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A Convenient Synthesis of 1,2-Naphthalic Anhydride[1]

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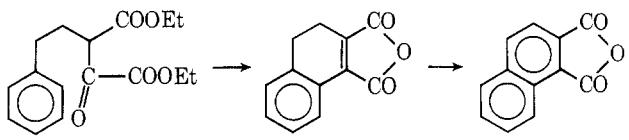
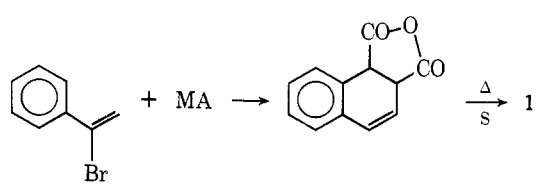
1,2-Naphthalic anhydride (**1**) has long been used for the elaboration of compounds in the benz[*a*]anthracene series.[3] An early synthesis of **1** involved cyclization of ethyl 3-carboethoxy-2-oxo-5-phenylpentanoate (**2**) to 3,4-dihydronaphthalene-1,2-dicarboxylic acid anhydride (**3**) followed by aromatization by heating with sulfur.[4] Later, 1,2-dihydronaphthalene-1,2-dicarboxylic anhydride (**4**), together with a small amount of **1**, were prepared by heating α-bromostyrene (**5**) with maleic anhydride[5] but no developmental work on this route to **1** has been reported. An attempt to convert β-bromostyrene to **4** by heating with maleic anhydride failed.[6] We have tried to use α-chlorostyrene instead of **5** but obtained none of the desired **4**. In this note we record our observations on an improved preparation of **1** by the styrene route.



2 **3** **1**

We have converted styrene dibromide to **5**, by using phase transfer catalysis.[7] In the final dehydrogenation of **4** by heating with sulfur, we have found that the **1** produced always contains some sulfur compound which is not removed by recrystallization. However, by boiling **1** in aqueous alkali, acidification, and cyclization to the anhydride pure sulfur-free **1** can be obtained in satisfactory overall yield.



5 **4**

In our opinion it is much easier to prepare quantities of **1** by the styrene route than by the earlier cyclization of the keto diester[4] as it is relatively simple to carry out the reactions involved on a large (2–3 mol) scale.

Experimental Section

α-Bromostyrene (5). In the best experiment, a solution of 336 g of bromine in 250 ml of $CHCl_3$ was added to a cooled (ca. 5 °C) solution of 208 g of freshly distilled styrene in 250 ml of $CHCl_3$. The temperature was not allowed to rise above 15 °C but cooling to 5 °C near the end is not advisable because the product will crystallize prematurely. After all of the bromine was added the reaction mixture was allowed to stand at room temperature for 1 h. The solvent was then removed to constant weight on a rotary evaporator. The yield of styrene dibromide is practically quantitative.

In the best of many runs, a solution of the crude styrene dibromide thus obtained in 800 ml of benzene was added fairly rapidly to a well-stirred mixture at 70 °C of 528 g of KOH, 800 ml of water, and 10 g of Aliquat 336.[8] The mixture was held at reflux for 3 h.[9] After cooling, the benzene layer was washed with water and saturated salt solution, passed through a cone of anhydrous $MgSO_4$, and fractionally distilled to afford 301 g (82.7%) of **5**, bp 80–83 °C (10 mm). In other similar runs which varied in detail yields of 52–79% were obtained. The yield is somewhat dependent on the skill of the operator in conducting a rapid distillation. If the distillation is conducted too slowly thermal decomposition occurs and the yield is lower. The use of a hot salt bath and a free flame to hasten distillation is recommended.[10]

