N-*p*-Toluenesulfonyl-2-oxazolidones *Via* the Cycloaddition Reaction of *p*-Toluenesulfonyl Isocyanate and Epoxides.

J. E. Herweh and W. J. Kauffman

Armstrong Cork Company, Research and Development Center, Lancaster, Pennsylvania 17604

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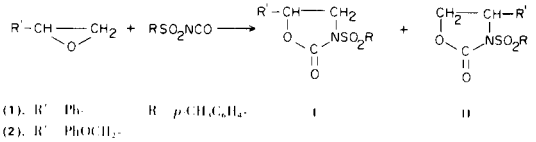
The reaction of styrene oxide and phenyl glycidyl ether with *p*-toluenesulfonyl isocyanate, employing a hydrocarbon-soluble adduct of tributylphosphine oxide and lithium bromide as catalyst, results in excellent yields of the *N*-*p*-toluenesulfonyl-2-oxazolidones. The 5-isomeric-2-oxazolidone is obtained from phenyl glycidyl ether, but in contrast to conventional isocyanates, the *p*-toluenesulfonyl isocyanate, upon reaction with styrene oxide, produces the 4-isomeric 2-oxazolidone as the major product. The effect of the *N*-sulfonyl group on the nmr spectra of 2-oxazolidones is discussed.

In connection with our interest in isocyanate chemistry and with particular regard to cycloaddition reactions of isocyanates, we have undertaken a brief investigation of the reaction of *p*-toluenesulfonyl isocyanate with epoxides. The catalyzed cycloaddition of aryl and alkyl isocyanates to terminal epoxides of the glycidyl ether and substituted ethylene oxide types to give 2-oxazolidones has been reported (1). In polar aprotic solvents such as DMF with lithium chloride catalyst, the cycloaddition reaction provides good yields of the desired 2-oxazolidones (2).

Most recently it was found that through the use of a hydrocarbon-soluble adduct of tributylphosphine oxide or hexamethylphosphoryltriamide with lithium bromide, solvents such as benzene or toluene could be used as reaction media (3). This modification resulted in excellent yields of 2-oxazolidones at moderate reaction conditions of time and temperature. It also provided for more suitable reaction conditions to examine the cycloaddition of sulfonyl isocyanates to epoxides. The former are known to react with solvents such as DMF and DMSO under relatively mild conditions (4).

Using the hydrocarbon-soluble adduct of tributylphosphine oxide with lithium bromide as a catalyst, *p*-toluenesulfonyl isocyanate reacts rapidly with phenyl glycidyl ether in refluxing benzene to give *N*-*p*-toluenesulfonyl-5-phenoxymethylene-2-oxazolidone (**2-I**) in high yield. In contrast, without the hydrocarbon-soluble catalyst and under otherwise similar reaction conditions, no 2-oxazolidone (**2-I**) formation could be detected. In the absence of a solvent and using lithium chloride catalyst no 2-oxazolidone was isolated after 3.5 hours at 160 ± 2°.



(1). R' Ph- R *p*-$CH_3C_6H_4$- I II

(2). R' $PhOCH_2$-

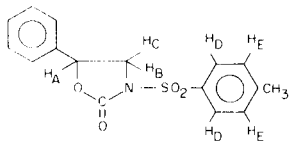
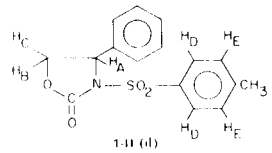
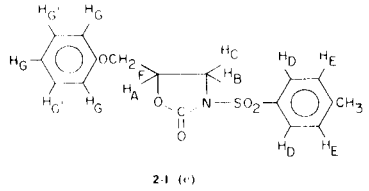
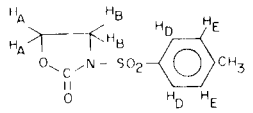
The identity of the reaction product as the 5-isomer (**2-I**) was demonstrated by an independent, unambiguous synthesis, namely, the reaction of *p*-toluenesulfonyl chloride with the sodium salt of 5-phenoxymethylene-2-oxazolidone (**3**). Nmr spectral assignments for **2-I** are given in Table I and will be discussed presently.

