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Synthesis of ^{18}O-labelled methyl xylopyranosides

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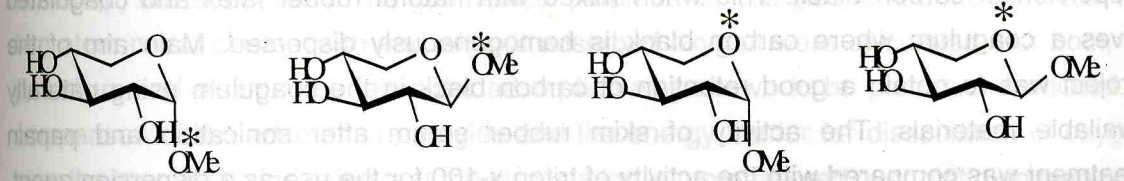
The synthesis of methyl α- and β-D-(1-^{18}O)xylopyranosides (1a), (1b) and methyl α- and β-D-(5-^{18}O)xylopyranosides (2a), (2b) is presented here. ^{18}O Kinetic isotope effects (KIE) were measured using 1a, 1b, 2a and 2b in the acid-catalyzed hydrolysis of methyl α- and β-D-xylopyranosides. Since methyl xylopyranosides are unsymmetrical acetals, in principle, three pathways are possible for the hydrolysis reaction; (a) endocyclic C-O bond cleavage, (b) exocyclic C-O bond cleavage and (c) O1-aglycon bond cleavage. The above mentioned ^{18}O KIE are very useful in determining the position of the C-O bond fission.



1a **1b** **2a** **2b**

[^{18}O label is marked with an asterisk (*) in each compound]

To synthesize 1a and 1b, 1,2,3,4-tetra-*O*-acetyl-β-D-xylopyranose was treated with methanol-^{18}O and $BF_3.Et_2O$ in dry CH_2Cl_2 at -78 °C under an atmosphere of dry N_2 gas. 1,2-*O*-isopropylidene-5-*O*-*p*-toluenesulfonyl-α-D-xylofuranose was treated with $CH_3C^{18}O_2Na$ in dry DMSO and heated at 85 °C for 24 hours to introduce ^{18}O at the C-5 position.

Compounds were fully characterized using ^1H and ^{13}C NMR, IR and MS data. The ^{18}O enrichments in 1a and 1b (96%) and 2a and 2b (55%) were estimated from the relative intensities of the M+1 ion peaks at 165 and 167 in the chemical ionization mass spectra.

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