

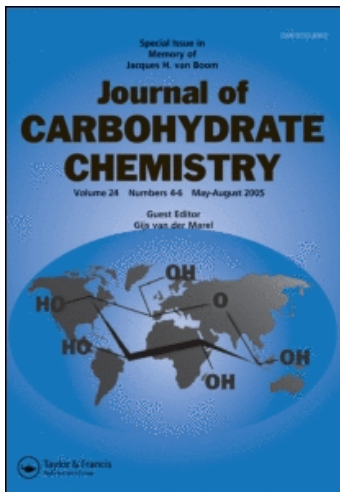
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### Convenient Preparation of 2,3,4, 6-Tetra-O-methyl- $\alpha$ -D-glocopyranosyl Bromide

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Communication

CONVENIENT PREPARATION OF

2,3,4,6-TETRA-Q-METHYL- $\alpha$ -D-GLUCOPYRANOSYL BROMIDE

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ABSTRACT

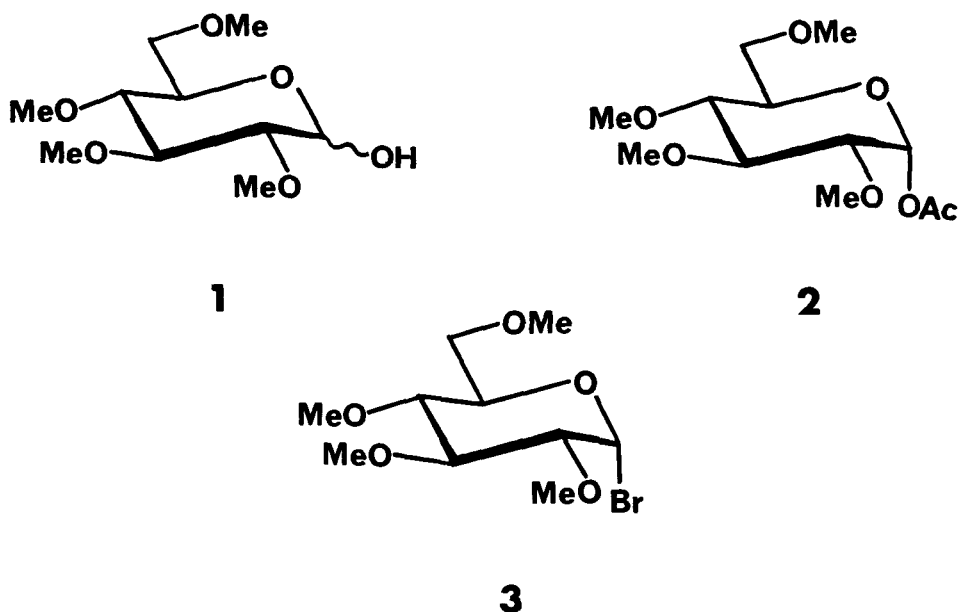
2,3,4,6-Tetra-Q-methyl- $\alpha$ -D-glucopyranosyl bromide (**3**) is conveniently prepared by treatment of 2,3,4,6-tetra-Q-methyl-D-glucose with HBr. This procedure is easier and produces higher yields than previously reported syntheses.

INTRODUCTION

As an exemplary glucopyranosyl halide with non-participating protecting groups, 2,3,4,6-tetra-Q-methyl- $\alpha$ -D-glucopyranosyl bromide (**3**) has frequently been utilized as a substrate for nucleophilic substitution reactions<sup>1-9</sup> and a starting material for glycal preparation.<sup>10</sup> Originally **3** of questionable purity was prepared by reaction of 2,3,4,6-tetra-Q-methyl-D-glucose (**1**) with phosphorus pentabromide.<sup>4</sup> In all subsequent syntheses, 2,3,4,6-tetra-Q-methyl-D-glucose (**1**) was converted into 1-Q-acetyl-2,3,4,6-tetra-Q-methyl-D-glucopyranose (**2**) which was treated with HBr under various conditions to afford **3**.<sup>5-9</sup>

RESULTS AND DISCUSSION

We now report that **1** reacts directly with HBr to afford a good yield of **3**. In our hands, this procedure is easier and produces higher yields than the previously reported syntheses of **3** which proceed via **2**. Only the  $\alpha$ -isomer was detected. In accord with previous reports, we observed that **3** decomposes quite rapidly, and therefore should be utilized directly after preparation.

EXPERIMENTAL

2,3,4,6-Tetra-O-methyl- $\alpha$ -D-glucopyranosyl bromide (3). 1,2-Dichloroethane (8 mL) was saturated with HBr<sup>11</sup> in a flask fitted with a drying tube and then mixed with 2,3,4,6-tetra-O-methyl-D-glucopyranose<sup>12</sup> (**1**, 213 mg, 0.90 mmol). After 12 min at 20 °C, the reaction was quenched with cold water, rinsed with saturated NaHCO<sub>3</sub> and water, and dried over CaCl<sub>2</sub>. Evaporation of the solvent afforded **3** as a syrup (217 mg, 81 % yield). Physical and spectral properties were in accord with literature.<sup>5</sup> <sup>1</sup>H NMR indicated only the  $\alpha$ -isomer. A sealed sample stored at -15 °C for three weeks became dark and showed extensive decomposition.

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