

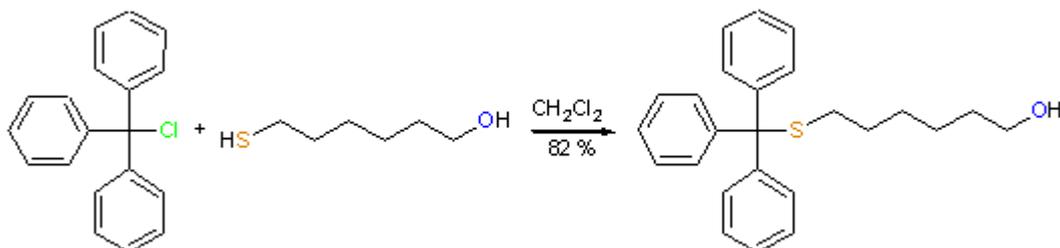
Molecules **2000**, *5*, M144

6-S-Trytyl-mercapto-hexan-1-ol

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Received: 16 February 2000 / Accepted: 20 February 2000 / Published: 28 April 2000



6-S-Trytyl-mercapto-hexan-1-ol is an important compound, because its *O*-b-cyanoethyl-*N,N*-diisopropyl phosphoramidite derivative is the most widely used building block for the synthesis of 5'-thiol modified oligonucleotides. The synthesis of this compound has been previously described from triphenylmethanethiol and 6-bromo-hexan-1-ol [1]. We have investigated the direct reaction of 6-mercapto-hexan-1-ol and trityl chloride. These reagents are cheaper and also readily commercially available. Moreover, the commonly used bases (e.g. pyridine and triethylamine) in the tritylation reaction give rise to the formation of large amounts of undesired side products which hampers the purification of the desired compound. On the other hand, if the reaction is carried out without any base [2] the desired compound is formed almost quantitatively; thus, the thiol group can be tritylated selectively. To a stirred solution of 6-mercapto-hexan-1-ol (2 ml, 15 mmol) in dichloromethane (20 ml), was added dropwise over 30 min a solution of trityl chloride (2.8 g, 10 mmol) in dichloromethane (50 ml) at room temperature. According to TLC (dichloromethane), all the trityl chloride was converted in 15 min. The reaction mixture was extracted with 0.1 M NaOH (in order to remove the unreacted mercaptohexanol from the mixture) then the organic layer was dried over MgSO₄, evaporated and the resulting yellow oil was crystallized from toluene/light petroleum ether to give 3.1 g (8.2 mmol, 82%) of the title compound.

Mp.: 73.5-74.5 °C.

¹H NMR (CDCl₃, 500 MHz): 1.26 (m, 5H, upon deuteration 4H, C(4)H₂-C(3)H₂, OH); 1.40 (m, 2H, C(5)H₂); 1.48 (m, 2H, C(2)H₂); 2.15 (t, J=7.3 Hz, 2H, C(6)H₂); 3.58 (t, J=6.6 Hz, 2H, C(1)H₂); 7.20 (m, 3H, aromatic CH); 7.27 (m, 6H, aromatic CH); 7.40(m, 6H, aromatic CH).

¹³C NMR (CDCl₃, 125 MHz, assignment based on *J*-modulated spin-echo, HMQC and HMBC experiments): 25.97 (C-3); 29.25 (C-5); 29.42 (C-4); 32.60 (C-6); 33.23 (C-2); 63.57 (C-1); 67.13 (Ph₃CS); 127.20; 128.49; 130.31 (aromatic C's); 145.77 (aromatic C_q).

Nano ESI-MS (LiCl additive in acetonitrile [3], *m/z*, %): 383 (100, [M+Li]⁺).

Anal. calcd. for C₂₅H₂₈OS (376.555): C, 79.74; H,7.49; S, 8.52; found C, 79.59; H, 7.35; S, 8.49 %.

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Sample availability: sample available from the authors.

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