easily at the sulfonyl group of the molecule, producing primarily sulfoacetyl chloride (**3**) and a smaller quantity of the isomeric carboxymethanesulfonyl chloride (**4**). The latter compound (**4**) is more readily isolated and purified by recrystallization and has thus been assumed by previous workers to be the major product of the partial hydrolysis reaction. Sulfoacetic acid (**2**) arises from the complete hydrolysis of the diacid chloride (**1**) (Scheme I).

Although the nucleophile water seems to attack preferentially at the sulfonyl portion of the diacid chloride, the nucleophiles phenol and aniline show a different selectivity in their attack on the diacid chloride molecule.

When the diacid chloride **1** is warmed gently with 1 equiv of phenol and the resulting crude monophenyl ester treated in cold ether with 2 equiv of aniline, the neutral product isolated in very high yield (80%) is carbophenoxymethanesulfonanilide (**9**). None of the isomeric anilide phenyl ester, phenyl N-phenylcarbamylmethanesulfonate (**10**), was isolated; it was, however, detected and identified along with diphenyl sulfoacetate (**11**),[3b] the dianilide (**12**)[2] of sulfoacetic acid, and phenol by thin layer chromatographic techniques. Apparently, in the first step of the reaction sequence, the nucleophilic phenol attacks mainly at the carbonyl portion of the diacid chloride **1**. Excess phenol readily attacks at both the carbonyl and sulfonyl functions to product diphenyl sulfoacetate (**11**) in good over-all yield (81%) (Scheme II).

When the diacid chloride **1** is treated in the cold with 2 equiv of aniline, followed by heating the crude monoanilide mixture gently with 1 equiv of phenol, the only product isolated in pure form (27%) is phenyl N-phenylcarbamylmethanesulfonate (**10**). The crude reaction product is initially an oily, solid mixture, and it is possible that other products are also formed in large quantities. Thin layer chromatographic analysis of the crude reaction mixture showed the presence of phenyl N-phenylcarbamylmethanesulfonate (**10**), the diphenyl ester **11**, the dianilide **12**, phenol, and the isomeric anilide phenyl ester, carbophenoxymethanesulfonanilide (**9**). As in the case of attack on the diacid chloride by phenol, attack of aniline on the diacid chloride appears to occur somewhat more easily at the carboxyl acid chloride function. Excess aniline readily attacks at both the carbonyl and sulfonyl functions of the diacid chloride to produce the dianilide (**12**)[2] of sulfoacetic acid.

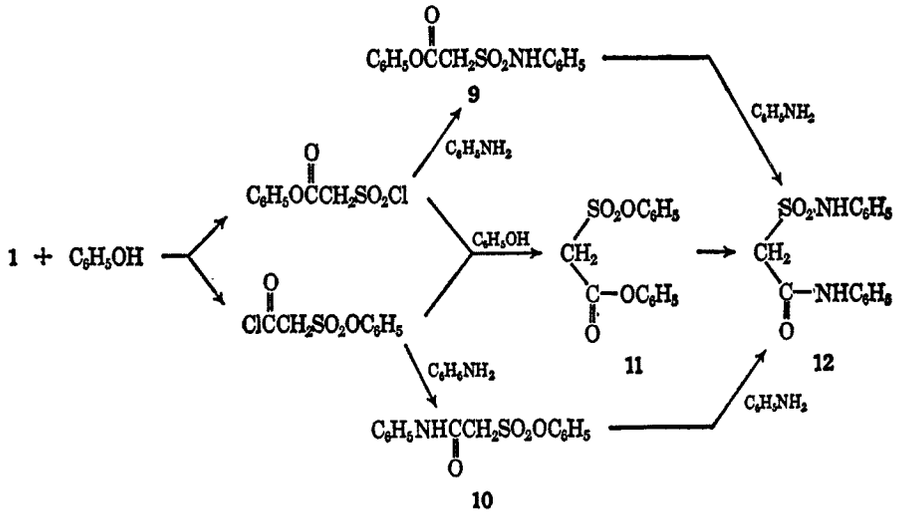
Experimental Section[5]

Partial Hydrolysis of Chlorosulfonylacetyl Chloride (1).—The partial hydrolysis was carried out as described elsewhere by Hinman and Locatell[3a] using either benzene, carbon tetrachloride, or anhydrous ether as the solvent. The chlorosulfonylacetyl chloride used was a freshly distilled practical grade obtained from Eastman Kodak. Recrystallization of the crude hydrolysis product from carbon tetrachloride usually gave poor (0–10%) yields of chlorosulfonylacetic acid (**4**), mp 77–78° (lit.[3a] mp 77–78°). This material was identified positively as the sulfonyl

(4) B. Loev, M. Kormendy, and K. Snader, *J. Org. Chem.*, **31**, 3531 (1966).

