a clear yellow solution. Deuteration of the reaction mixture with excess D_2O and NMR analysis of the reisolated acid **1** confirmed that the enolate **2** was formed in ca. 80% yield. Some carbonyl addition had also taken place. Since excess *n*-BuLi had to be avoided in view of the troublesome carbonyl addition, we decided to use LDA as base catalyst. At −78 °C in THF and 100% excess LDA the phenoxyacetic acid (**1**) was converted in over 90% yield to the α-lithiocarboxylate **2** as yellow solution, confirmed by NMR analysis of the deuterated reaction mixture.

The reaction of the phenoxy enolate **2** with common electrophiles proceeded smoothly. Discoloration of the characteristic yellow color of the α-lithiocarboxylate **2** took place immediately on addition of the electrophile. Thus, the enolate **2** afforded 2-phenoxypropionic acid (**3a**) with methyl iodide, 2-phenoxy-3-phenylpropionic acid (**3b**) with benzyl bromide, and the diastereomeric 3-hydroxy-2-phenoxy-3-phenylpropionic acids (**3c**) with benzaldehyde in good yields. These condensation products were identified on the basis of literature reported physical constants and NMR and IR spectral data. With acetone as electrophile the enolate **2** gave the unknown 3-hydroxy-3-methyl-2-phenoxybutyric acid (**3d**) in ca. 80% yield, mp 62 °C (from hexane–benzene, as hydrate) and 77 °C (sublimed, as free acid). The structure of **3d** is based on the correct elemental analysis and IR and NMR spectral data.

We are now extending this direct lithiation method to alkoxy-, diaryloxy-, and dialkoxyacetic acids and are planning to utilize these novel enolates as synthons.

Experimental Section

Microanalyses were performed by Galbraith Laboratories Inc., Knoxville, Tenn. Melting points are uncorrected. NMR spectra were run on an Hitachi Perkin-Elmer R-24B instrument and IR spectra on a Perkin-Elmer 237B Infracord. Solvents and reagents were purified and starting materials were prepared and purified according to standard, published procedures.

General Procedure. A dry, 50-mL, two-necked, round-bottom flask, provided with magnetic spinbar, rubber septum, and three-way stopcock, was attached to a nitrogen manifold and flushed with dry nitrogen for at least 5 min while flame-drying. While under a positive nitrogen gas pressure (ca. 50 mm, regulated with a mercury bubbler), the reaction vessel was charged by means of a syringe with 1.62 g (16 mmol) of diisopropylamine (freshly distilled from calcium hydride) and 20 mL of anhydrous THF (freshly distilled from benzophenone ketyl radical). By means of a dry ice–methanol bath the reaction flask was cooled to −78 °C and while stirring vigorously 16 mmol of *n*-butyllithium in *n*-hexane (standardized by acidimetry) was added by means of a syringe. After complete addition (ca. 5 min) the cooling bath was removed and the reaction mixture was allowed to reach room temperature (ca. 30 °C) while stirring. After ca. 10 min the contents were cooled again to −78 °C and by means of a syringe 608 mg (4 mmol) of phenoxyacetic acid in 5 mL of anhydrous THF was added while magnetically stirring. The yellow solution was stirred at −78 °C for 15 min and subsequently ca. 8 mmol (200% excess) of the electrophile was added and allowed to stir at −78 °C for 15–60 min until complete disappearance of the yellow color.

The reaction mixture was poured onto ca. three to five times crushed ice and extracted with 2 × 20 mL of ether and the aqueous layer acidified with 10% hydrochloric acid until pH ca. 3. The product was extracted with 5 × 20 mL of ether, the combined extracts were dried over anhydrous $MgSO_4$, and after rotoevaporation, first at ca. 30 °C (25 mmHg) and finally at ca. 30 °C (1 mmHg), the residue was purified by recrystallization. The individual cases are detailed below. Yields have not been optimized.

2-Phenoxypropionic acid (3a) was prepared in 70% yield by the above procedure, mp 112–114 °C from water (lit.[9] mp 115–116 °C), by adding 1.15 g (8 mmol) of methyl iodide at −78 °C and stirring for 30 min.

2-Phenoxy-3-phenylpropionic acid (3b) was prepared in 50% yield by the above procedure, mp 82 °C from methanol/water (1:2) (lit.[10] mp 81 °C), by adding 1.37 g (8 mmol) of benzyl bromide at −78 °C and stirring for 60 min.

