(dec.), 72% yield. The compound was recrystallized from water and dried *in vacuo* at 78° over phosphoric anhydride; m.p. 262–263° (dec.).

Anal. Calcd. for $C_{13}H_{20}N_2O_4S$: C, 52.0; H, 6.71; N, 9.33. Found: C, 52.1; H, 6.76; N, 9.22.

(b) XIX from N^{α}-tosyl-DL-lysine. N^{α}-tosyl-DL-lysine, 0.765 g. (2.55 mmol.), was esterified[6] by heating at 100° for 3 hr. with 12 ml. of benzyl alcohol saturated with dry hydrogen chloride, to give 0.860 g. of XIX, m.p. 163–165° (with softening at 160–163°), 79% yield. XIX was recrystallized from 2*N* hydrochloric acid and dried *in vacuo* at 78° over phosphoric anhydride and potassium hydroxide, m.p. 176–177°.

Anal. Calcd. for $C_{20}H_{27}N_2O_4SCl$: C, 56.3; H, 6.38; N, 6.56. Found: C, 55.6; H, 6.36; N, 6.57.

N^{α}-Tosyl-L-lysine methyl ester (XX). Dry ammonia was passed through a solution of 0.431 g. of XVI (1.23 mmol.), m.p. 148–150°, in dry chloroform for 15 min. The solution was cooled in an ice bath to 0°. The reaction mixture was then washed twice with 25 ml. of water. After drying over anhydrous sodium sulfate, the chloroform was evaporated under reduced pressure (30–40°). The residual oil solidified on cooling and triturating with petroleum ether to give 0.306 g. of XX, m.p. 93–95°, after drying *in vacuo* over calcium chloride, 79% yield. Recrystallization of XX from benzene–petroleum ether gave needle clusters, m.p. 93–95°. The recrystallized XX was dried *in vacuo* at room temperature over paraffin chips for 24 hr.; $[\alpha]_D^{23}$ −5.4° (*c* 2.5, 95% ethanol).

Anal. Calcd. for $C_{14}H_{22}N_2O_4S$: C, 53.5; H, 7.05; S, 10.2; NH_2—N, 4.46. Found: C, 53.7; H, 7.06; S, 10.5; NH_2—N, 4.26.

N^{α}-Tosyl-DL-lysine benzyl ester (XXI). A suspension of 1.08 g. of XVII (2.29 mmol.) in 25 ml. of chloroform was cooled to −15° in an ice-salt mixture. Dry ammonia was passed into the suspension for 10 min. The resulting clear solution was allowed to warm slowly to room temperature and the ammonium bromide which precipitated from the reaction mixture was removed by filtration and washed with chloroform. The slightly turbid filtrate and washings were combined and extracted twice with 15 ml. of water. The chloroform solution was dried over anhydrous sodium sulfate and then taken to dryness under reduced pressure (35–40°). The residual oil crystallized when it was covered with petroleum ether and cooled, to give 0.80 g. of XXI, m.p. 103–104°, 90% yield. XXI was recrystallized from chloroform–petroleum ether, m.p. 104°.

Anal. Calcd. for $C_{20}H_{26}N_2O_4S$: C, 61.5; H, 6.71; NH_2—N, 3.59. Found: C, 61.3; H, 6.61; NH_2—N, 3.52.

Attempts to prepare N^{α}-acetyl-DL-lysine methyl ester by esterification of N^{α}-acetyl-DL-lysine[16] with diazomethane[17] yielded a basic oil which, as judged from the elemental analysis, appeared to be N^{α}-acetyl-N^{ϵ}-methyl-DL-lysine methyl ester.

Anal. Calcd. for $C_{10}H_{20}N_2O_3$: C, 55.5; H, 9.32; N, 12.96. Found: C, 54.9; H, 8.99; N, 12.84.

N-Methylation of primary amines by diazomethane is a known reaction.[25] The reaction here probably proceeds *via* the intermediate formation of N^{α}-acetyl-DL-lysine methyl ester.

