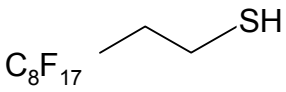


1H,1H,2H,2H-Perfluorodecane-1-thiol

Chemical Formula:	$C_{10}H_5F_{17}S$
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 Fluoro*Flash*® 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-nitrobenzenesulfnyl)-serine methyl ester (100 mg, 0.33 mmol) and K_2CO_3 (227 mg, 5 equiv) in MeCN (3 ml), 1*H*, 1*H*, 2*H*, 2*H*-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 Fluoro*Flash* solid phase extraction (F-SPE) cartridge that had been pre-conditioned with MeOH:H_2O (4:1). The cartridge was then eluted with 15 mL of MeOH:H_2O (4:1). Evaporation of the solvent afforded serine methyl ester in 96% yield. Table 1 shows a series of deprotection from the protected amines.

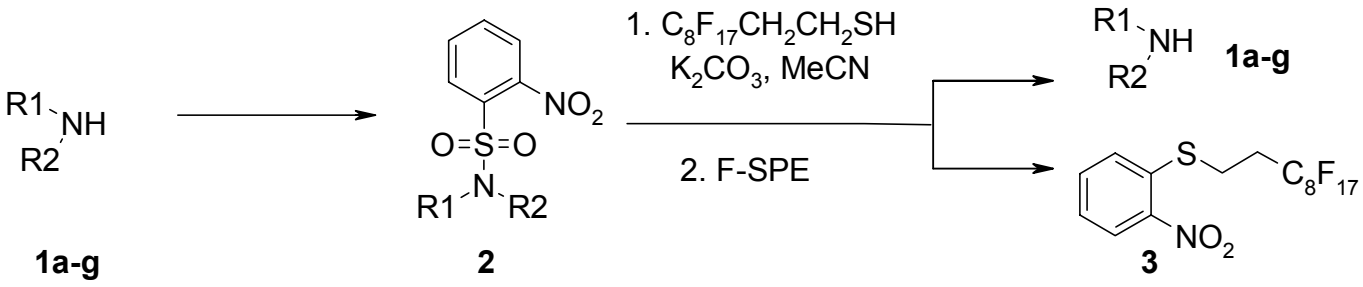
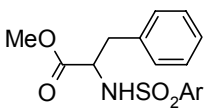
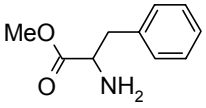
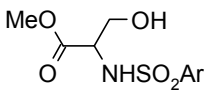
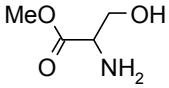
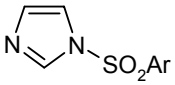
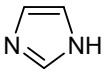
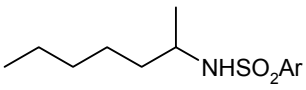
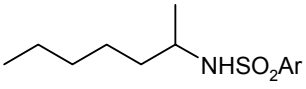
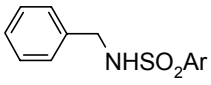
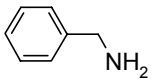
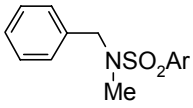
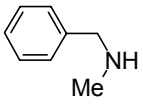
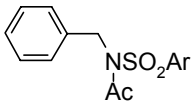
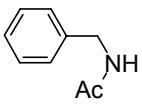


Table 1. Deprotection of 2-nitrobenzenesulfonamides **2a-g**

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

REFERENCES:

1) Christensen, C.; Clausen, R. P.; Begtrup, M.; Kristensen, J. L. *Tetrahedron Letters* **2004**, *45*, 7991.
2) 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.
3) Curran, D. P. *Synlett* **2001**,1488.
4) Please refer to FTI Application Note "Fluorous Solid Phase Extraction" for additional information

20041112