3-Hydroxy-2-phenoxy-3-phenylpropionic acid (3c) was prepared in 50% yield by the above procedure, mp 116–117 °C from hexane/benzene (1:1) (lit.[11] mp 93–94 °C), by adding 0.85 g (8 mmol) of benzaldehyde at −78 °C and stirring for 15 min.

3-Hydroxy-3-methyl-2-phenoxybutyric acid (3d) was prepared in 80% yield by the above procedure, mp 62 °C as hydrate (needles from benzene/hexane) and 77 °C after sublimation, by adding 0.93 g (16 mmol) of acetone at −78 °C and stirring for 15 min. The spectral data are: IR ($CHCl_3$) 3500–2500 (OH and CO_2H) and 1740 cm^{-1} (C=O); ^1H NMR (60 MHz) δ ($CDCl_3$, Me_4Si) 1.45 (6 H, s, CH_3), 4.40 (2 H, s, OH and CO_2H), 4.45 (1H, s, O–C–H), and 6.7–7.4 (5 H, m, C_6H_5); mass spectrum (70 eV) *m/e* (rel) 210 (11.0), 152 (99.6), and 107 (100). Anal. Calcd for $C_{11}H_{14}O_4H_2O$: C, 57.89; H, 7.07. Found: C, 57.75; H, 7.15.

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Registry No.—**3a,** 940-31-8; **3b,** 64682-83-3; **3c,** 64682-84-4; **3d,** 64682-85-5; phenoxyacetic acid, 122-59-8; LDA, 4111-54-0; *n*-butyllithium, 109-72-8; methyl iodide, 74-88-4; benzyl bromide, 100-39-0; benzaldehyde, 100-52-7; acetone, 67-64-1.

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Pyrolysis of *N*-*p*-Toluenesulfonylsulfilimines[1]

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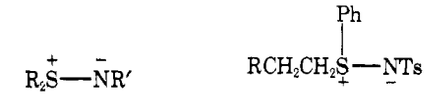
Sulfilimines (**1**) have received considerable attention in the past several years with respect to their synthesis[2] as well as their chemistry.[3] Of particular interest to us have been studies relating to the pyrolysis of sulfilimines. Both Swern[4] and Oae[5] have noted that pyrolysis of sulfilimines in which one of the sulfur substituents contains a β hydrogen (e.g., **2**)



1 **2**

results in a facile elimination yielding an alkene and a sulfenamide. More recently the pyrolysis of *N*-toluenesulfonylsulfilimines in which no β hydrogens are present has been reported.[6] Various solvents were used, and the products obtained depended markedly on the nature of the solvent. We have found[1] that in the absence of solvent the pyrolysis of *N*-toluenesulfonylsulfilimines yields a significantly different mixture of products, and we wish to report those results.

Results and Discussion

The pyrolytic decomposition of *S*,*S*-dimethyl-*N*-*p*-tosylsulfilimine **3** in the absence of solvent at 300 °C yielded the

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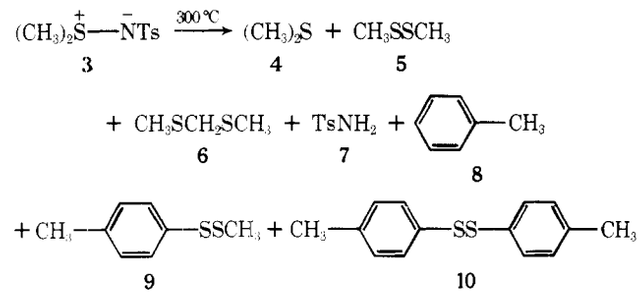
Table I. Pyrolysis of *S,S*-Dimethyl-*N*-tosylsulfilimine (3)[a]

Compd	Registry no.	Yield, mmol	yield (rel),[b] mol	% yield[c]
4	75-18-3	0.339	0.109	11
5	624-92-0	0.288	0.092	18
6	1618-26-4	0.578	0.185	37
7	70-55-3	0.877	0.281	28
8	10888-3	0.500	0.160	16
9	57266-34-9	0.15	0.048	10
10	103-19-5	0.264	0.085	17

[a] Yields are given for pyrolysis of 3.12 mmol of **3** at 300 °C for 1 h. [b] Assuming number of moles of **3** = 1.00. [c] Calculated based on available sulfur and tolyl groups.

