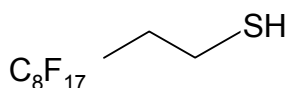


1H,1H,2H,2H-Perfluorodecane-1-thiol



Chemical Formula:	C ₁₀ H ₅ F ₁₇ S
Formula Weight:	480.18
Description:	Deprotecting Agent
CAS Number:	34143-74-3
Appearance:	Clear liquid
Properties:	Density: 1.64 Boiling Point: 82°C/12mm
Soluble in:	Dichloromethane, chloroform, THF, ether, toluene and most other typical organic solvents
Stability:	Solidifies near room temperature

DESCRIPTION AND USES:

- Fluorous Thiol can be used as a deprotecting agent of 2-nitrobenzenesulfonamides.^{2a, b}
- The monoalkylation of primary amines often requires a protecting group to avoid di-alkylation, followed by alkylation and subsequent deprotection. Many protecting groups have been used, but Fukuyama and co-workers introduced a 2-nitrobenzenesulfonamide, which has become very popular due to its ease of synthesis, enhanced nucleophilicity and mild deprotecting condition.¹
- The resulting fluorous by-product was removed by a quick fluorous solid phase extraction over FluoroFlash® silica gel to give the pure deprotected amine.^{3,4}

TYPICAL DEPROTECTION PROCEDURE:¹ The principle of the use of the fluorous thiol as a deprotection agent for 2-nitrobenzenesulfonamide is demonstrated in a 2-step synthesis in which several amines **1a-g** were protected with 2-nitrobenzenesulfonamide, and then deprotected using the following procedure:

To a solution of *N*-(2-nitrobenzenesulfonyl)-serine methyl ester (100 mg, 0.33 mmol) and K₂CO₃ (227 mg, 5 equiv) in MeCN (3 ml), 1H, 1H, 2H, 2H-perfluorodecane-1-thiol (289 μL, 2.5 equiv of 2b) was added, and the mixture was stirred at 50°C for 16 h. The resulting yellow solution was filtered, evaporated in vacuo, redissolved in MeOH:H₂O (4:1), loaded onto a 5 g FluoroFlash solid phase extraction (F-SPE) cartridge that had been pre-conditioned with MeOH:H₂O (4:1). The cartridge was then eluted with 15 mL of MeOH:H₂O (4:1). Evaporation of the solvent afforded serine methyl ester in 96% yield. Table 1 shows a series of deprotection from the protected amines.

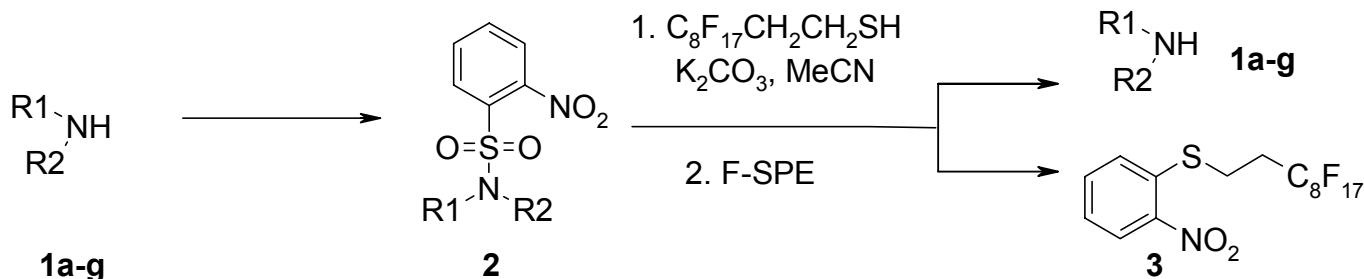
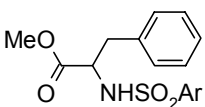
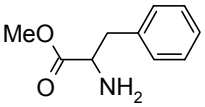
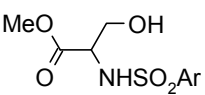
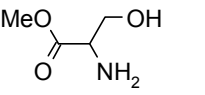
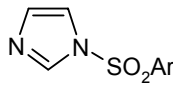
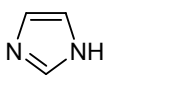
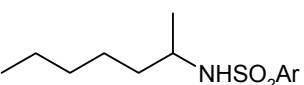
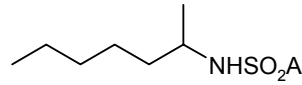
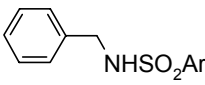
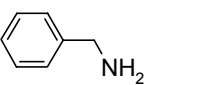
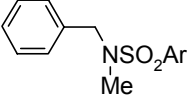
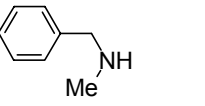
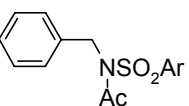
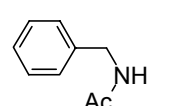


Table 1. Deprotection of 2-nitrobenzenesulfonamides 2a-g

Entry	Sulfonamide 2	Product 1	Yield ^a (%)
2a			91 (>95)
2b			96 (>95)
2c			76 (86)
2d			43 ^b (>95)
2e			72 (>95)
2f			77 (>95)
2g			81 (>95)

a Yields in brackets refer to isolated yields of the fluorinated byproduct

b The modest yield is due to the volatility of the product

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- Christensen, C.; Clausen, R. P.; Begtrup, M.; Kristensen, J. L. *Tetrahedron Letters* **2004**, 45, 7991.
- a. Fukuyama, T.; Cheung, M.; Jow, C. K.; Hidai, Y.; Kan, T. *Tetrahedron Letters* **1997**, 38, 5831. b. Review: Kan, T.; Fukuyama, T. *Chem. Commun.* **2004**, 353.
- Curran, D. P. *Synlett* **2001**, 1488.
- Please refer to FTI Application Note "Fluorous Solid Phase Extraction" for additional information

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