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(25) L. I. Smith, *Chem. Revs.*, **23**, 193 (1938).

[CONTRIBUTION FROM THE DEPARTMENT OF CHEMISTRY, STATE UNIVERSITY OF IOWA]

Chemistry of the 1,2,4-Thiadiazine Ring System. II. A New Synthesis of 1,2,4,2*H*-Thiadiazine-3,5(4*H*,6*H*)-dione-1,1-dioxide

BERNARD E. HOOGENBOOM, RICHARD ABBOTT, LOUIS LOCATELL, JR., AND R. L. HINMAN[1]

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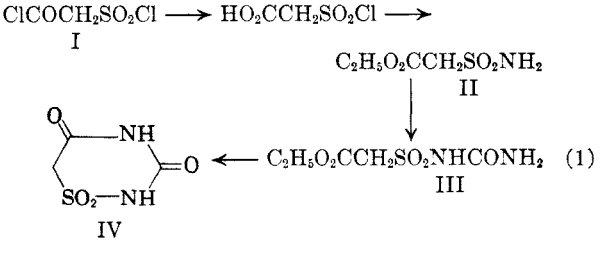
1,2,4,2*H*-Thiadiazine-3,5(4*H*,6*H*)-dione-1,1-dioxide (IV) has been synthesized in 25% over-all yield by the following sequence: sulfoacetic acid, sulfoacetic diacid chloride (I), diphenyl sulfoacetate (V), sulfamylacetamide (VI), carbamylmethanesulfonylurea (VII). The final ring closure of VII to IV was carried out in refluxing pyridine; other bases were ineffective. The same method was used to cyclize β-ureidoethanesulfonamide (XI) to 1,2,4(2*H*)-thiadiazine-3(4*H*,5*H*,6*H*)-one-1,1-dioxide (XII), an analog of dihydrouracil. Attempted ring closure of sulfamylacetylurea (IX) to IV was unsuccessful as were attempts to synthesize IV from sulfamylacetamide by reaction with ethyl carbonate, ethyl chloroformate, or urea

1,2,4(2*H*)-Thiadiazine-3,5(4*H*,6*H*)-dione-1,1-dioxide (IV) has recently been synthesized and its properties have been fully described for the first time.[2] Attention has been called to its similarity to barbituric acid, particularly its pronounced acidity (*p*Ka′ 2.7). The method used is shown in Equation 1. To circumvent difficulties in the conversion of sulfoacetic diacid chloride (I) to ethyl sulfamylacetate (II),[2] an alternative synthesis of IV was sought.



The present paper describes several new approaches to the synthesis of IV, including one new and improved method, summarized in Equation 2. Sulfoacetic diacid chloride (I) was converted to diphenyl sulfoacetate (V) which reacted with liquid ammonia in a sealed tube, forming sulfamylacetamide (VI). (Only the more reactive[2] carboxyl end of

(1) Author to whom inquiries should be addressed. Present address: Union Carbide Research Institute, 32 Depot Plaza, White Plains, N. Y.

(2) R. L. Hinman and L. Locatell, Jr., *J. Am. Chem. Soc.*, **81**, 5655 (1959). Previously reported attempts to synthesize IV are discussed in this reference.

$$ClCOCH_2SO_2Cl \longrightarrow C_6H_5O_2CCH_2SO_3C_6H_5 \longrightarrow$$
I V

$$NH_2COCH_2SO_2NH_2$$
VI

$$IV \xleftarrow[\text{2. Dowex-50}]{\text{1. refluxing pyridine}} NH_2COCH_2SO_2NHCONH_2 \quad (2)$$
VII

V reacted with liquid ammonia at atmospheric pressure. Phenyl carbamylmethanesulfonate (VIIIa) was the product. In like manner liquid methylamine at atmospheric pressure changed V to VIIIb.[3])

$$C_6H_5O_2CCH_2SO_3C_6H_5 \xrightarrow{RNH_2} \begin{cases} \xrightarrow[\text{sealed tube}]{} VI \\ \xrightarrow[\text{atm. press.}]{} RNHCOCH_2SO_3C_6H_5 \end{cases}$$
V

VIIIa. R = H
b. R = CH_3

Sulfamylacetamide (VI) was readily converted to carbamylmethanesulfonylurea (VII), by taking advantage of the fact that sulfonamides are more reactive toward potassium cyanate in basic solution than are carboxamides.[4] The conditions for the reaction were similar to those used previously[2] for the conversion of ethyl sulfamylacetate (II) to carbethoxymethanesulfonylurea (III).