The use of styrene oxide as a coreactant with the sulfonyl isocyanate was also examined. Using conditions similar to those found to be successful for the preparation of **2-I**, the catalyzed cycloaddition proceeded in high yield to give a mixture consisting of approximately 80% 4-isomeric (**1-II**) and 20% 5-isomeric (**1-I**) 2-oxazolidones, as shown by nmr. In the absence of the phosphine oxide-lithium bromide adduct as catalyst or with tributylphosphine oxide present alone under similar conditions of time, temperature, and mode of addition, 2-oxazolidone formation was negligible.

Prior to determining the effectiveness of the hydrocarbon-soluble catalyst, styrene oxide and *p*-toluenesulfonyl isocyanate were reacted under a variety of conditions with variable results as briefly described below. A mixture of styrene oxide and the sulfonyl isocyanate (2:1 mole ratio), in the absence of solvent or catalyst, underwent an exothermic reaction (temperature rise from 25 to $>170°$) after a short induction period. The reaction products were not identified. In subsequent studies, the

TABLE I

Nmr Spectral Data (a)

Compounds (b,c)	δ	Solvent	H's (assign.)	Type	J (Hz)
 1-I (d)	3.84	$CDCl_3$ 15% (w/v)	H_C	dd	$J_{CA} = 8.00, J_{CB} = 9.50$
	4.39		H_B	dd	$J_{BA} = 8.00, J_{BC} = 9.50$
	5.48		H_A	t	$J_{AB}, J_{AC} = 8.00$
 1-II (d)	4.25	$CDCl_3$ 20% (w/v)	H_C	dd	$J_{CB} = 8.75, J_{CA} = 3.75$
	4.72		H_B	t	$J_{BA}, J_{BC} = 8.75$
	5.46		H_A	dd	$J_{AB} = 8.75, J_{AC} = 3.75$
 2-I (e)	3.9-4.1	$CDCl_3$ 10% (w/v)	H_C	masked by H_F	--
	4.07		H_F	d	$J_{FA} = 4.50$
	4.20		H_B	t	$J_{BA}, J_{BC} = 9.00$
	4.85		H_A	m	--
 5	3.98	DMSO-d_6 10% (w/v)	H_C	dd	$J_{CA} = 6.25, J_{CB} = 9.00$
	4.27		H_B	t	$J_{BA}, J_{BC} = 9.00$
	5.05		H_A	m	--
	4.05	DMSO-d_6 10% (w/v)	H_B	m	--
	4.36		H_A	m	--

(a) The assignment of H_C & H_B protons was based upon comparison of chemical shifts and nmr results of other 2-oxazolidones (2 and 7). (b) Methyl protons of the *p*-tolyl group absorb in the range of δ 2.38 to 2.44. (c) The aromatic protons (H_D & H_E) of the *p*-tolyl function absorb in the range of δ 7.85 to 7.95 (d, J = 8.25 Hz) and δ 7.27 to 7.45 (d, J = 8.25 Hz), respectively. In compound **1-II** these absorptions were shifted upfield to δ 7.45 and 7.13 (d, J = 8.52 Hz), respectively. (d) Phenyl protons appear as multiplets in the range of δ 7.30 to 7.35. (e) The o, m & p protons of the phenoxy group appear as multiplets at δ 7.23, 6.69 and 6.97, respectively.

sulfonyl isocyanate was added to a solution of the styrene oxide (1:1 mole ratio) in solvents, such as benzene, toluene, and/or acetonitrile. In all cases, the only identifiable product was the 4-isomeric-2-oxazolidone (**1-II**). The yield of **1-II** was consistently low, 16-23% in hydrocarbon solvents at room temperature or at reflux for variable periods of time, and 20-25% in refluxing acetonitrile with or without tetraethylammonium bromide catalyst.

The identity of the reaction product from the cycloaddition of the sulfonyl isocyanate to styrene oxide was established as a 2-oxazolidone by elemental analysis, molecular weight, and its ir spectrum. Elucidation of its

isomeric nature was obtained by inference, when the 5-isomer was prepared from *p*-toluenesulfonyl chloride with the sodium salt of 5-phenyl-2-oxazolidone (**4**). Nmr spectral assignments for **1-I** and **1-II** are summarized in Table I.

From a consideration of these results the use of the hydrocarbon-soluble catalyst is preferred, providing for high yields of the 2-oxazolidones. In using the hydrocarbon-soluble catalyst the epoxide and isocyanate are added concurrently to the catalyst solvent solution. This procedure was adopted in order to minimize any catalyzed epoxide rearrangement (5) and, also, in view of the earlier observations of an apparent uncatalyzed cycloaddition of the sulfonyl isocyanate to styrene oxide that resulted in low yields of 2-oxazolidone (**1-II**).