(5) Melting points are uncorrected and were determined on a Fisher-Johns melting point apparatus. Microanalyses were performed by Galbraith Laboratories, Inc., Knoxville, Tenn. Infrared spectra were obtained with a Beckman IR-8 instrument in Nujol mulls.

SCHEME II



chloride 4 by its conversion, on heating with phenol for a period of 3 hr, into phenyl carboxymethanesulfonate (5), mp 86–87° (lit.[3a] mp 86–87°). On repeating the partial hydrolysis of 10 g (0.057 mol) of the diacid chloride in carbon tetrachloride, the crude hydrolysis product (6.5 g), separated from the reaction mixture by filtration, was treated with excess aniline. The resulting solid mixture was extracted repeatedly with ether. The ether extracts were washed with 5% hydrochloric acid to remove excess aniline and then dried over magnesium sulfate. Removal of the drying agent and the ether under aspirator pressure left a solid, which on recrystallization from benzene, yielded 0.27 g (2.3%) of carboxymethanesulfonanilide (6), mp 112–113° (lit.[4] 118.5–119°). When ether was used as solvent for the hydrolysis of the diacid chloride, the dianilide (12, 6.1%), mp 149–150° (lit.[2] mp 151°), rather than carboxymethanesulfonanilide (6) was obtained from the ether extracts.

One-half of the solid remaining after the ether extraction was dissolved in water and passed through a column of Dowex-50W-X4 cationic exchange resin. The strongly acidic eluant was evaporated to near dryness and the residual oil treated with excess pyridine. Further evaporation and recrystallization of the residual solid from absolute ethanol yielded 0.41 g (6.6%) of the pyridine salt (8) of sulfoacetic acid, mp 151–152°. This material was found to be identical with an authentic sample, mp 148–151°, prepared from sulfoacetic acid.

The other half of the solid remaining after the ether extraction was recrystallized from ethanol, affording 2.15 g (24.6%) of leafy white crystals, mp 223–236°. A second recrystallization provided pure material, mp 234–236°, identified as anilinium N-phenylcarbamylmethanesulfonate (7) (lit.[2] mp 234°). This material was identical with the aniline salt prepared from an authentic sample of N-phenylcarbamylmethanesulfonic acid.

Preparation of Carbophenoxymethanesulfonanilide (9).—Over a period of 12 hr, a total of 6.0 g (0.064 mol) of phenol was added in small portions to 11.3 g (0.064 mol) of chlorosulfonylacetyl chloride (1) warmed gently on a steam bath. After addition of the phenol, the mixture was warmed for another 7 hr. The reaction mixture was dissolved in anhydrous ether and the ether then removed by evaporation under aspirator pressure to remove the last traces of hydrogen chloride. The residual oil was then redissolved in anhydrous ether, chilled in an ice bath, and treated over a period of 2.5 hr, with rapid stirring, with a solution of 11.9 g (0.128 mol) of aniline in 30 ml of anhydrous ether. After addition of the aniline, the mixture was stirred for 1 hr. The ether was then removed and the residual grey solid triturated with aqueous ethanol. The solid was finally separated by filtration and rinsed with water, affording 23.55 g of crude product, mp 125–135°. Recrystallization from 95% ethanol gave 14.11 g (76%) of carbophenoxymethanesulfonanilide (9), mp 133–135°. From the filtrates was isolated a small yield (6.1%) of diphenyl sulfoacetate (11).[3b] Thin layer chromatographic analysis[6] of the filtrates indicated the additional presence of the dianilide 12, phenyl N-phenylcarbamylmethanesulfonate (10), and phenol. Carbophenoxymethanesulfonanilide (9) exhibited infrared absorption peaks at 3.03 (N–H), 5.7 (C=O), 8.3–8.65, and 8.9 (SO_2) μ.

Anal. Calcd for $C_{14}H_{13}O_4NS$ (9): C, 57.72; H, 4.50; N, 4.81. Found: C, 57.54; H, 4.52; N, 5.02.

