

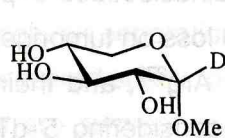
## Synthesis of deuterated methyl xylopyranosides

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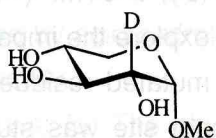
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Acid-catalyzed hydrolysis of methyl  $\beta$ -D-xylopyranoside is approximately twice as faster as that of methyl  $\alpha$ -D-xylopyranoside. The difference in the rates may be due to the fact that hydrolysis of methyl xylopyranosides proceeds through different transition states leading to different oxocarbenium ions. In order to probe the finer mechanistic details of the hydrolysis reaction, a multiple kinetic isotope effect study was carried out on the acid-catalyzed hydrolysis of methyl  $\beta$ -D-xylopyranoside and methyl  $\alpha$ -D-xylopyranoside.

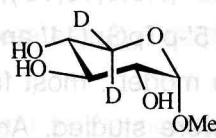
Presented here are the syntheses of the deuterated methyl xylopyranosides that have been used to measure kinetic isotope effects. Methyl  $\alpha$ - and  $\beta$ -D-(1-<sup>2</sup>H)xylopyranosides (1a and 1b), methyl  $\alpha$ - and  $\beta$ -D-(2-<sup>2</sup>H)xylopyranosides (2a and 2b), methyl  $\alpha$ - and  $\beta$ -D-(5-<sup>2</sup>H<sub>2</sub>)xylopyranosides (3a and 3b) were synthesized. Compounds were fully characterized using <sup>1</sup>H and <sup>13</sup>C NMR, IR and MS data.



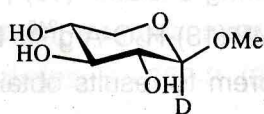
1a



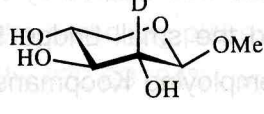
2a



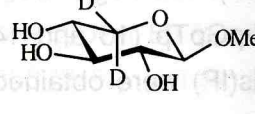
3a



1b



2b



3b

Reduction of 2,3,4,6-tetra-*O*-acetyl-D-glucono-1,5-lactone using NaBD<sub>4</sub> followed by acetylation gave 1,2,3,4,6-penta-*O*-acetyl-D-(1-<sup>2</sup>H) glucopyranose. This was hydrolyzed using NaOMe in methanol to give D-(1-<sup>2</sup>H)glucose. D-(1-<sup>2</sup>H)glucose was used as the starting material for the synthesis of (1a) and (1b). (2a) And (2b) were synthesized starting from D-(2-<sup>2</sup>H) xylose. The two deuteriums at C-5 position of xylose were introduced by the reduction of methyl 1,2-*O*-isopropylidene- $\alpha$ -D-xylofuranuronate with LiAlD<sub>4</sub>.

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