Initial attempts to cyclize VII to IV were unsuccessful. From treatment of VII with ethanolic sodium ethoxide (the method used for the cyclization of III to IV), or with sodamide in liquid ammonia, ethyl ether, or *N,N*-dimethylaniline, only salts of VII were obtained. Refluxing an aqueous solution of VII brought about hydrolysis to sulfamylacetamide (VI), as did the presence of moisture under the other conditions just described. Simple heating of a carefully dried sample of VII led only to charring and general decomposition, as did heating a mixture of VII and *N,N*-dimethylaniline at the reflux temperature. Cyclization was finally achieved by refluxing a solution of VII in pyridine, from which the pyridine salt of 1,2,4,2*H*-thiadiazine-3,5(4*H*,6*H*)-dione-1,1-dioxide was isolated.[5] The free acid (IV) was obtained from the salt with the aid of a cationic exchange resin. The product obtained by this synthesis was identical to that previously reported,[2] and confirms the structure assigned to IV in the previous paper of this series. Each step of the synthesis gave a good yield of product (>65%); the over-all yield of IV from sulfoacetic acid was 25%.

The success of this synthesis suggested that sulfamylacetylurea (IX), the isomer of VII, might be induced to cyclize under similar conditions, as shown in Equation 3. Compound IX was prepared from the known[6] chlorosulfonylacetylurea by reaction with ammonia. However, none of the desired product (IV) was obtained from refluxing solutions

$$I \longrightarrow ClSO_2CH_2CONHCONH_2 \longrightarrow NH_2SO_2CH_2CONHCONH_2 \xrightarrow{/-/-/} IV \quad (3)$$
IX

of IX in anhydrous pyridine. Heating IX in refluxing nitrobenzene, diphenyl ether, or in the absence of solvents led only to extensive decomposition. The use of aqueous ethanolic sodium carbonate, a technique which has been used[7] for the synthesis of sulfonylureas from sulfonamides and urea, was also unsuccessful.

Although cyclization of IX to IV could not be effected, refluxing pyridine was employed successfully in the related cyclization of β-ureidoethanesulfonamide (XI) to 1,2,4,2*H*-thiadiazine-3(4*H*,5*H*,-6*H*)-one-1,1-dioxide (XII), an analog of dihydrouracil. Attempts to bring about the cyclization of XI to XII in triethylamine or tri-*n*-butylamine were unsuccessful.

$$\overset{-}{Cl}\overset{+}{N}HC_3H_2CH_2SO_2NH_2 \longrightarrow$$
X

$$NH_2CONHCH_2CH_2SO_2NH_2 \longrightarrow \begin{array}{l} \text{NH} \\ \text{CO} \\ SO_2\text{—NH} \end{array}$$
XI XII

Compound XI was prepared from tauramide hydrochloride (X) by reaction with potassium cyanate. The structure of XI was assigned on the basis of the fact that amines react readily with potassium cyanate in acidic media, whereas sulfonamides are converted to sulfonylureas in basic media. The conversion of sulfanilamide to *p*-ureidobenzenesulfonamide has been effected in the same way.[4] The infrared spectrum of XI has a strong band at 1648 cm.$^{-1}$ which is in the region where *N*-alkylureas

(3) Phenyl methanesulfonate ($C_6H_5O_3SCH_3$) was recovered unchanged after having been heated with liquid ammonia in a sealed tube at 75°, conditions under which V is converted to VI. Ammonolysis of the sulfonate group in V is apparently made possible by the activating effect of the carbonyl group in the α position.