Preparation of Phenyl N-Phenylcarbamylmethanesulfonate (10) from Chlorosulfonylacetyl Chloride (1).—To a chilled solution of 3.81 g (0.0215 mol) of chlorosulfonylacetyl chloride (1) in 30 ml of anhydrous ether was added dropwise and with rapid stirring a solution of 4.00 g (0.0430 mol) of aniline in 30 ml of anhydrous ether over a period of 1.5 hr. After the addition of the aniline, the mixture was stirred for an additional 30 min. The ether solvent was then removed under aspirator pressure, and the residual yellow, oily solid was warmed gently with 2.03 g (0.0216 mol) of phenol for a period of 16 hr. After about 12 hr the mixture became solid. Ice and ethanol were added to triturate the oily solid. By filtration and rinsing with ethanol–water mixtures and finally with water, a total of 2.18 g (34.7%) of pale yellow solid, mp 116–121°, was obtained. Thin layer chromatographic analysis[6] of this material indicated it to be nearly pure phenyl N-phenylcarbamylmethanesulfonate (10) containing only a trace of the dianilide 12. Recrystallization from 95% ethanol afforded 1.68 g (27%) of pure white product, mp 124–125°. The aqueous filtrate remaining after filtration of the crude product was extracted with ether, and the extracts were dried over anhydrous sodium sulfate. Removal of the ether in a rotary evaporator left 3.05 g of an oil which partially crystallized to yield a small additional quantity of 10. Thin layer chromatographic analysis indicated the uncrystallized oil to be a mixture of both isomeric anilide phenyl esters (9 and 10), the dianilide 12, diphenyl sulfoacetate (11), and phenol.

Phenyl carboxymethanesulfonate (5) was converted into the acid chloride by warming with excess thionyl chloride for 45 min. The excess thionyl chloride was removed under aspirator pressure. The residual acid chloride was dissolved in anhydrous ether and treated with excess aniline. The ether solution was then washed several times with dilute hydrochloric acid, dried, and then concentrated under reduced pressure. Recrystallization of the residual solid from 95% ethanol afforded pure phenyl N-phenylcarbamylmethanesulfonate (10), mp 122–123.5°, in 65% yield.

The samples of phenyl N-phenylcarbamylmethanesulfonate (10) prepared by these methods exhibited identical infrared absorption spectra, with peaks at 2.98 (N–H), 5.94 (C=O), and 8.59 (SO_2) μ.

Anal. Calcd for $C_{14}H_{13}O_4NS$ (10): C, 57.72; H, 4.50; N, 4.81. Found: C, 57.90; H, 4.55; N, 4.79.

Registry No.—1, 4025-77-8; 9, 16753-80-3; 10, 16753-81-4.

(6) Thin layer chromatograms were developed on Eastman Chromatogram sheets (silica gel) using chloroform as solvent. Spots were visualized in an atmosphere containing iodine vapor.

Acknowledgment.—Support of this investigation by an F. G. Cottrell Grant from the Research Corporation and by Public Health Service Research Grant GM 12153 from the National Institutes of Health is gratefully acknowledged.

Formation of 1,3-Dianions of 2- and 3-Acetamidopyridines by Means of *n*-Butyllithium. Condensations with Carbonyl Compounds and Nitriles.[1]

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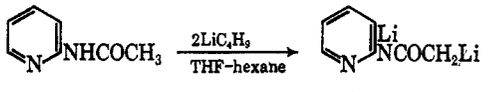
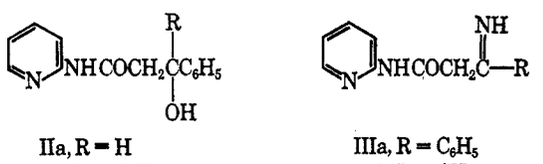
As an extension of our recent method for effecting condensations at the α carbon of acetanilide,[2] we have found that 2- and 3-acetamidopyridines undergo α metalation, as well as N metalation, with excess *n*-butyllithium to form the corresponding dilithioamides, as evidenced by condensations at the terminal position with electrophilic compounds to give C derivatives. Thus, 2-acetamidopyridine (I) was converted into dilithioamide I″, which underwent addition reactions with benzaldehyde, benzophenone, and benzonitrile, and benzoylation with methyl benzoate, to afford IIa, IIb, IIIa, and IV, respectively. Also, I″ was condensed with acetonitrile to give IIIb, but the yield was low presumably because of predominant ionization of an α hydrogen of the nitrile by I″.



