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

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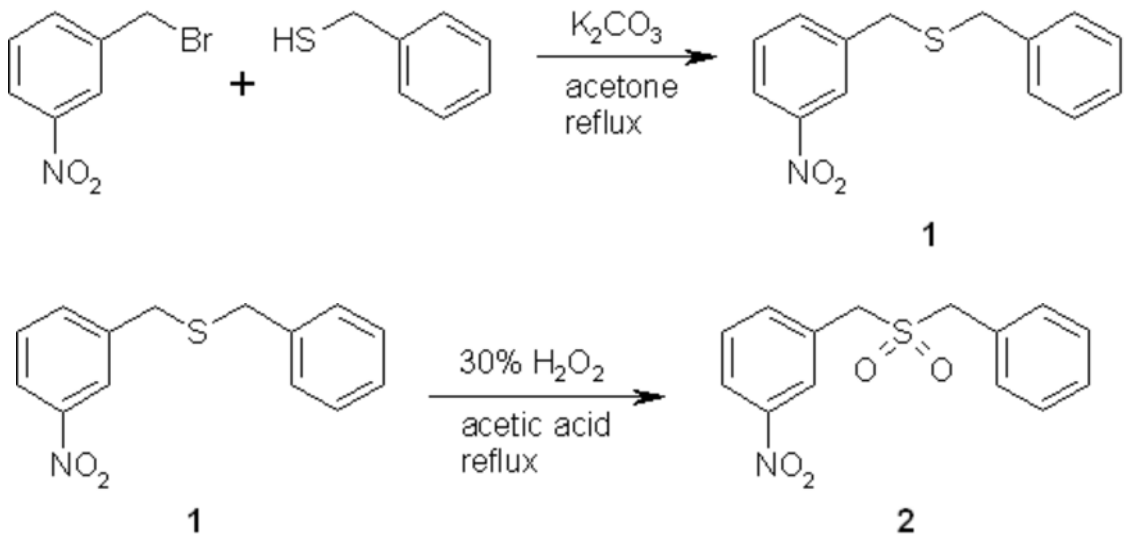
In the course of our work to prepare inhibitors of the enzyme dihydrofolate reductase, we desired to prepare sulfone analogues of some similar sulfides [1, 2]. Therefore, we prepared the nitrosulfide **1**, and oxidized it with hydrogen peroxide in acetic acid [3, 4] to prepare the corresponding nitrosulfone,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



A mixture of 3-nitrobenzyl bromide (6.482 g, 30.00 mmoles), benzyl mercaptan (3.744 g, 30.14 mmoles), and potassium carbonate (4.247 g, 30.73 mmoles) were added to a round bottom flask containing 60 mL acetone. The reaction mixture was refluxed overnight. The potassium bromide that formed in the reaction was removed by vacuum filtration. The acetone was removed from the filtrate by using the rotavap to yield the crude 1-nitro-3-(benzylthio)methyl]benzene, **1**, as a dark viscous liquid in quantitative yield. This product was used without further purification in the next reaction.

A mixture of 1-nitro-3-[(benzylthio)methyl]benzene, **1**, (5.192 g, 20.02 mmoles), 10 mL of 30% hydrogen peroxide, and 40 mL glacial acetic acid were combined in a round bottom flask and refluxed for 24 hours. The reaction mixture was allowed to cool. The solid which had formed was collected via vacuum filtration and allowed to dry, to yield 4.336 g (14.88 mmoles) of 1-nitro-3-[(benzylsulfonyl)methyl]benzene, **2**. The percent yield of this reaction was 74%.

Melting Point: 151 ^{o}C (decomposes)

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

^{1}H-NMR (300 MHz, DMSO-d_6): δ= 8.25 (1H, doublet, J = 8 Hz), 8.15 ppm (1H, singlet), 7.7 (1H,

doublet, J= 8 Hz), 7.55 (1H, triplet, J = 8 Hz), 7.4 (5H, multiplet), 4.3 (2H, singlet), 4.2 (2H, singlet).

^{13}C-NMR (75 MHz, DMSO-d_6): δ= 148.3, 137.1, 130.7, 129.9, 129.4, 129.3, 129.2, 127.2, 125.9, 124.0, 59.4, 56.7.

GC-MS [E.I., m/z (relative intensity)]: 91 (100), 89 (10), 77 (10), 291 (M^+, trace).

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