(4) F. Kurzer, *Chem. Revs.*, **50**, 1 (1953). In our hands acetamide and benzamide did not react with potassium cyanate under the conditions employed for the reaction of sulfamylacetamide and ethyl sulfamylacetate.[2] Further proof for the structure of VII was obtained by preparation of the isomeric sulfamylacetylurea (IX) by an established method (see below).

(5) The cyclization probably takes place by way of an intermediate of the sulfonylisocyanate type (NH_2COCH_2-SO_2NCO). Sulfonylisocyanates have been prepared [O. C. Billeter, *Ber.*, **37**, 690 (1904); J. R. Geigy, Brit. Pat. **604,259**, June 30, 1948; *Chem. Abstr.*, **43**, 1061 (1949)], and isocyanates in general are known to react with amides to form substituted ureas [see for example: P. F. Wiley, *J. Am. Chem. Soc.*, **71**, 1310, 3746 (1949)]. A sulfonylisocyanate of the type shown would react rapidly with water to give sulfamylacetamide, in accord with the facile conversion of the sulfonylurea to the amide by trace amounts of water in the pyridine.

(6) K. Bodendorf and N. Senger, *Ber.*, **72**, 571 (1939). These authors reported the synthesis of (*N*-phenylsulfamyl)-acetylurea by a method similar to that used for IX. They were unable to cyclize it by heating the dry solid.

(7) E. Haack, U. S. Patent **2,385,571**, Sept. 25, 1945; *Chem. Abstr.*, **40**, 603 (1946).

absorb.[8a] Carbonyl absorption in sulfonylureas occurs at higher frequencies, as shown by carbamylmethanesulfonylurea (VII) (1686 cm.$^{-1}$), XII (1700 cm.$^{-1}$), and methanesulfonylurea (1710 cm.$^{-1}$).[8b]

The product of cyclization (XII) is a considerably weaker acid than IV, since the former was isolated directly from the pyridine solution, rather than in the form of the pyridine salt. Compound XII could nevertheless be titrated to a satisfactory end point with dilute sodium hydroxide. The neutralization equivalent agreed closely with that calculated for formula XII. The infrared spectrum of XII has a strong band at 1700 cm.$^{-1}$, near that of methanesulfonylurea, and at 1148 and 1324 cm.$^{-1}$. The spectrum of methanesulfonylurea has a band at 1150 cm.$^{-1}$ as well as two bands in the sulfonyl region at 1318 and 1331 cm.$^{-1}$

Although the methods commonly used for the preparation of barbituric acid (*i.e.*, urea and the diester or diacid chloride of malonic acid) cannot be used for the synthesis of 1,2,4,2*H*-thiadiazine-3,5-(4*H*,6*H*)-dione-1,1-dioxide (IV),[2,6] we had hoped that the related reaction of sulfamylacetamide (VI) with a suitable derivative of carbonic acid might provide a more direct route to IV. However, neither refluxing a large excess of ethyl carbonate with VI in the presence of sodium (mol. ratio of VI/Na = 1/2) nor of ethyl chloroformate with VI in the presence of sodium ethoxide (mol. ratio of ethyl chloroformate/sodium ethoxide/VI = 1/2/1) produced any of the desired product. When sulfamylacetamide was fused with urea, only unchanged VI and a higher melting solid, probably biuret, were obtained. These experiments are summarized in equation 4:

$$\mathrm{NH_2COCH_2SO_2NH_2} + \begin{matrix}\mathrm{C_2H_5O_2COC_2H_5}\\ \text{or}\\ \mathrm{C_2H_5O_2CCl}\\ \text{or}\\ \mathrm{NH_2CONH_2}\end{matrix} \longrightarrow \mathrm{IV} \quad (4)$$

EXPERIMENTAL[9]

Diphenyl sulfoacetate (V). A mixture of 96 g. (0.54 mol.) of chlorosulfonylacetyl chloride[2] and 105 g. (1.13 mol.) of freshly distilled phenol was heated for 10–12 hr. in an oil bath held at a temperature of 125°. After cooling, the mixture crystallized, and the solid material was recrystallized from a mixture of benzene and petroleum ether, yielding 129 g. (82%) of pinkish white crystals, m.p. 77.5–80°. Recrystallization from benzene gave 86 g. (54%) of a white amorphous powder m.p. 80–81° (lit.[10] m.p. 77.5°). (Found: C, 57.90; H, 4.32. Calcd. for $C_{14}H_{10}O_5S$: C, 57.65; H, 4.11.)

