JOC The Journal of Organic Chemistry

J. Org. Chem., 1997, 62(21), 7074-7075, DOI:10.1021/jo971511f

General Procedure for Conjugation of Protected Oligonucleotides. All solutions were prepared in the appropriate reaction solvents (DMF or acetonitrile: 1,1-dichloroethane (1:1)). A solution of 2,2'-pyridyl disulfide (50 μL, 0.11 M) and a solution of triphenyl phosphine (50 μL, 0.11 M) were added to a solution (100 μL) of carboxylic acid substrate (56 mM) and DMAP (56 mM). The solution was stirred at room temperature for 5 min, at which time 30 μL of it was added to the protected oligonucleotide (e.g. **5**, 83 nmol). The reaction was stirred at the appropriate temperature (25°C or 55°C) for 2 h, and then quenched with MeOH (1 μL). The reaction mixture was evaporated to dryness on a Savant Speed-Vac, and the residue treated with concentrated aqueous ammonia (1 mL) at 55°C overnight. Following evaporation as above, the residue was treated with 80% acetic acid (200 μL) for 20 min. The reaction was quenched with EtOH (600 μL) and evaporated to dryness. The residue was dissolved in dichloromethane (500 μL) and extracted with H_2O (3 x 250 μL). The aqueous layers were combined, evaporated to dryness, and purified by either anion exchange HPLC, or denaturing polyacrylamide gel electrophoresis.