I I″



IIa, R = H; b, R = C_6H_5 IIIa, R = C_6H_5; b, R = CH_3

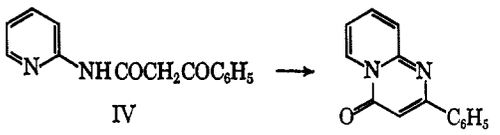
Attempts to effect acid-catalyzed conversions of adducts IIa–b into the corresponding unsaturated and cyclic products, similar to those previously obtained from the benzophenone adduct of acetanilide,[2] were unsuccessful; in the reaction of IIa with cold sulfuric acid, cinnamic acid was obtained. Imine IIIa was hydrolyzed to give the β-ketoamide IV, which was also prepared by the known method from 2-aminopyridine and ethyl benzoylacetate.[3]

(1) Supported at Duke University by Public Health Service Research Grant No. CA-04455 from the National Cancer Institute, and at Virginia Polytechnic Institute by Public Health Service Research Grant No. GM 14340 from the National Institute of General Medical Sciences.

(2) R. L. Gay and C. R. Hauser, *J. Amer. Chem. Soc.*, **89**, 1647 (1967).

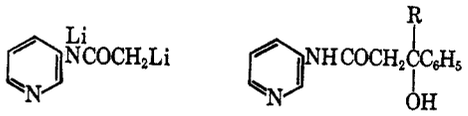
(3) O. Seide, *Ber.*, **58**, 352 (1925).

In the benzoylation of I″, the yield of IV was 56% employing 2 molecular equiv of I″ to one of the ester, but only 30% using molecular equivalents of two reactants; under the latter condition, about half of I″ was presumably neutralized in converting the monolithio salt of IV into its dilithio salt.[4] In connection with identification, β-ketoamide IV was cyclized by means of sulfuric acid to form the rearranged pyrido pyrimidone V.

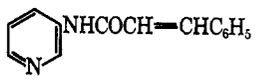


IV V

Similarly, 3-acetamidopyridine was converted by *n*-butyllithium into dilithioamide VI″, which was condensed with benzaldehyde and benzophenone to form adducts VIIa and VIIb, respectively. In contrast to the isomeric adduct IIa, VIIa underwent dehydration with cold sulfuric acid to form the unsaturated amide VIII, which was independently synthesized from 3-aminopyridine and cinnamoyl chloride.



VI″ VIIa, R = H; b, R = C_6H_5



VIII

Experimental Section

Infrared spectra were measured either as mulls in Nujol and hexachlorobutadiene (Perkin Elmer spectrophotometer-Model 137) or as solutions in chloroform (Beckman IR-5A spectrophotometer). Microanalyses were carried out by Jannsen Pharmaceutica, Beerse, Belgium, and also by Galbraith Laboratories, Knoxville, Tenn. Melting points were recorded on a Thomas-Hoover capillary melting point apparatus and are uncorrected unless otherwise stated.

Preparation of 1,3-Dilithio Salt I″.—This was effected by adding, during 10–15 min, a slight excess of 2 molecular equiv of a *ca.* 1.6 M solution of *n*-butyllithium in hexane[5] to a stirred solution of 2-acetamidopyridine in dry tetrahydrofuran (THF) at 0° under nitrogen. Addition of the first molecular equivalent of *n*-butyllithium afforded an amber solution which became reddish orange toward the end of the addition. The resulting solution was then stirred for 15–30 min before addition of the appropriate electrophilic compound.

Addition Reaction of I″ with Benzaldehyde.—Freshly distilled benzaldehyde (5.3 g, 0.05 mol) was added during 3 min to the dilithioamide solution prepared from 2-acetamidopyridine (6.8 g, 0.05 mol), THF (100 ml), and *n*-butyllithium in hexane (66.7 ml). The resulting turbid, pale yellow solution was stirred at 0° for 0.5 hr and then poured into stirred water (100 ml) to furnish a cream precipitate which quickly dissolved. The dark yellow organic layer was combined with an ether extract (50 ml) of the almost colorless aqueous layer and allowed to evaporate thus affording a mixture of a brownish orange oil together with a yellow solid. This mixture was dissolved in boiling toluene and, on cooling, the toluene solution deposited the carbinol-amide IIa (6.68 g, 55%) as a pale cream solid, mp 105.5–106.5°. Recrystallization (chilling) from acetonitrile provided an analytical

(4) See C. R. Hauser, F. W. Swamer, and J. T. Adams, "Organic Reactions," **8**, 59 (1954).

(5) Used as supplied by Foote Mineral Co., Exton, Pa.