Scheme I



products shown in Scheme I. The yields, as noted in Table I, account for 71% of the "*S*-methyls" and 66% of the "tolyl" groups present in the starting sulfilimine. In order to determine if products **8**, **9**, or **10** were arising from thermal decomposition of initially formed toluenesulfonamide (**7**), pyrolysis of this substance was carried out. The only product which could be identified was toluene; disulfides **9** and **10** were not found to be present.

In marked contrast to the complexity of the pyrolysis of **3**, decomposition of *S,S*-diphenyl-*N*-*p*-tosylsulfilimine **11** under identical conditions yielded only diphenyl sulfide, toluenesulfonamide, and a trace of toluene. The low yield of toluenesulfonamide is presumably accounted for by the tarry residue also formed in the pyrolysis which, when totalled with the products isolated, raises the material recovery to over 90%.

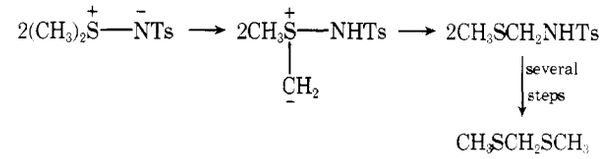
$$\underset{\mathbf{11}}{(C_6H_5)_2S^+-N^-Ts} \xrightarrow{300\ ^\circ C} \underset{(90\%)}{(C_6H_5)_2S} + \underset{(32\%)}{TsNH_2} + \underset{(\text{trace})}{C_6H_5CH_3}$$

The complexity of the pyrolysis of **3** precludes writing a simple mechanism for the process, and in the absence of any direct experimental evidence, mechanistic details must remain speculative. Certain points are worthy of note, however. The relative simplicity of the pyrolysis of **11** suggests that the α hydrogens of **3** play a significant role in the decomposition process. The pyrolysis of the *N*-acetylsulfilimine **12** was found[4] to give, as the major product, the sulfide **6**. It was suggested that abstraction of an α hydrogen followed by a Pummerer type rearrangement would lead to the observed product. Such a process might be proposed for the formation of **6** as the major product in the pyrolysis of **3**, as outlined in Scheme II.

$$\underset{\mathbf{12}}{(CH_3)_2S^+-N^-COCH_3} \xrightarrow{\Delta} \underset{\mathbf{6}}{CH_3SCH_2SCH_3} + \text{other products}$$

One other mechanistic point worthy of mention involves the possible intermediacy of a nitrene (or nitrene-like) species in the decomposition process. Several authors[2c,d,7] have suggested initial formation of nitrenes in sulfilimine pyrolyses, although these species have not been trapped, as they have been in studies of sulfilimine photolysis.[3d] In our work, the formation of dimethyl sulfide might be viewed as arising from heterolytic cleavage of the S-N bond, simultaneously generating a tosylnitrene (or nitrene-like) species. Tolyl radical formation (presumably necessary for formation of toluene and disulfides **9** and **10**) could then be the result of decomposition of this species. As we have no direct experimental evidence bearing on this point, however, we can only speculate as to the intermediacy of a nitrene in our system.

Scheme II



Experimental Section

IR spectra were obtained as $CHCl_3$ or CCl_4 solutions using a Perkin-Elmer Model 137 infracord spectrophotometer. NMR spectra were obtained with a Perkin-Elmer Model R-20 or R-24 spectrometer using $CDCl_3$ as solvent and Me_4Si as internal standard. Mass spectra were obtained on a Hitachi Perkin-Elmer Model RMU-6E spectrometer.[8] Melting points were obtained with a Thomas-Hoover apparatus and are uncorrected. VPC analyses were carried out on a Varian Model 920 chromatograph fitted with a 15 ft., 10% SE-30 on Chromosorb W column.

***S,S*-Dimethyl-*N*-*p*-toluenesulfonylsulfilimine (3).** Following the procedure of Mann and Pope,[9] a solution of chloramine-T (10.0 g, 0.035 mol) in 100 mL of water was chilled in ice and shaken with 2.5 g (3.0 mL, 0.040 mol) of dimethyl sulfide. The white solid which formed was filtered, washed with cold water, and allowed to air dry. Recrystallization with benzene yielded 5.00 g (53%) of the pure product as white needles, mp 157–158 °C (lit.[10] mp 157–158 °C).