Of some interest is the apparent preference of the more highly polarized sulfonyl isocyanate for the lithium bromide catalyzed abnormal styrene oxide ring opened intermediate, as shown by isolation of the 4-isomeric-2-oxazolidone (**1-II**) as the major reaction product (ratio of 4 to 5 isomer was 4 to 1 by weight). The formation of **1-II** from uncatalyzed reactions presumably by direct addition of the dipolarophile, sulfonyl isocyanate, to the 1,3-dipolar epoxide may be contributing to the reverse isomer distribution observed. However, the unanticipated isomer distribution described above is in direct contrast to earlier results reported for the reaction of conventional isocyanates (*p*-tolyl and *n*-butyl) with styrene oxide catalyzed by lithium chloride in DMF. It may also be added that the phosphine oxide-lithium bromide catalyzed cycloaddition of *p*-tolyl isocyanate to styrene oxide in refluxing benzene gives the corresponding 5-isomeric-2-oxazolidone as the predominant product (3).

Recent work on the mechanism of epoxide rearrangement using the hydrocarbon-soluble lithium bromide adduct indicates the intermediacy of halohydrin salts (6). The isomeric distribution of the 2-oxazolidones isolated may be influenced by the half-life of the halohydrin intermediates. Consideration of the reverse reaction, *i.e.*, epoxide formation, leads to the postulation that the half-life of the intermediate leading to **1-II** could be shorter than that leading to **1-I**. If the reaction of conventional isocyanates with the halohydrin salt intermediates does not occur at a fast enough rate to trap the intermediate of shorter half-life, the 5-isomeric 2-oxazolidone is produced. The increased reactivity of the sulfonyl isocyanates allows the reactive species to be trapped and the isomeric distribution of the 2-oxazolidones becomes more dependent on the rate of formation of the reverse reaction to the epoxide. This could explain the difference in isomeric distribution of the 2-oxazolidones obtained from the same epoxide (styrene oxide). However, kinetic data is needed to substantiate the above proposal. Table I summarizes the nmr data obtained for the *N*-sulfonyl-2-oxazolidones. The isomeric pair **1-I** and **1-II** may be distinguished by nmr; the influence of the sulfonyl group is apparent when comparisons are made with nmr spectral results obtained for analogs prepared from *p*-tolyl isocyanate (2a).

The parent *N-p*-toluenesulfonyl-2-oxazolidone (**5**) was prepared in an attempt to assess the effect of the *N*-sulfonyl group on the 2-oxazolidone ring protons. As expected, enhancement of the electronegativity of the ring nitrogen by the sulfonyl group resulted in a downfield shift of the methylene protons adjacent to nitrogen. The shift is on the order of 0.45 ppm when compared to the H_B methylene protons of 2-oxazolidone itself. The complex multiplicity of both the H_A and H_B protons of the methylenes was not resolved (8).

In the 5-isomer (**1-I**) the vicinal *cis* and *trans* couplings are equivalent and smaller than the geminal coupling. This is in contrast to the *N-p*-tolyl-5-phenyl-2-oxazolidone spectrum in which the geminal and vicinal *cis* couplings are equivalent and greater than the vicinal *trans*. However, although coupling constants change, the chemical shifts are not significantly altered by introduction of the sulfonyl group. Increasing the electronegativity of the ring nitrogen by the sulfonyl group in (**1-I**) would be expected to increase the more sensitive geminal coupling, and this is observed. The difference in multiplicity in these two compounds may suggest that there is a difference in conformation about the carbon-carbon bond. Such a change in conformation may be due to increased interaction of the phenyl group and the sulfonyl substituent, since in compound (**2-I**) the multiplicity is that normally observed for 2-oxazolidones based on conventional isocyanates and epoxides: geminal and vicinal *cis* couplings are equivalent and greater than the vicinal *trans*. In compound (**2-I**), any interactions with the sulfonyl group can be relieved without affecting the ring carbon-carbon bond. This is not the case in compound (**1-I**).

In the case of the 4-isomer (**1-II**), the chemical shifts were essentially the same as for the *N-p*-tolyl-4-phenyl-2-oxazolidone and in both compounds the geminal and vicinal *cis* couplings are equal and greater than the vicinal *trans* coupling.