1,2-Naphthalic Anhydride (1). In the best of many runs, a solution of 408 g of **5** and 326 g of maleic anhydride in 1.5 l. of xylene was refluxed for 48 h (to ensure escape of most of the HBr formed). The solvent was then distilled and the residue was rapidly vacuum distilled to yield crude **4**, bp 150–165 °C (1 mm), which was immediately placed in a Claisen flask with 71 g of sulfur. The mixture was heated rapidly to 230–235 °C with a salt bath and held at this temperature for 1 h, then at 250 °C for 1 h. Rapid vacuum distillation afforded 300 g (69%) of crude **1**, mp in the 160–164 °C range. The yields in this step varied from 63 to 75% (small-scale run). This product contains sulfur-containing impurities and cannot be effectively purified by crystallization. The best method of purification involved heating at reflux for 4 h 131.5 g of crude **1** with excess 20% NaOH. On acidification of the filtered solution followed by heating of the acid with 1 l. of $(Ac)_2O$, there was obtained 116.9 g (89%) of **1** in three crops. The melting points lay in the 165–167 °C range and this material was suitable for further work. No sulfur was present as shown by the absence of *m/e* over 198.

Registry No.—**1**, 5343-99-7; **4**, 60224-29-5; **5**, 98-81-7; styrene, 100-42-5; styrene dibromide, 93-52-7; maleic anhydride, 108-31-6.

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The Chemistry of a Ketene–Sulfur Dioxide Adduct. 3. Reactions with Azines

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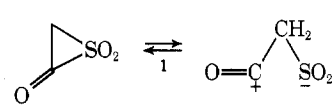
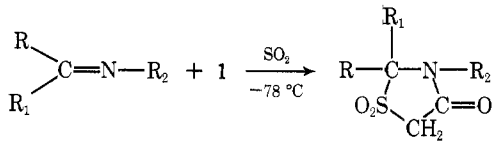
The cycloadditions of imines with ketene–sulfur dioxide adduct (**1**)[1b,c] to give substituted thiazolidin-4-one 1,1-dioxides was described in earlier publications,[1a,b] and extended by Kagan et al.[2] to substituted ketenes. More recently, Bellus reported that ketenes containing carbanion stabilizing substituents, generated in situ in the presence of sulfur dioxide,



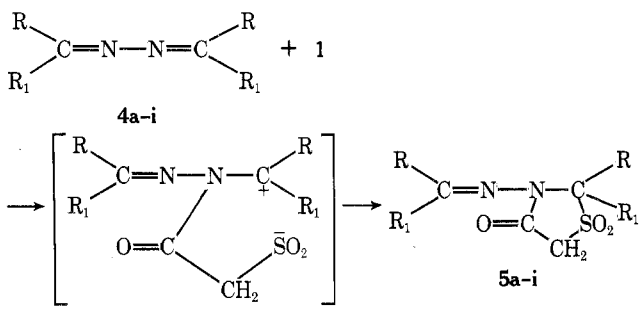
also gave the corresponding substituted thiazolidin-4-one 1,1-dioxides, when treated with benzylideneaniline.[3]

The reaction of imines with 1 was first shown to occur with benzylideneaniline,[1a,b] but we have since found that other compounds containing the imino function also react readily, thereby illustrating the generality of this reaction.



2, R = C_6H_5; R_1 = H; R_2 = CH_3
3, R, R_1 = 9-fluorenyl; R_2 = C_6H_5

In order to further extend the reactions of the ketene–sulfur dioxide adduct to other dipolarophiles, we have examined the reactions of both aldehyde and ketone azines (**4a–i**) with **1** at −78 °C, in liquid sulfur dioxide, and observed the following results.



4a–i

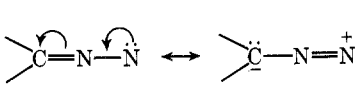
5a–i

a, R = 2-furyl	R_1 = H
b, R = 5-methyl-2-furyl	R_1 = H
c, R = 2-thienyl	R_1 = H
d, R = phenyl	R_1 = H
e, R = 3,4-dimethoxyphenyl	R_1 = H
f, R = 3-methoxyphenyl	R_1 = H
g, R = styryl	R_1 = H
h, R = furyl	R_1 = CH_3
i, R = phenyl	R_1 = CH_3

In contrast with two recent reports,[4,5] ketone azines underwent cyclization with **1**. This observation not only supports the existence of a ketene–sulfur dioxide adduct,[1b,c] since diphenyl ketene in ether was found unreactive, but also suggests that **1** is a reactive 1,3-dipolar species.