Phenylcarbamylmethanesulfonate (VIIIa). To approximately 75 ml. of liquid ammonia in a Dewar flask was added in portions and with stirring 26.3 g. (0.09 mol.) of diphenyl sulfoacetate. The mixture was stirred gently for a period of 24 hr., and then transferred to a flask, rinsing with portions of absolute ethanol. After the excess ammonia had been removed by slight warming of the mixture, the remaining alcoholic solution was heated to boiling, filtered, and then chilled. The white crystals which separated were filtered and washed with several small portions of ether. In this way 13.2 g. (68%) of product m.p. 97.5–99° was obtained. (Found: C, 44.75; H, 4.40. $C_8H_9NO_4S$ requires: C, 44.64; H, 4.22.)

Phenyl N-methylcarbamylmethanesulfonate (VIIIb). This compound was prepared by the reaction of diphenyl sulfoacetate and liquid methylamine in the same manner as the above experiment. It was also synthesized *via* ethyl phenyl sulfoacetate in the following manner. A mixture of 25.2 g. (0.24 mol.) of carbethoxymethanesulfonylchloride,[2] and 14.0 g. (0.15 mol.) of freshly distilled phenol was heated for a period of 9 hr. in an oil bath held at a temperature of 120–130°. After cooling, the mixture was dissolved in 100 ml. of absolute ethanol. The ethanolic solution was heated to boiling, decolorized, filtered, and chilled. Even by the addition of petroleum ether, no crystallization could be induced. The solvents were removed leaving an oil which would not crystallize. The oil, presumably *ethyl phenyl sulfoacetate*, was taken up in a small amount of ethanol and added slowly to approximately 60 ml. of liquid methylamine contained in a tube immersed in a bath of Dry Ice and isopropyl alcohol. The mixture was allowed to stand with occasional stirring for a period of 40 hr. It was then rinsed into a flask and warmed slightly to remove excess methylamine. The residual ethanolic solution was heated to boiling, filtered, and allowed to cool. By filtration, 20.2 g. (66% crude) of a light amorphous solid was obtained. Recrystallization of this material from CCl_4 produced a white crystalline solid, m.p. 82–83°. (Found: C, 46.95; H, 4.94. $C_8H_{11}NO_5S$ requires: C, 47.20; H, 4.84.)

Sulfamylacetamide (VI). A solution of 25.0 g. (0.086 mol.) of diphenyl sulfoacetate in approximately 30 ml. of liquid ammonia contained in a sealed tube was heated at a temperature of 75° for a period of 17 hr. At the end of the heating period the tube was cautiously opened and its contents were transferred to a flask, rinsing with small portions of methanol. After evaporation of excess ammonia by slight warming, the methanol solution was heated to boiling, filtered, and allowed to cool thoroughly. By filtration of the cold mixture, 10.9 g. (92% crude) of a light tan solid, m.p. 132–135°, was obtained. Two recrystallizations from absolute ethanol yielded white granular crystals, m.p. 134–135°. (Found: C, 17.62; H, 4.37; N, 20.33. $C_2H_6N_2O_3S$ requires: C, 17.40; H, 4.35; N, 20.03.)

