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1-Nitro-3-[(phenylsulfonyl)methyl]benzene

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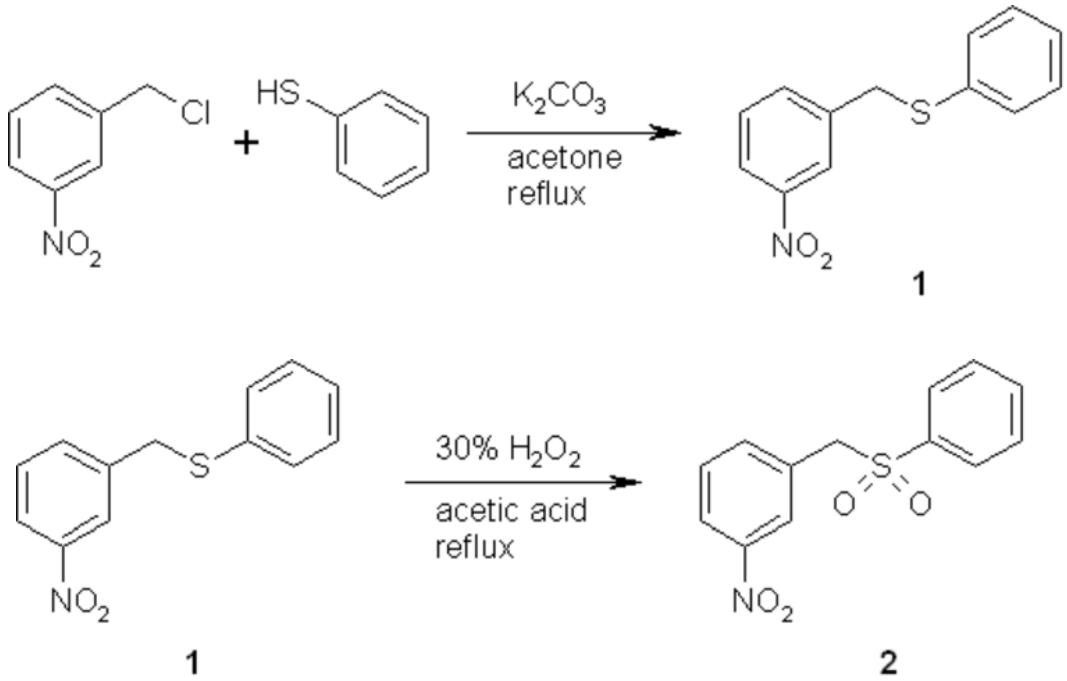
In the course of our work to prepare inhibitors of the enzyme dihydrofolate reductase, we desired to prepare sulfone analogues of some previously reported sulfides [1, 2]. Therefore, we prepared the nitrosulfide 1 [1], and oxidized it with hydrogen peroxide in acetic acid [3, 4] to prepare the corresponding nitrosulfone, 2.



A mixture of 3-nitrobenzyl chloride (6.868 g, 40.03 mmoles), thiophenol (4.453 g, 40.42 mmoles), and potassium carbonate (5.585 g, 40.41 mmoles) were placed in a round bottom flask with 35 mL of acetone, and this mixture was refluxed for 16 hours. The potassium chloride that formed in the reaction was removed by vacuum filtration. The acetone was removed from the filtrate by using the rotovap. The product was purified by adding 40 mL of 5 *M* NaOH to the product in the flask. The nitro compound was extracted using 2 portions of ether and 1 portion of dichloromethane. The organic extracts were combined and dried with anhydrous magnesium sulfate. The organic solvents were removed with the rotavap to yield 9.027 g (36.80 mmoles) 1-nitro-3-(phenylthio)methyl]benzene, 1, as a dark-yellow viscous liquid. The percent yield for this reaction was 92%. This product was used without further purification in the next reaction.

1-nitro-3-(phenylthio)methyl]benzene, **1** (3.830 g, 15.61 mmoles), 12 mL of 30% hydrogen peroxide, and 30 mL of concentrated acetic acid were combined in a round bottom flask and refluxed for 24 hours. Initially, the reaction mixture was yellow, but after refluxing, a white solid became evident. The solution

was allowed to cool, then the product was collected by suction filtration and allowed to dry. 3.266 g (11.78 mmoles) of 1-nitro-3-[(phenylsulfonyl)methyl]benzene, **2**, was collected. The percent yield of this reaction was 75%.

Melting Point: 163-164 ^{oC}

IR (cm^{-1}): 1520, 1351, 1319, 1297, 1280, 1112, 809, 730, 712, 695, 680, 671

^{1}H-NMR (300 MHz, DMSO-d_6): δ= 8.2 (1H, multiplet), 8.0 (1H, singlet), 7.7 (3H, multiplet), 7.6 (4H, multiplet), 4.9 (2H, singlet).

^{13}C-NMR (75 MHz, DMSO-d_6): δ= 147.3, 137.8, 137.6, 134.1, 131.0, 129.8, 129.2, 128.1, 125.5, 123.3, 59.4.

GC-MS [E.I., m/z (relative intensity)]: 136 (100), 90 (32), 89 (26), 177 (11), 277 (M^+, 10), 155 (10), 77 (10).

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