Pyrolysis of 3. Crystalline sulfilimine (0.72 g, 3.12 mmol in a typical run), contained in either a Pyrex or a procelain boat, was placed in a 25 mm o.d. Vycor tube horizontally mounted and connected to a cold trap. The system was flushed with N_2 and then heated to 300 °C for 1 h with a tube furnace (Lindberg "Hevi-Duty"). Throughout the heating period a flow of ca. 30 mL/min of N_2 was maintained. Volatile products were collected by cooling the trap to −196 °C. After warming to room temperature the products in the trap were dissolved in a small amount of benzene, and this solution used for VPC analysis. The products were identified as follows: Dimethyl sulfide (**4**) (21.0 mg, 0.339 mmol[11]) identified by IR comparison with a known sample. Dimethyl disulfide (**5**) (27.1 mg, 0.288 mmol): IR (CCl_4) 1420, 1300, 955 cm^{-1} (identical with the IR of an authentic sample[12]); NMR δ 2.38(s); mass spectrum *m/e* 94 (M^+, $C_2H_6S_2$), 79. Bis(thiomethoxy)methane (**6**) (62.4 mg, 0.578 mmol): IR (CCl_4) 1430, 1200, 985 cm^{-1}; NMR δ 2.15 (s, 6 H, CH_3S), 3.58 (s, 2 H, SCH_2S); mass spectrum *m/e* 108 (M^+, $C_3H_8S_2$), 93, 61 ($M - CH_3S$). Toluene (**8**) (46.0 mg, 0.500 mmol) was identified by comparison with a known sample. Methyl *p*-tolyl disulfide (**9**) (ca. 25 mg, 0.15 mmol): NMR δ 2.30 (s, 3 H), 2.38 (s, 3 H), 7.2 (A B quartet, 4 H, *p*-tolyl); mass spectrum *m/e* 170 (M^+, $C_8H_{10}S_2$), 155, 123 ($M - SCH_3$), 91 ($C_7H_7^+$). Di-*p*-tolyl disulfide (**10**) (65 mg, 0.264 mmol): IR ($CHCl_3$) 1480, 1030 cm^{-1}; NMR δ 2.24 (s, 6 H, CH_3Ar), 7.15 (A B quartet, 8 H, *p*-tolyl); mass spectrum *m/e* 246 (M^+, $C_{14}H_{14}S_2$), 123 ($M - C_7H_7S$), 91 ($C_7H_7^+$). The inlet tube to the pyrolysis trap was washed with methanol and yielded, after evaporation, 150 mg (0.877 mmol) of a white solid, identified as *p*-toluenesulfonamide (**7**) by spectral comparison with an authentic sample.

***S,S*-Diphenyl-*N*-*p*-toluenesulfonylsulfilimine (11).** Using a procedure[13] somewhat modified from the one described above, a solution of 4.18 g (14.9 mmol) of chloramine-T in 50 mL of 50% ethanol–water was stirred at room temperature with 1.84 g (9.92 mmol) of diphenyl sulfide. After heating on a steam bath for 0.5 h the mixture was allowed to stand at room temperature overnight. Reheating the mixture on a steam bath, addition of water until cloudiness was noted, and cooling yielded the crude product as a white solid. After suction filtration and recrystallization from benzene–hexane, 2.68 g (7.54 mmol, 76%) of product was obtained as white needles, mp 110–111 °C (lit.[14] mp 108–110 °C).

Pyrolysis of 11. Using the apparatus described above for the pyrolysis of **3**, the pyrolytic decomposition of **11** (0.770 g, 2.16 mmol) was

carried out at 300 °C for 2 h. The reaction products were identified by spectral and VPC comparison with authentic samples as diphenyl sulfide (0.361 g, 1.94 mmol), *p*-toluenesulfonamide (0.117 g, 0.684 mmol), and toluene (3.2 mg, 0.035 mmol).[11] A nonvolatile black residue (0.218 g) was also recovered from the pyrolysis tube.

An attempt to pyrolyze **11** at 250 °C resulted in only a trace amount of decomposition and the recovery of 98% starting sulfilimine.

Pyrolysis of *p*-Toluenesulfonamide (7). Under conditions identical to those described above, **7** (0.605 g, 3.54 mmol) was pyrolyzed at 300 °C for 1 h. VPC analysis showed toluene (22.8 mg, 0.248 mmol, 7%) as the only product present. Considerable black char was noted in the pyrolysis boat.

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Registry No.—**3,** 13150-75-9; **11,** 13150-76-0; chloramine-T, 127-65-1; diphenyl sulfide, 139-66-2.