It can be seen that in going from the 5-isomer (**1-I**) to the 4-isomer (**1-II**) and in their *p*-tolyl analogs, the vicinal *trans* coupling is lowered. This lowering of the *trans* coupling is more markedly reduced in the case of the sulfonyl compound (**1-II**). Consideration of Dreiding Models indicates that this observed trend can be rationalized by a perturbation of bond angles at position 4, due to interaction of the phenyl group with the *N*-substituent. The nmr results indicate that this interaction is greater for the *N*-sulfonyl substituent, and is also manifested in an

upfield shift of the *p*-tolyl protons (see Table I, footnote c).

EXPERIMENTAL

General.

p-Toluenesulfonyl isocyanate (Upjohn Company, Carwin Research Laboratory), phenyl glycidyl ether, and styrene oxide were distilled prior to use. Solvents used as reaction media were dried by appropriate means.

Melting points are uncorrected. Elemental analyses were performed by Galbraith Laboratories, Inc., Knoxville 21, Tenn. Infrared absorption spectra were obtained on a Perkin-Elmer Model 337 spectrophotometer. The nmr spectra were determined on a Japan Electron Optics Lab 4H-100 spectrometer using TMS as an internal standard and solvents as indicated.

Reaction of Styrene Oxide with *p*-Toluenesulfonyl Isocyanate. A/In Acetonitrile.

A solution of *p*-toluenesulfonyl isocyanate (39.4 g., 0.2 mole) in 50 ml. of acetonitrile was added dropwise in 2 hours to a stirred, gently refluxing solution of styrene oxide (24 g., 0.2 mole) in 200 ml. of acetonitrile. Upon completing the addition, the clear pale-green reaction mixture was heated at reflux for 12 hours. The cooled, clear pale-amber reaction mixture was evaporated *in vacuo* (rotary evaporator, still temperature 45-55°) to yield a viscous residual oil (63.9 g.).

A solution of the viscous still residue (63.9 g.) in 200 ml. of benzene was extracted consecutively with four 25 ml. portions of cold, aqueous 10% sodium hydroxide, 2 times with 25 ml. of water, and finally with aqueous saturated sodium chloride. The first two basic washings caused an oil to precipitate with the evolution of some heat. In each case the oil was removed with the aqueous base. Distillation of the benzene solution *in vacuo* left 25.2 g. of a brown solid possessing a strong odor of styrene oxide. One recrystallization from carbon tetrachloride (300 ml.) gave 13.5 g. (0.043 mole, 21.3%) of crude **1-II**, m.p. 131-135°. Repeated recrystallization of crude **1-II** from carbon tetrachloride gave an analytically pure sample (white plates) of *N*-*p*-toluenesulfonyl-4-phenyl-2-oxazolidone (**1-II**), m.p. 145-146°; ir (potassium bromide) 1770 (s, C=O) cm^{-1}.

Anal. Calcd. for $C_{16}H_{15}NO_4S$ (**1-II**): C, 60.55; H, 4.76; N, 4.41; S, 10.10; molecular weight 317.4. Found: C, 60.78; H, 4.81; N, 4.57; S, 9.92; molecular weight 327.7 (cryoscopically in benzene).

The combined aqueous base washings and immiscible oil was acidified with hydrochloric acid. The aqueous acidic insoluble oil was extracted with ether. Distillation of the combined and dried ether washings left 33.3 g. of a viscous, amber oil that was not further identified.

The reaction was repeated in the presence of a catalytic amount (0.2 g.) of tetraethylammonium bromide using 0.1 mole of the sulfonyl isocyanate and styrene oxide, and proportionate amounts of solvent. A 24% yield of crude **1-II** was realized.

B/In Benzene Using Soluble Lithium Bromide Catalyst.

The sulfonyl isocyanate (19.7 g., 0.1 mole) and styrene oxide (12.0 g., 0.1 mole), each in 20 ml. of benzene, were added concurrently to a stirred refluxing solution of tributylphosphine oxide (0.76 g.) and lithium bromide (0.22 g.) in 150 ml. of benzene over a period of 2 hours. After completing the addition and heating at reflux for 0.5 hour, the reaction mixture no longer contained the sulfonyl isocyanate as determined by ir.

The cooled reaction mixture was filtered and gave 6.0 g. of a white solid. Distillation of the benzene filtrate *in vacuo* left a solid residue that was triturated with petroleum ether and filtered to give 25.1 g. of a white solid melting over a broad range (*ca.* 90-115°).

