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Note

Synthesis of alkyl 1-thio-β-D-galactopyranosides

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Among the different methods[1–4] for the synthesis of alkyl 1-thio-β-D-glycopyranosides, the method of Černý and Pacák[1,2] has several advantages. It avoids the use of thiols, which have unpleasant smells, and starts with the easily accessible *O*-acetyl-1-thio-β-D-glycopyranoses[1]. In a previous communication[5], we described the synthesis of alkyl 1-thio-β-D-xylopyranosides by this method. In this work, the synthesis of alkyl 1-thio-β-D-galactopyranosides by the same method is reported.

EXPERIMENTAL

A solution of 2,3,4,6-tetra-*O*-acetyl-1-thio-β-D-galactopyranose[6] (30 mmoles) and alkyl iodide (30 mmoles) in acetone (30 ml) was mixed with a solution of potassium carbonate (30 mmoles) in water (15 ml), and gently refluxed for 90 min. The organic layer was separated, dried (Na_2SO_4), and evaporated in *vacuo*. Because noneof the galactose acetates crystallized, and t.l.c. of the reaction mixture indicated partial deacetylation of the acetates, the crude syrup was deacetylated with sodium methoxide[7]. The alkyl 1-thio-β-D-galactosides were then crystallized from the appropriate solvent (Table I).

TABLE I

ALKYL 1-THIO-β-D-GALACTOPYRANOSIDES

Aglycon group	*Yield* (%)	*Crystallization solvent*	*M.p.* (*degrees*)	$[\alpha]_{5890}^{24}$ (*degrees*)	*Found* (%)		*Formula*	*Calc.* (%)	
					C	*H*		*C*	*H*
Pentyl	75	water–acetone	111–112	−39.5	49.5	8.4	$C_{11}H_{22}O_5S$	49.6	8.3
3-Pentyl	53	butanone	140–142	−40.2	49.4	8.5	$C_{11}H_{22}O_5S$	49.6	8.3
Heptyl	68	water	95–96	−36.0	52.9	8.7	$C_{13}H_{26}O_5S$	53.0	8.9
Octyl	63	methanol	106–107	−35.0	54.4	9.3	$C_{14}H_{28}O_5S$	54.5	9.2
3-Phenyl-propyl	51	water–ethanol	97–98	−38.4	57.3	7.1	$C_{15}H_{22}O_5S$	57.3	7.1

The melting points were determined with a Mettler FP2 instrument and are uncorrected. The optical rotations were measured on 0.5% solutions in methanol with a Perkin–Elmer Model 141 photoelectric polarimeter. T.l.c. tests were performed on Silica Gel G (Merck) with acetic acid–water–ethyl acetate (1:1:3). Detection was effected with 5% sulphuric acid in ethanol (10 min at 120°).

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