In all cases (**5a–i**), the reaction took place at only one of the two available azomethine functions. This finding is in agreement with previously unpublished observations that hydrazones from either aldehydes or ketones do not react with **1** under a variety of conditions. Starting material was quantitatively recovered from these reactions along with ketene polymer. The azomethine group of the hydrazone structural unit (>C=N–N–) is inert to **1**, ketenes, and alkoxydiazenium salts, although in the latter case a reaction occurred on the amino nitrogen.[5]

Although the reason for this lack of reactivity has not been established, it is possible that delocalization of the type shown



would decrease both the nucleophilicity of the carbon–nitrogen double bond and the electron deficiency of the carbon. This delocalization is similar to that observed in the anion of hydrazones in Wolff–Kishner reductions. In the azine, once cycloaddition has occurred at one of the azomethine groups, the product is structurally analogous to a hydrazone and therefore capable of similar delocalization.

Aldehyde and ketone azines containing aliphatic substituents polymerized under the conditions used.

Experimental Section

Ir spectra were recorded on a Perkin-Elmer Model 137 Infracord and NMR spectra were recorded on a Varian A-60A spectrometer with tetramethylsilane as an internal standard. All melting points were determined on a Thomas-Hoover Unimelt apparatus and are corrected. Ketene was generated by the pyrolysis of acetone over a nichrome coil at 500–700 °C in a calibrated ketene generator and collected at dry ice–acetone temperature. Anhydrous grade sulfur dioxide was obtained from commercial sources and dried further by first bubbling it through concentrated sulfuric acid and then a column containing calcium chloride, phosphorus pentoxide, and indicator grade calcium sulfate desiccants at which point it was condensed at −78 °C for use. With one exception, **4b**, all aldehyde and ketone azines (**4a–i**) were known compounds.

Reactions of Imines with 1. 3′-Phenyl-4′-oxospiro[fluorene-9,2′-thiazolidine] 1′,1′-Dioxide (3). Fluorenylideneaniline (0.2 g, 0.001 mol) was mixed with 100 ml of liquid sulfur dioxide at −78 °C. Ketene was then generated and bubbled through the suspension for 15 min at a rate of 0.24 mol/h. When the addition was completed, the delivery tube was replaced with a stopper. The suspension was stirred for 1 h at room temperature whereupon excess sulfur dioxide was removed in vacuo and the residue was heated with 15 ml of methanol, separated, washed with cold methanol, and recrystallized from acetic acid to give 0.22 g (80% yield) of **3**, mp 264–265 °C. Anal. Calcd for $C_{21}H_{15}NSO_3$: C, 69.80; H, 4.16; N, 3.88; S, 8.86. Found: C, 69.64; H, 4.14; N, 3.80; S, 9.06. Ir (KBr) 1675 (C=O), 1340 and 1140 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 5.00 (s, 2 H, CH_2), 6.65–7.30 (m, 5 H, NC_6H_5), and 7.30–8.00 (m, 8 H, fluorene nucleus).

