Correction to "Chemoselective Synthesis of Sialic Acid 1,7-Lactones"

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J. Org. Chem. 2010, 75 (16), 5542-5548. DOI:10.1021/jo100732j

A new general protocol has been prepared to address issues relating to the required excess of benzyloxycarbonyl chloride (CbzCl) due to its low concentration in the commercial reagent used for the synthesis of 2-methyl-Nacetyl- β -neuraminic acid 1,7-lactone 8, 2-benzyloxycarbonyl-Nacetyl- β -neuraminic acid 1,7-lactone 10, 2-benzyloxycarbonyl-N-benzyloxyacetylneuraminic acid 1,7 lactone 14, 2-benzyloxycarbonyl-N-glycolyl- β -neuraminic acid 1,7-lactone 15, and 2benzyloxycarbonyl-3-deoxy-D-glycero-D-galacto-2-nononic acid 1,7-lactone 16. The general unified procedure uses a CbzCl ratio (2.5 molar equiv) reduced in respect to that reported (9.6 mol equiv).

GENERAL PROCEDURE FOR LACTONIZATION

The appropriate sialic acid (1.0 mmol), dissolved in DMF (3.5 mL) at 60 °C, under stirring, was cooled at 0 °C and diluted with THF (3.0 mL). Then CbzCl (356 μ L, 2.5 mmol) in THF (1.5 mL) was added in a single portion, followed by and Et₃N (417 μ L, 3 mmol, in a single addition). The mixture was then stirred for 30 min at 23 °C. After this, methanol (0.7 mL) was added, and the stirring was continued for 15 min. Evaporation of the solvent under reduced pressure (22 mmHg and then at 10^{-1} mmHg) afforded a crude residue, which after purification by flash chromatography (eluting with AcOEt/MeOH, 9:1, v/ v), afforded the appropriate pure Neu5Ac 1,7-lactone. 1,7-Lactone 8 was obtained in 78% yield (operating on 0.3 mmol scale); 1,7-lactone 10 was obtained in 81% yield (operating on a 3 mmol scale); 1,7-lactone 14 was obtained in 86% yield (operating on 0.1 mmol scale); 1,7-lactone 15 was obtained in 78% yield (operating on 0.3 mmol scale); and 1,7-lactone 16, was obtained, as a single compound, in 76% yield (operating on 3 mmol scale). All compounds showed physicochemical properties identical to those already reported in the paper.

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Published: March 10, 2015