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Publication details, including instructions for authors and subscription information: http://www.informaworld.com/smpp/title~content=t713618290

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To cite this Article Voss, Jürgen , Schulze, Oliver , Olbrich, Falk and Adiwidjaja, Gunadi(1997) 'Preparation and Reactions of 2-Oxa-6-thiabicyclo[3.2.0]-heptanes and 2-Oxa-5-thiabicyclo[2.2.1]heptanes from Pentoses', Phosphorus, Sulfur, and Silicon and the Related Elements, 120: 1, 389 - 390

To link to this Article: DOI: 10.1080/10426509708545562 URL: http://dx.doi.org/10.1080/10426509708545562

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# Preparation and Reactions of 2-Oxa-6-thiabicyclo[3.2.0]-heptanes and 2-Oxa-5-thiabicyclo[2.2.1]heptanes from Pentoses

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Abstract: The preparation of the two diastereoisomeric 3-methoxy-2-oxa-6-thiabicyclo-[3.2.0]heptan-4-ols 4 and 5 from D-xylose 1 via methyl 2,3-anhydro-α-D-ribofuranoside and the corresponding β-anomer is described. Oxidation of 4 and 5 yields the sulfoxides 6 and 7 and the sulfones 8. — On the other hand, the two diastereoisomeric 3-methoxybicyclo[2.2.1]heptan-7-ols 11 and 12 are obtained from methyl 5-acetylthio-5-deoxy-2-O-mesyl-D-xylofuranosides 9 and 10 via Mitsunobu reaction and intramolecular cyclization. — The stereoisomeric counterparts of 4 and 5, 13 and 14, are obtained in only four steps from L-arabinose.

Starting from D-xylose 1 the two oxathiabicyclo[3.2.0]heptan-4-ols 4 and 5 were prepared in a multistep synthesis via Mitsunobu reaction and intramolecular cyclization of the thioacetates 2 and 3. The two bicyclic compounds were obtained with good overall-yields as pure crystalline diastereoisomers. The structures of 4 and 5 were established by NMR spectroscopic methods and confirmed by X-ray structural analyses. The thietane ring of the  $\beta$ -isomer 5 turned out to be very flat (2.9°) whereas in the  $\alpha$ -isomer 4 the angle was 16.5°.

Oxidation of 4 and 5 with hydrogen peroxide yielded the corresponding sulfoxides 6 and 7 as well as the sulfones 8. The  $\beta$ -isomer of 8 exhibits a nearly planar thietane S,S-dioxide ring (2.0°) according to an X-ray structural analysis.

In a different way but using again the Mitsunobu reaction and cyclization of the thioacetates 9 and 10 we obtained the isomeric 2-oxa-5-thiabicyclo[2.2.1]heptan-7-ols 11 and 12 from the same precursor D-xylose 1.

Starting from inexpensive L-arabinose it is also possible to prepare in only four steps two further diastereomeric 2-oxa-6-thiabicyclo[3.2.0]heptan-4-ol derivatives, the mesylates 13 and 14. Once more, the Mitsunobu and the intramolecular cyclization reaction are applied.