Carbamylmethanesulfonylurea (VII). A mixture of 6.8 g. (0.05 mol.) of sulfamylacetamide, 4.2 g. (0.05 mol.) of potassium cyanate, and 250 ml. of absolute ethanol was refluxed on a steam bath for 4 hr. After heating for approximately 0.5 hr. a voluminous white precipitate began to separate, causing considerable bumping of the mixture. At the end of the reflux period, the mixture was thoroughly chilled and then filtered. The crystalline white solid was washed with several portions of ether and when completely free of solvents weighed 9.2 g. (83%) in crude form. Recrystallization from 95% ethanol yielded 8.3 g. (74%) of purified potassium carbamylmethanesulfonylureide. (Found: C, 16.38; H, 2.77; N, 19.60. $C_3H_5KN_3O_4$ requires: C, 16.45; H, 2.74; N, 19.20.) A solution of 3.0 g. (0.022 mol.) of potassium car-

(8) (a) J. L. Bowin and P. A. Bowin, *Can. J. Chem.*, **32**, 561 (1954). (b) Methanesulfonylurea was pelleted in potassium bromide. The other compounds were in Nujol mull. Additional peaks appear in the spectra of many of the sulfonylureas when obtained from potassium bromide pellets. We have therefore relied almost entirely on Nujol mulls in this work. The two peaks at 1714 and 1698 cm.$^{-1}$ reported[2] for compound IV were obtained in Nujol. In potassium bromide a single band at 1710 cm.$^{-1}$ was observed. Whether this is due to reactions which take place under the conditions of pelleting, or whether it reflects changes in the per cent enolization of the compound is not known.

(9) Melting points and boiling points are uncorrected. Infrared spectra were obtained with a Model 21 Perkin-Elmer recording spectrophotometer equipped with a sodium chloride prism.

(10) R. Vieillefosse, *Bull. soc. chim. France*, 6, 34 (1939).

bamylmethanesulfonylureide in 20 ml. of distilled water was passed through a column of Dowex-50 resin (acid form), and eluted with water. The strongly acidic portion of the eluant was evaporated under a jet of filtered air to about one fourth its original volume. After thorough chilling of the concentrated solution a white crystalline solid separated. This material, when filtered and washed with an ethanol ether mixture and, finally, with ether, weighed 2.0 g. (80% conversion) and melted with decomposition at 165–170°. Recrystallization from absolute ethanol yielded soft white crystals, m.p. 171–172° (dec.). (Found: C, 19.95; H, 3.66; N, 23.63. $C_3H_7N_3O_4S$ requires: C, 19.91; H, 3.87; N, 23.22.)

Pyridinium 1,2,4,2H-thiadiazine-3,5(4H,6H)-dione-1,1-dioxide. A mixture of 1.0 g. (0.0065 mol.) of carbamylmethanesulfonylurea and 20 ml. of anhydrous, freshly distilled pyridine, protected from atmospheric moisture by means of either a calcium chloride or barium oxide drying tube, was refluxed for a period of 2.5 hr. The solid was completely dissolved after 0.5 hr. refluxing. After the reflux period the mixture was chilled in an ice bath. By the addition of a small amount of absolute ether a granular white solid was made to precipitate. This material, after filtration and washing with several portions of an ethanol ether mixture, weighed 0.91 g. (68% crude) and melted at 172–179° (dec.). Two recrystallizations from absolute ethanol yielded a pure product, m.p. 176–178° (dec.). (Found: C, 39.56; H, 3.93; N, 17.47. $C_8H_9N_3O_4S$ requires: C, 39.55; H, 3.71; N, 17.47.)

1,2,4,2H-Thiadiazine-3,5(4H,6H)-dione-1,1-dioxide (IV). Pyridinium 1,2,4,2*H*-thiadiazine-3,5(4*H*,6*H*)-1,1-dioxide (4.3 g., 0.018 mol.), obtained in the manner described above, was dissolved in approximately 20 ml. of distilled water and eluted from a column of Dowex-50 cationic exchange resin. The portion of the eluant strongly acidic to litmus was evaporated on a steam bath with a gentle jet of filtered air. The light tan material thus obtained was recrystallized from absolute ethanol, whereby a total of 2.6 g. (88% conversion from the pyridinium salt) of white, powdery material m.p. 226–227° (dec.) was obtained. This material was identical in m.p. and infrared spectrum so that previously reported.[2]

