## SOP – Thiol-ene "Click" coupling of dendritic thiols



Polyester bis-MPA dendrons are available from Polymer Factory and Sigma Aldrich with thiol functionality at the core, with these dendrons having a unique ability to act as bifunctional linkers in biochemistry and materials science. These are readily functionalized through thiol-ene "click" chemistry (TEC) with a range of alkenes. The dendrons are available with hydroxyl and protected amine functionality at the periphery for further modification.

### Protocol: coupling of alkene to thiol-functional dendron

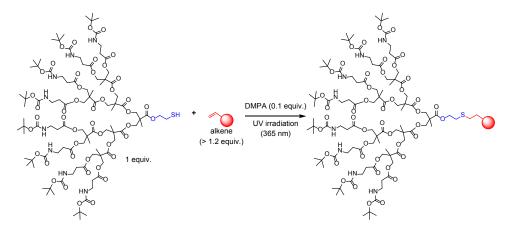


Figure 1: Example reaction scheme for functionalization of core thiol

- Dissolve the thiol functional dendron (1 equiv.), DMPA (0.2 equiv) and alkene (≥ 1.2 equiv.) at room temperature with stirring. A wide range of solvents are tolerated, including water, methanol, acetonitrile, DMSO, DMF and THF. The solvent should be chosen to dissolve well all reactants.
- Deoxygenate the reaction mixture via purging with inert gas (N<sub>2</sub> or argon) for 15 minutes.
- Irradiate the reaction mixture with 365 nm UV light for 1 hour.
- Follow the reaction with MADLI-TOF MS, monitoring the appearance of the mass corresponding to the product, or with 1H-NMR monitoring the shift of the peak corresponding to the  $CH_2$ -SH (ca. 2.80 ppm, 2H) protons shifts upfield (ca. 2.60 ppm,  $CH_2$ -SS-, 2H)
- When complete, evaporate the solvent and purify via flash column chromatography. Higher molecular weight products may be purified by dialysis.

### Reference

MV Walter, M Malkoch et al. "Novel Macrothiols for the Synthesis of a Structurally Comprehensive Dendritic Library using Thiol-Ene Click Chemistry" J. Polym. Sci. A Polym. Chem., 2011, 49(13), 2990-2995.

#### Disclaimer

The "click" coupling reactions are well established, and are robust and thoroughly investigated by the scientific community. However, these protocols are intended to serve as a guide for your own research, and are not guaranteed to work with all substrates.

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# Thiol dendrons available from Sigma-Aldrich

Product Name	Generation	Functional groups	
		Core	End group ( <i>n</i> )
Polyester bis-MPA dendron, 8 hydroxyl, 1 thiol (core)	3	Thiol	ОН (8)
Polyester bis-MPA dendron, 16 hydroxyl, 1 thiol (core)	4	Thiol	OH (16)
Polyester bis-MPA dendron, 32 hydroxyl, 1 thiol (core)	5	Thiol	OH (32)
Polyester bis-MPA dendron, 2 NHBoc, 1 thiol (core)	1	Thiol	NHBoc (2)
Polyester bis-MPA dendron, 4 NHBoc, 1 thiol (core)	2	Thiol	NHBoc (4)
Polyester bis-MPA dendron, 8 NHBoc, 1 thiol (core)	3	Thiol	NHBoc (8)
Polyester bis-MPA dendron, 16 NHBoc, 1 thiol (core)	4	Thiol	NHBoc (16)

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