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Migration of Acyl Groups in Acetyl–Alkoxycarbonyl Mixed Diacyl Derivatives of *o*-Aminophenol

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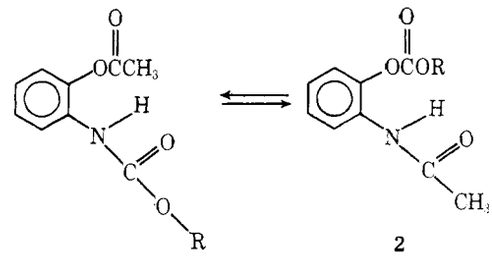
In an earlier article it was shown that the migration results obtained with several typical acyl groups were generally consistent with the hypothesis that the more stable isomer was that one with the poorer electron releasing group attached to nitrogen.[1] As discussed in some detail by Amundsen and Ambrosio, all reported work with acylalkoxycarbonyl mixed diacyls has resulted in isolation of only the isomer in which the alkoxycarbonyl group is attached to nitrogen.[2] Since these results are not consistent with the usually assumed order of the relative electron releasing powers of alkyl and alkoxy groups, it is felt desirable to investigate the synthesis and isomerization behavior of representative acetyl–alkoxycarbonyl systems. The present work presents results obtained with the acetyl–benzyloxycarbonyl and the acetyl–*n*-butoxycarbonyl systems **1–2.** System **1a** and **2a** was studied earlier



1 2

a, R = $PhCH_2$
b, R = *n*-Bu

by Amundsen and Ambrosio who were unable to prepare **2a** and found only the urethane on saponification of **1a.** System **1b** and **2b** had not been studied prior to this work and it was chosen since it represents a more clear-cut comparison of the relative electron-donating powers of an alkyl and an alkoxy functional group.

The diacyl derivatives were prepared by *O*-acylation of the *N*-alkoxycarbonyl and *N*-acyl compounds. Isomerization of purified samples of **1a** and **2a** in absolute alcohol was complete in 2 h at 26 °C and resulted in the formation of an equilibrium mixture containing 96.5% of 1a. Isomerization of **1b** and **2b** was very much slower than for **1a** and **2a** but after 380 h an equilibrium mixture was obtained containing 94.5% **1b.**

In pyridine solution, isomerization was much slower for both pairs of isomers. However, the same equilibrium composition was reached for **1a** and **2a** in about 24 h standing in pyridine at 25 °C. With **1b** and **2b, 1b** contained only 2% isomerized product after 382 h of standing time, while **2b** contained 54% of the isomerized product.

Saponification of either **1a** or **2a** gave a mixture containing 32, 50, and 18% of benzyl *o*-hydroxycarbanilate, benzoxazolone, and *o*-hydroxyacetanilide, respectively. Converting the benzoxazolone weight to benzyl *o*-hydroxycarbanilate from which it was derived[3] showed that saponification must have initially produced 83% benzyl *o*-hydroxycarbanilate. Since both isomers gave the same composition of saponification products, it seems clear that isomerization in the alkaline solution was rapid relative to saponification. It also seems clear that **2a** saponified faster than **1a** since it may be calculated that equal rates of saponification of the equilibrium mixture would yield a mixture of monoacyls containing 97.8% of benzyl *o*-hydroxycarbanilate.

Saponification of **1b** and **2b** produced only 4% of benzoxazolone in contrast to the 50% obtained with **1a** and **2a.** Correcting for this by-product as before, isomer **1b** yielded 93% *n*-butyl o-hydroxycarbanilate while isomer **2b** gave only 84% of this monoacyl. Thus, while isomerization in this system is much faster than saponification, there appears to be less difference in these rates than was the case for the **1a–2a** system. Had the saponification rates of **1b** and **2b** been equal and equilibrium attained instantly, it may be calculated that the saponification mixture would have contained 96.9% *n*-butyl *o*-hydroxycarbanilate. The most likely explanation of these results is that **2b** saponifies more rapidly than **1b** and that some saponification of **2b** occurs before it has had time to completely isomerize to the equilibrium mixture.

Experimental Section

Melting points are uncorrected and were taken on a Fisher digital melting point analyzer. Infrared spectra were recorded from potassium bromide disks on a Perkin-Elmer Model 21 spectrophotometer. Ultraviolet spectra were recorded using a Bausch and Lomb Model 600 UV–visible spectrophotometer.

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