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Synthesis of S,O-Diesterified Dithiophosphate Ion and its X-Ray Crystal Structure as the N,N-Dipropylammonium Salt

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Synthesis of S,O-Diesterified Dithiophosphate Ion and its X-Ray Crystal Structure as the N,N-Dipropylammonium Salt

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Elimination of one methyl group by excess of N,N-dipropylamine transformed S-(3,4,6-tri-O-acetyl-2-deoxy- α -D-glucopyranosyl)-O,O'-dimethyl-dithiophosphate¹ into the N,N-dipropylammonium (R)S-(3,4,6-tri-O-acetyl-2-deoxy- α -D-glucopyranosyl)-O-methyl-dithiophosphate with chiral centre on phosphorus atom.

Crystal data: C₁₉H₃₆O₉NPS₂, space group P2₁, Z = 4
a = 16.721(9) Å
b = 9.023(4) Å β = 109.95(4)°
c = 19.797(8) Å

The crystal structure has been determined by direct methods and was refined anisotropically with fixed parameters of H atoms to R = 0.052 (R_w = 0.045) for 4072 MoK α independent reflections. The P atoms have almost identical tetrahedral arrangement. The average (av.) P-S bond lengths are 2.110(2) and 1.940(2) Å for esterified and non-esterified S atoms. These lengths indicate single-bond character in the first and some double-bond character in the second case. The av. P-O bond lengths are 1.588(2) and 1.490(6) Å for esterified and non-esterified O atoms. The av. S-P-S and O-P-O angles are 105.1(9) and 103.6(1)°. The av. non-esterified angle S-P-O is 119.3(3)°. The dithiophosphate groups have the ap,ap conformation with O-P-O-C and S-P-S-C torsion angles in the range 170.9(6) - 179.9(8)°. The av. C-S-P valency angle is 99.0(4)°.

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