Sulfamylacetylurea (IX). Chlorosulfonylacetyl chloride (17.7 g., 0.1 mol.), was added to 6 g. (0.1 mol.) of urea and the mixture was stirred until it became a viscous mass. This salve-like material, which contained chlorosulfonylacetylurea,[6] was allowed to stand in a vacuum desiccator over potassium hydroxide for 3 days and was then dissolved in 100 ml. of dry tetrahydrofuran. To this solution was added 3.4 g. (0.2 mol.) of ammonia in 100 ml. of tetrahydrofuran. After 1 hr. the ammonium chloride formed was removed by filtration. Evaporation of the filtrate yielded about 10 g. (56%) of crude product. Three recrystallizations from ethanol water mixtures gave the pure compound, m.p. 185–186°. (Found: C, 19.97; H, 3.60; N, 23.75. $C_3H_7N_3O_4S$ requires: C, 19.89; H, 3.87; N, 23.21.)

β-Ureidoethanesulfonamide (XI). A solution of 2.8 g (0.035 mol.) of potassium cyanate in 6 ml. of water was added to a solution of 5.5 g. (0.035 mol.) of β-aminoethanesulfonamide[11] hydrochloride in 25 ml. of 80% ethanol, and the resulting solution was allowed to stand for 4 hr. at room temperature. It was then evaporated to dryness under reduced pressure. The residue was extracted thrice with 20-ml. portions of absolute ethanol and twice with 15-ml. portions of 95% ethanol. Evaporation of the combined extracts left a clear paste, which crystallized when cooled on Dry Ice. Two recrystallizations from absolute ethanol and one from 95% ethanol produced 1.5 g. (26%) of white needles, m.p. 144–145°. The product was soluble in water and insoluble in chloroform. (Found: C, 21.83; H, 5.30; N, 25.14. $C_3H_9N_3O_3S$ requires: C, 21.52; H, 5.43; N, 25.14.)

1,2,4,2H-Thiadiazine-3(4H,5H,6H)-one-1,1-dioxide (XII). One g. (0.006 mol.) of β-ureidoethanesulfonamide was subjected to the procedure described for the cyclization of VII, except that the volume of ether added to precipitate the product was equal to the volume of pyridine used for the reaction. In this way 0.6 g. (67%) of white crystals m.p. 272–273° (dec.), was obtained after recrystallization from absolute ethanol. [Found: C, 23.93; H, 4.10; N, 18.49; neut. equiv. (by potentiometric titration) 149. $C_3H_6N_2O_3S$ requires: C, 23.99; H, 4.03; N, 18.66; neut. equiv. 150.]

Methanesulfonylurea. This compound was prepared from methanesulfonamide[12] and potassium cyanate by the method described above for carbamylmethanesulfonylurea (VII). After two recrystallizations from absolute ethanol, the white crystals melted at 153–5°. (Found: C, 17.43; H, 4.53; N, 20.24; S, 23.13. $C_2H_6N_2O_3S$ requires: C, 17.39; H, 4.38; N, 20.28; S, 23.21.)

The infrared spectrum of a sample in potassium bromide showed strong absorption bands at 1710 (C=O), 1331, 1318, and 1157 cm.$^{-1}$ (SO_2 of $-SO_2N-$).

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(11) Prepared from taurine by the method of R. Winterbottom, J. W. Clapp, W. H. Miller, J. P. English, and R. O. Roblin, Jr., *J. Am. Chem. Soc.*, **69**, 1393 (1947). Although either the phthalyl or benzoyl group could be used, as described by these authors, to protect the amino group of taurine, we found that the latter gave better results. We also used a refluxing mixture of phosphorus oxychloride (instead of thionyl chloride) and a suspension of the sodium salt of *N*-benzoyltaurine in benzene to prepare β-benzamidoethanesulfonyl chloride. In this case the yield was low.

(12) L. Field and F. A. Greenwald, *J. Am. Chem. Soc.*, **75**, 934 (1953).