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(4R)-1'-Acetyl-2,3-O-isopropyldiene-methylspiro [4,6-dIDEOxy-ß-D-ribo-hexopyranosid-4,5'-imidazolidin]-2',4'-dione

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Acetylation of hydantoins (imidazolidin-2,4-diones) and their 5-substituted derivatives normally occurs more readily in the 1-position [1]. A mixture of (4R)-2,3-O-isopropyldiene-methylspiro[4,6-dIDEOxy-ß-D-ribo-hexopyranosid-4,5'-imidazolidin]-2',4'-dione [2] (0.86 g, 3 mmol), acetic anhydride (2.5 ml) and dry pyridine (5 ml) was heated at 50 °C for 8 h followed by concentration and co-evaporation with toluene under diminished pressure to give the crude product. This was purified on a column of silica gel using CHCl₃/MeOH 4:1 as an eluent. The fractions with Rf 0.35 were collected and evaporated to afford the title compound (0.87 g, 89%) as a colourless oil which solidified on standing.

M.p. 98-99 °C.

[a]D 8° (c = 10 mg.cm⁻³, methanol).

TLC (CHCl₃/MeOH 4:1, silica gel) Rf 0.35.

¹H-NMR (CDCl₃): 5.26 (q, J=6.5 Hz, 1H, H-5); 4.54 (d, J=7.3 Hz, 1H, H-1); 4.37 (d, J=5.7 Hz, 1H, H-3); 4.27 (dd, J=7.3 and 5.7 Hz, 1H, H-2); 3.56 (s, 3H, OMe); 2.58 (s, 3H, COMe); 1.53 and 1.33 (2s, 2 x 3H, CMe₂); 1.27 (d, J=6.5 Hz, 3H, H-6).

¹³C-NMR (CDCl₃): 169.9, 169.5 and 153.5 (3 x C=O), 111.0 (CMe₂), 103.5 (C-1), 76.3 (C-2), 76.2 (C-3), 70.2 (C-4), 68.0 (C-5), 57.2 (OMe), 27.6 and 25.8 (CMe₂), 27.0 (COMe), 14.8 (C-6).

CI-MS (70 eV, pyridine): m/z 408 (100%, [M + C₅H₅NH]+).


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References
Sample availability: Available from authors.

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