2-Phenyl-3-methyl-4-thiazolidinone 1,1-Dioxide (2). Benzylidenemethylamine (12 g, 0.1 mol) was mixed with 100 ml of liquid sulfur dioxide at −78 °C. Ketene was generated and bubbled through the suspension for 30 min at a rate of 0.30 mol/h. The reaction mixture was then treated as described for 3 with the exception that the residue was treated with 800 ml of diethyl ether instead of methanol. The product was recrystallized from ethanol to give pure **2**, mp 121.5–123.5 °C. Anal. Calcd for $C_{10}H_{11}NSO_3$: C, 53.33; H, 4.88; N, 6.22; S, 14.00. Found: C, 53.02; H, 5.18; N, 6.23; S, 14.12. Ir (KBr) 1675 (C=O), 1320 and 1130 cm^{-1} (SO_2); NMR (acetone-d_6) δ 2.90 (s, 3 H, NCH_3), 4.00 (s, 2 H, CH_2), 5.85 (s, 1 H, CH), and 7.50 (m, 5 H, C_6H_5).

5-Methyl-2-furfuraldehyde Azine (4b). A solution of 1.6 g (0.05 mol) of 95% hydrazine in 10 ml of absolute ethanol was added dropwise to a stirred solution of 11.0 g (0.10 mol) of 5-methyl-2-furfuraldehyde in 20 ml of absolute ethanol. The precipitate that formed on cooling was collected and recrystallized from absolute ethanol to give 20.1 g (93% yield) of yellow needles: mp 114–115 °C; ir (KBr) 1630 (C=N) and 1580 cm^{-1} (C=C); NMR ($CDCl_3$) δ 2.45 (s, 3 H, CH_3), 6.10–7.00 (q, 2 H, furyl), and 8.70 (s, 1 H, CH=N). Anal. Calcd for $C_{12}H_{12}N_2O_2$: C, 66.67; H, 5.56; N, 12.96. Found: C, 66.53; H, 5.76; N, 12.71.

General Procedure for the Reaction of 1 with Azines. Sulfur dioxide (25 ml) was condensed at −78 °C into a two-necked flask fitted with a Dewar condenser filled with a dry ice–acetone mixture. Ketene was then generated and bubbled into the flask for 30 min at the rate of 0.25 mol/h. A solution containing the appropriate azine (0.015 mol) in 25 ml of methylene chloride was then added dropwise to the solution of 1 over a period of 10 min. The reaction mixture was then warmed to room temperature, and stirred for 2 h. Methylene chloride and excess sulfur dioxide were removed under reduced pressure and the resulting residue was triturated with absolute methanol or ethanol. The solid that formed was removed by filtration and recrystallized from acetone. Additional product could be obtained by evaporation of the mother liquor. Yields of products ranged between 69 and 97%.

2-(2-Furyl)-3-[(furfurylidene)amino]-4-thiazolidinone 1,1-Dioxide (5a). The reaction was carried out as described for **5a** using 2.82 g (0.015 mol) of **4a**.[6] Methanol was added to the residue at 0 °C to afford a white solid which was recrystallized from acetone to give 4.27 g (97% yield) of **5a**: mp 160–161 °C dec; ir (KBr) 1700 (C=O), 1625 (C=N), 1320 and 1145 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 4.69 (s, 2 H, CH_2), 7.10 (s, 1 H, CH), 6.50–7.90 (m, 6 H, furyl), and 8.30 (s, 1 H, CH=N). Anal. Calcd for $C_{12}H_{10}N_2SO_5$: C, 48.98; H, 3.40; N, 9.52; S, 10.88. Found: C, 49.08; H, 3.38; N, 9.46; S, 11.12.

2-(5-Methyl-2-furyl)-3-[(5-methylfurfurylidene)amino]-4-thiazolidinone 1,1-Dioxide (5b). The reaction was carried out as described for **5a** using 3.24 g (0.015 mol) of **4b**. Addition of methanol to the reaction mixture at 0 °C gave a white solid which was recrys-

tallized from acetone to yield 4.30 g of **5b** (89% yield): mp 161–162 °C dec; ir (KBr) 1725 (C=O), 1625 (C=N), 1320, and 1145 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 2.22 (s, 3 H, CH_3), 2.42 (s, 3 H, CH_3), 4.60 (s, 2 H, CH_2), 6.00–7.00 (m, 4 H, furyl), and 8.15 (s, 1 H, CH=N). Anal. Calcd for $C_{14}H_{14}N_2O_5S$: C, 52.18; H, 4.38; N, 8.69; S, 9.93. Found: C, 52.29; H, 4.51; N, 8.84; S, 9.95.

