

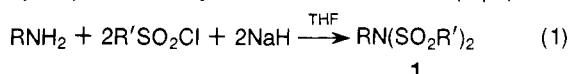
One-Step Synthesis of Primary Alkyl- and Aralkyl-*N,N*-disulfonimides

Richard A. Bartsch,* James R. Allaway, Roger B. Yandell, Jong Gun Lee, and Douglas W. McCann
Department of Chemistry, Texas Tech University, Lubbock, Texas 79409

An improved synthesis of primary alkyl- and aralkyl-*N,N*-disulfonimides is reported. Reaction of 2 equiv of sulfonyl chloride with 1 equiv of amine and 2 equiv of NaH in THF produces 40–85 % isolated yields of the title compounds.

Substitution and elimination reactions of compounds containing the sulfonimide leaving group, $-N(SO_2R)_2$, have recently aroused considerable attention (1, 2). Published two-step procedures for the preparation of alkyl- and aralkyl-*N,N*-disulfonimides (1) by Baumgarten and co-workers (3) and by Pan and Fletcher (5) require isolation of the intermediate sulfonamides. A one-step synthesis of benzyl- and *n*-hexyl-*N,N*-di(trifluoromethane)sulfonimides has been reported (4), but experimental details are almost completely lacking and extension of the method to less reactive sulfonating agents is uncertain.

We wish to describe an efficient, one-step general synthesis of primary alkyl- and aralkyl-*N,N*-disulfonimides, **1** (eq 1).



Two equivalents of the sulfonyl chloride in THF are slowly added to a cooled and vigorously stirred mixture of 1 equiv of the amine and 2 equiv of NaH in THF. Foaming of the reaction mixture during the addition and subsequent reflux is controlled by small amounts of the Union Carbide Corp. antifoaming agent SAG-100 (dimethylpolysiloxane). During workup, a major portion of the THF is recovered by distillation and may be used in succeeding synthesis.

Yield (mostly 55–86 %) and melting point data for nine primary alkyl and aralkyl-*N,N*-disulfonimides are presented in Table I. Assignment of structures **1a–i** is supported by C, H elemental, infrared spectral (3), and proton magnetic resonance spectral (3) analyses.

For a given primary alkyl- or aralkylamine, the yield of *N,N*-di(*p*-toluene)sulfonimide is always somewhat higher than that of the *N,N*-dimethanesulfonimide. Compounds **1b** and **1c** were previously reported by Baumgarten and co-workers using a two-step method (3). Yields of these compounds from the current one-step procedure compare favorably with those obtained from the two-step synthesis.

Experimental Section

Melting points were measured with a Fisher-Johns melting point apparatus and are uncorrected. Elemental analyses were performed by Chemalytics Inc., Tempe, Ariz. For all new compounds, carbon and hydrogen analyses agree with the theoretical values within ± 0.4 %.

General Synthetic Method. Into a 1000-mL three-necked flask equipped with a nitrogen atmosphere, reflux condenser, addition funnel, and magnetic stirrer is placed 10.0 g of NaH (52 % oil dispersion, 0.217 mol). Mineral oil is removed from the NaH by washing (3 $\times$ 25 mL) with pentane. To the dry NaH is added 200 mL of THF and 6.4 mL of a 0.05 % solution of SAG-100 in THF. The amine (0.10 mol) is added with stirring and the reaction flask is then cooled in an ice-salt bath. With maximum stirring, 0.20 mol of the sulfonyl chloride in 100 mL of THF is slowly added at such a rate that foaming is controlled. After addition of the sulfonyl chloride, the ice-salt bath is removed and the mixture is slowly heated to a gentle reflux. After 24 h, the apparatus is modified and most of the THF is distilled into a receiver (but not to dryness). The reaction flask is cooled in an ice-salt bath and residual NaH is destroyed by careful addition of wet CH_2Cl_2 . Approximately 300 mL of water is added, the mixture is extracted (3 $\times$ 75 mL) with CH_2Cl_2 or $CHCl_3$, and the combined extracts are in turn washed with 300 mL of water. The organic layer is dried over $MgSO_4$ and filtered, and the solvent is removed in vacuo. The crude residue is purified by recrystallization.

Literature Cited

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Table I. Yield and Melting Point Data for Primary Alkyl- and Aralkyl-*N,N*-disulfonimides^a

Compd	Registry no.	R	R'	Yield, ^b %	Mp, °C	Recryst solvent
1a		$CH_3(CH_2)_5$	CH_3	66	42–43	MeOH
1b	24332-41-0	$CH_3(CH_2)_5$	<i>p</i> - $CH_3C_6H_4$	81	118–118.5 ^c	MeOH
1c	52374-07-9	$CH_3(CH_2)_5$	<i>m</i> - $NO_2C_6H_4$	70	113.5–114 ^d	EtOH-acetone (4:1)
1d		$PhCH_2$	CH_3	40	117–117.5	MeOH
1e		$PhCH_2$	<i>p</i> - $CH_3C_6H_4$	57	162–163	Acetone
1f		$PhCH_2CH_3$	CH_3	68	70–71	MeOH
1g		$PhCH_2CH_2$	<i>p</i> - $CH_3C_6H_4$	78	103–104	MeOH
1h		Furfuryl	CH_3	67	122–122.5	MeOH
1i		Furfuryl	<i>p</i> - $CH_3C_6H_4$	86	139–140	MeOH

^a Elemental analyses (for these compounds) in agreement with theoretical values were obtained. ^b Yield of recrystallized product. ^c Reference 3 reports mp 114.9–115.2 °C and 97 % yield. ^d Reference 3 reports mp 113.0–114.5 °C and 47 % yield.