The crude product (31.1 g.) was found by nmr to consist of *ca.* 80% 4-isomeric 2-oxazolidone (**1-II**) and *ca.* 20% of the 5-isomeric-2-oxazolidone (**1-I**). One recrystallization of the mixture (31.1 g.) from boiling carbon tetrachloride gave 17.4 g. of solid, melting point 110-135°, whose isomer distribution, as determined by nmr, was essentially unchanged. No further attempt was made to resolve the mixture of isomers.

Reaction of Phenyl Glycidyl Ether with *p*-Toluenesulfonyl Isocyanate.

The reaction was carried out in benzene in the presence of the phosphine oxide-lithium bromide adduct as catalyst, using phenyl glycidyl ether (15 g., 0.1 mole) and *p*-toluenesulfonyl isocyanate (19.7 g., 0.1 mole). The procedure described above for styrene oxide and the preparation of the 4- and 5-isomeric 2-oxazolidones (**1-I** & **1-II**) was essentially repeated.

After completing the addition of reactants, heating at reflux was continued for 2 hours, at which point the reaction was judged to be complete, based upon the absence of the characteristic isocyanate absorption in the infrared. The reaction mixture was concentrated to dryness at reduced pressure. One recrystallization of the solid residue (31.9 g., m.p. 152-155°) from benzene gave **2-I**, a crystalline white solid (29 g.), m.p. 154-156°.

Analytically pure **2-I**, m.p. 156.5-157.5°, was obtained after one additional recrystallization from benzene; ir (potassium bromide) 1775 (s, C=O) cm^{-1}.

Anal. Calcd. for $C_{17}H_{17}NO_5S$ (**2-I**): C, 58.77; H, 4.93; N, 4.03; S, 9.23; molecular weight 347.4. Found: C, 58.76; H, 4.94; N, 4.01; S, 9.19; molecular weight 350 (determined in tetrahydrofuran by vapor-phase osmometry).

5-Phenyl-2-Oxazolidone (**4**).

Compound **4** was prepared (80% yield) according to the procedure of Poos and coworkers (9). The crude product (**4**), melting at 88-91° (reported m.p. 88.5-89.5°), was used without further purification; nmr (deuteriochloroform) δ 7.34 (s, 5H's, phenyl), 6.76 (s, broad, 1H, N-H), 5.75 (t, 1H, methine, $J_{cis} = J_{trans} = 8.75$ Hz), 3.95 (t, 1H, *cis* proton of ring methylene, $J_{cis} = J_{gem} = 8.75$ Hz), and 3.51 (t, 1H, *trans* proton of ring methylene, $J_{trans} = J_{gem} = 8.75$ Hz).

N-*p*-Toluenesulfonyl-5-Phenyl-2-Oxazolidone (**1-I**).

A solution of **4** (7.5 g., 0.047 mole) in ethylene glycol dimethyl ether (monoglyme, 100 ml.) was added dropwise in 40 minutes to a stirred slurry of sodium hydride (2.04 g. of a 57% dispersion in mineral oil) in 50 ml. of monoglyme at room temperature. Upon completing the addition, the pale greyish-white reaction mixture was stirred at ambient temperature for 2.25 hours and then at 50° for 0.5 hour. The off-white reaction mixture was cooled and *p*-toluenesulfonyl chloride (8.9 g., 0.047 mole) in 50 ml. of monoglyme was added in 50 minutes with stirring. After 2.5 hours at 50-55° followed by *ca.* 16 hours at room temperature, the turbid reaction mixture containing a finely divided solid was filtered through a Celite mat. The slightly turbid filtrate was concentrated under reduced pressure to dryness. Cold carbon tetrachloride was added to the white solid residue and the resulting slurry filtered. The filter-cake, washed with pentane, air-dried, gave 12.7 g. of crude **1-I**, m.p. 118-122°.

Several recrystallizations from boiling carbon tetrachloride (*ca.* 35 ml. of solvent per gram) afforded analytically pure **1-I**, m.p. 122.5-123.5°; ir (potassium bromide) 1775 (vs, C=O) cm^{-1}.

Anal. Calcd. for $C_{16}H_{15}NO_4S$ (**1-I**): C, 60.55; H, 4.76; N, 4.41; S, 10.10; molecular weight 317.4. Found: C, 60.73; H, 4.70; N, 4.37; S, 9.91; molecular weight 320 (determined in tetrahydrofuran by vapor-phase osmometry).