2-(2-Thienyl)-3-[(thenylidene)amino]-4-thiazolidinone 1,1-Dioxide (5c). The procedure described for **5a** was employed with 3.3 g (0.015 mol) of **4c**.[7] The reaction mixture was treated with methanol at 0 °C to give 4.41 g (90% yield) of **5c** as a white solid which was recrystallized from a 1:1 mixture of acetone–carbon tetrachloride: mp 160–161 °C dec; ir (KBr) 1700 (C=O), 1600 (C=N), 1310, and 1145 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 4.72 (broad s, 2 H, CH_2), 7.00–8.00 (m, 7 H, thienyl and H-2), and 8.60 (s, 1 H, CH=N). Anal. Calcd for $C_{12}H_{10}N_2S_3O_3$: C, 44.17; H, 3.07; N, 8.59; S, 29.45. Found: C, 44.17; H, 3.07; N, 8.43; S, 29.22.

2-(Phenyl)-3-[(benzylidene)amino]-4-thiazolidinone 1,1-Dioxide (5d). This reaction was carried out as described for **5a** using 3.12 g (0.015 mol) of **4d**.[6] Addition of methanol to the reaction mixture, at 0 °C, gave 3.15 g of **5d** (67% yield). The product was recrystallized from a 1:1 mixture of chloroform–acetone: mp 199–201 °C; ir (KBr) 1700 (C=O), 1600 (C=N), 1310, and 1140 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 4.70 (broad s, 2 H, CH_2), 6.85 (s, 1 H, H-2), 7.50 (s, 10 H, phenyl), 8.15 (s, 1 H, CH=N). Anal. Calcd for $C_{16}H_{14}N_2O_3S$: C, 61.15; H, 4.46; N, 8.92; S, 10.19. Found: C, 61.22; H, 4.65; N, 9.10; S, 10.24.

2-(3,4-Dimethoxyphenyl)-3-[(veratrylidene)amino]-4-thiazolidinone 1,1-Dioxide (5e). The same procedure described for **5a** was used with 4.92 g (0.015 mol) of **4e**.[8] Addition of methanol yielded 6.18 g (95% yield) of **5e**. This compound was recrystallized from acetone: mp 164–166 °C; ir (KBr), 1710 (C=O), 1600 (C=N), 1310, and 1140 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 3.8 (s, 12 H, OCH_3), δ_A 4.72, δ_B 4.53 (q, 2 H, J_{AB} = 17 Hz, CH_2), 6.70 (s, 1H, CH), 7.20–7.30 (m, 8 H, phenyl), and 8.25 (s, 1 H, CH=N). Anal. Calcd for $C_{20}H_{22}N_2SO_7$: C, 55.30; H, 5.10; N, 6.45; S, 7.37. Found: C, 55.44; H, 5.15; N, 6.34; S, 7.66.

2-(3-Methoxyphenyl)-3-[(3-methoxybenzylidene)amino]-4-thiazolidinone 1,1-Dioxide (5f). The same procedure described for **5a** was followed with 4.02 g (0.015 mol) of **4f**.[9] The white solid obtained was recrystallized from acetone to give 4.77 g of **5f** (85% yield): mp 155–156 °C; ir (KBr) 1710 (C=O), 1600 (C=N), 1320 and 1135 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ_A 4.88, δ_B 4.70 (q, 2 H, J_{AB} = 17.5 Hz, CH_2), 3.75 (s, 3 H, OCH_3), 6.85 (s, 1 H, CH), 6.90–7.60 (m, 8 H, phenyl), and 8.25 (s, 1 H, CH=N). Anal. Calcd for $C_{18}H_{18}N_2O_5S$: C, 57.75; H, 4.85; N, 7.48; S, 8.55. Found: C, 57.49; H, 4.72; N, 7.28; S, 8.68.

