Synthesis of N-Alkylsulfonylphthalimides from Phthalimide and Sulfenes

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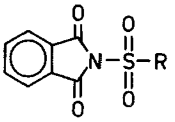
Two methods have been reported for the preparation of N-alkylsulfonylphthalimides:



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Reaction of potassium phthalimide with alkanesulfonyl chlorides in refluxing acetonitrile gives **1** (R = CH_3, C_2H_5, n-C_3H_7, n-C_4H_9)[1].

Reaction of phthaloyl chloride with unsubstituted alkanesulfonamides at elevated temperatures gives **1** (R = CH_3, C_2H_5, n-C_3H_7, n-C_4H_9, n-C_8H_{17}, 2-octyl)[1,2].

We now report an alternative method that affords excellent yields of the N-substituted phthalimides from readily available starting materials. This method involves the novel reaction of phthalimide with sulfenes[3], generated *in situ* from alkanesulfonyl chlorides and triethylamine, in cold acetonitrile:

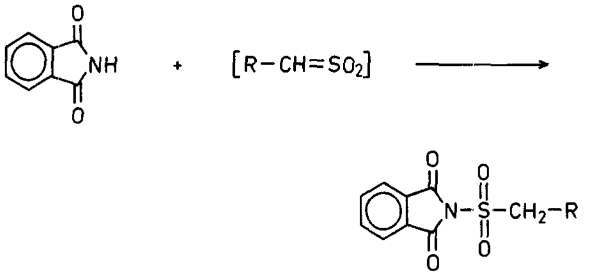

Table 1. N-Alkylsulfonylphthalimides Prepared from Phthalimides and Sulfenes.

R	Yield %	m.p.
H	86	227–229°
CH_3	95	182–183°
C_2H_5	89	138–139°
n-C_3H_7	93	113.5–115°

General procedure: To a stirred mixture of phthalimide (10 mmol) and triethylamine (15 or 20 mmol) in dry acetonitrile (30 ml) at 0° under nitrogen was added, dropwise during 0.5 hr, a solution containing an equivalence of alkanesulfonyl chloride equal to that of the tertiary amine in dry acetonitrile (15 ml). The resulting slurry was allowed to warm to room temperature and stirred at this temperature for 1 hr. The mixture was poured into 100 ml cold water, and the white solid that separated was removed by filtration, washed with water, and dried. The unrecrystallized products, which were reasonably pure and on which the yields are based, were characterized by mixture melting point determinations and I.R.-spectra comparisons with authentic samples. Phthalimide and ethanesulfonyl chloride do not interact in the absence of the tertiary amine under the reaction conditions used.

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