5-Phenoxymethylene-2-Oxazolidone (**3**).

The procedure of Oda and Hata was used with some modification (10), namely, a mixture of urea (120 g., 2.0 moles) and phenyl glycidyl ether (171.7 g., 1.28 moles) was heated to 150°. At *ca.* 150° a relatively violent exothermic reaction occurred and the temperature rose to 215° with frothing. The reaction mixture was left to cool to room temperature and then extracted with 1600 ml. of hot chloroform and filtered. Enough pentane was added to the hot chloroform to produce incipient turbidity. The resulting mixture was cooled to ice bath temperatures and the precipitate filtered.

One recrystallization of the dried filter-cake from ethyl acetate gave **3**, (15%), m.p. 121.5-123° (lit. m.p. 124°); ir (potassium bromide) 3320 (>N-H) and 1745 (vs, C=O) cm^{-1}; nmr (deuteriochloroform) δ 7.27 (m, 2 ortho H's of phenyl), 6.93 [m, 3H's (2 meta & 1 para) of phenyl], 6.62 (s, broad, 1H, > N-H), 4.93 (m, 1H, methine) 4.08 (d, 2H's oxymethylene, J = 5.00 Hz), 3.72 (t, 1H, *cis* proton of ring methylene, $J_{cis} = J_{gem} = 8.50$ Hz), and 3.54 (dd, 1H, *trans* proton of ring methylene, $J_{trans} = 6.50$, $J_{gem} = 8.50$ Hz).

N-p-Toluenesulfonyl-5-Phenoxymethylene-2-Oxazolidone (**2-I**).

The procedure described for *N-p*-toluenesulfonyl-5-oxazolidone (**1-I**) was repeated using 9.6 g. (0.05 mole) of 5-phenoxymethylene-2-oxazolidone (**3**), 2.18 g. (57% in mineral oil) of sodium hydride, and 9.5 g. (0.05 mole) of *p*-toluenesulfonyl chloride.

After completing the 2-stage reaction, an attempt to filter the reaction mixture containing a considerable amount of solid was unsuccessful; consequently, warm chloroform (*ca.* 400 ml.) was added to dissolve the organic portion of the precipitate. The resulting turbid solution was filtered through a Celite mat and the relatively clear filtrate concentrated under reduced pressure (water aspirator). The oily residue slurried with pentane and filtered afforded 11.5 g. of crude **2-I**, m.p. 153-155°; a mixture melting point with authentic **2-I** gave no depression. In addition, the two products were shown to be identical by ir and nmr.

N-p-Toluenesulfonyl-2-oxazolidone (**5**).

A solution of 2-oxazolidone (8.71 g., 0.1 mole) in 150 ml. of monoglyme was added fairly rapidly to a stirred suspension of sodium hydride (4.35 g. of a 57% dispersion in mineral oil) in 80 ml. of monoglyme at room temperature. After completing the addition the reaction mixture containing a grayish-white solid was heated at 50° for 2.5 hours and then cooled. A solution of *p*-toluenesulfonyl chloride (19.1 g., 0.1 mole) in 200 ml. of monoglyme was added dropwise with stirring to the reaction mixture and, upon completing the addition, the reaction mixture was heated at 50° for 3 hours.

After cooling, the reaction mixture was filtered and the white filter-cake washed consecutively with monoglyme and ether. The air-dried solid (18.7 g.) was slurried with water to remove sodium chloride; a crude residue of (18 g.) of **5**, m.p. 188-190°, was obtained. Concentration of the combined monoglyme and ether filtrates to *ca.* 150 ml., followed by cooling, afforded 3.2 g. of **5**, m.p. 185-186°. Finally the filtrate from the above concentrate was added to water and gave an additional 4.2 g. of **5**.

The combined (20.4 g.) crude fractions of **5** was recrystallized once from 350 ml. of chloroform to give 14 g. of **5**, m.p. 190-192°. Two additional recrystallizations from chloroform gave analytically pure *N-p*-toluenesulfonyl-2-oxazolidone (**5**), melting at 190.5-192.5°; ir (potassium bromide) 1765 (s, C=O) cm^{-1}.

Anal. Calcd. for $C_{10}H_{11}NO_4S$ (**5**): C, 49.78; H, 4.60; N, 5.81; S, 13.29. Found: C, 49.81; H, 4.58; N, 5.92; S, 13.09.

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