2-*trans*-Styryl-3-[(cinnamylidene)amino]-4-thiazolidinone 1,1-Dioxide (5g). The procedure described for **5a** was followed using *trans*-cinnamaldehyde azine[6] (3.90 g, 0.015 mol). The product obtained was recrystallized from acetone to afford 4.40 g (80% yield) of **5g**: mp 153–155 °C dec; ir (KBr) 1720 (C=O), 1640 (C=C), 1590 (C=N), 1330, and 1140 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ_A 4.77, δ_B 4.58 (9, 2 H, J_{AB} = 17 Hz, CH_2), 6.30–7.83 (m, 15 H, aromatic and olefinic protons), 8.45 (q, 1 H, CH=N). Anal. Calcd for $C_{20}H_{18}N_2SO_3$: C, 65.57; H, 4.95; N, 7.65; S, 8.74. Found: C, 65.27; H, 5.23; N, 7.80; S, 8.80.

2-(2-Furyl)-2-methyl-3-[(α-methylfurfurylidene)amino]-4-thiazolidinone 1,1-Dioxide (5h). This reaction was carried out as described for **5a** using 3.24 g (0.015 mol) of **4h**.[7] The product separated as a white solid and was recrystallized from acetone to afford 4.12 g (85% yield) of **5h**: mp 189–190 °C dec; ir (KBr) 1700 (C=O), 1600 (C=N), 1325, and 1145 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 1.90 (s, 3 H, CH_3), 2.20 (s, 3 H, $=CCH_3$), 4.54 (d, 2 H, CH_2), and 6.50–7.90 (m, 6 H, furyl). Anal. Calcd for $C_{14}H_{14}N_2O_5S$: C, 52.18; H, 4.38; N, 8.28; S, 9.93. Found: C, 52.34; H, 4.50; N, 8.48; S, 9.92.

2-Methyl-2-phenyl-3-[(α-methylbenzylidene)amino]-4-thiazolidinone 1,1-Dioxide (5i). The procedure described for **5a** was carried out using 3.54 g (0.015 mol) of **4i**:[10] mp 174–175 °C; ir (KBr) 1710 (C=O), 1600 (C=N), 1305, and 1145 cm^{-1} (SO_2); NMR (Me_2SO-d_6) δ 2.10 (s, 3 H, CH_3), 2.14 (s, 3 H, $=CCH_3$), δ_A 4.67, δ_B 4.48 (q, 2 H, J_{AB} = 17 Hz, CH_2), and 7.45 (s, 10 H, phenyl). Anal. Calcd for $C_{18}H_{18}N_2O_3S$: C, 63.15; H, 5.30; N, 8.18; S, 9.35. Found: C, 63.44; H, 5.46; N, 8.07; S, 9.60.

Registry No.—**1**, 27393-94-8; **2**, 43056-12-8; **3**, 60253-51-2; **4a**, 5428-37-5; **4b**, 60253-52-3; **4c**, 24523-46-4; **4d**, 588-68-1; **4e**, 17745-86-7; **4f**, 40252-74-2; **4g**, 13362-71-5; **4h**, 24523-53-3; **4i**, 729-43-1; **5a**, 60253-53-4; **5b**, 60253-54-5; **5c**, 60253-55-6; **5d**, 60253-56-7; **5e**, 60253-57-8; **5f**, 60253-58-9; **5g**, 60253-59-0; **5h**, 60253-60-3; **5i**, 60253-61-4; fluorenylideneaniline, 10183-82-1; sulfur dioxide, 7446-09-5; ketene, 463-51-4; benzylidenemethylamine, 622-29-7; hydrazine, 302-01-2; 5-methyl-2-furfuraldehyde, 620-02-0.

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