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2-(2`,3`,5`-Tri-*O*-acetyl-b -*D*-ribofuranosyl)-4-(3-nitrophenylazo)-5-trifluoromethyl-2,4-dihydropyrazol-3-one

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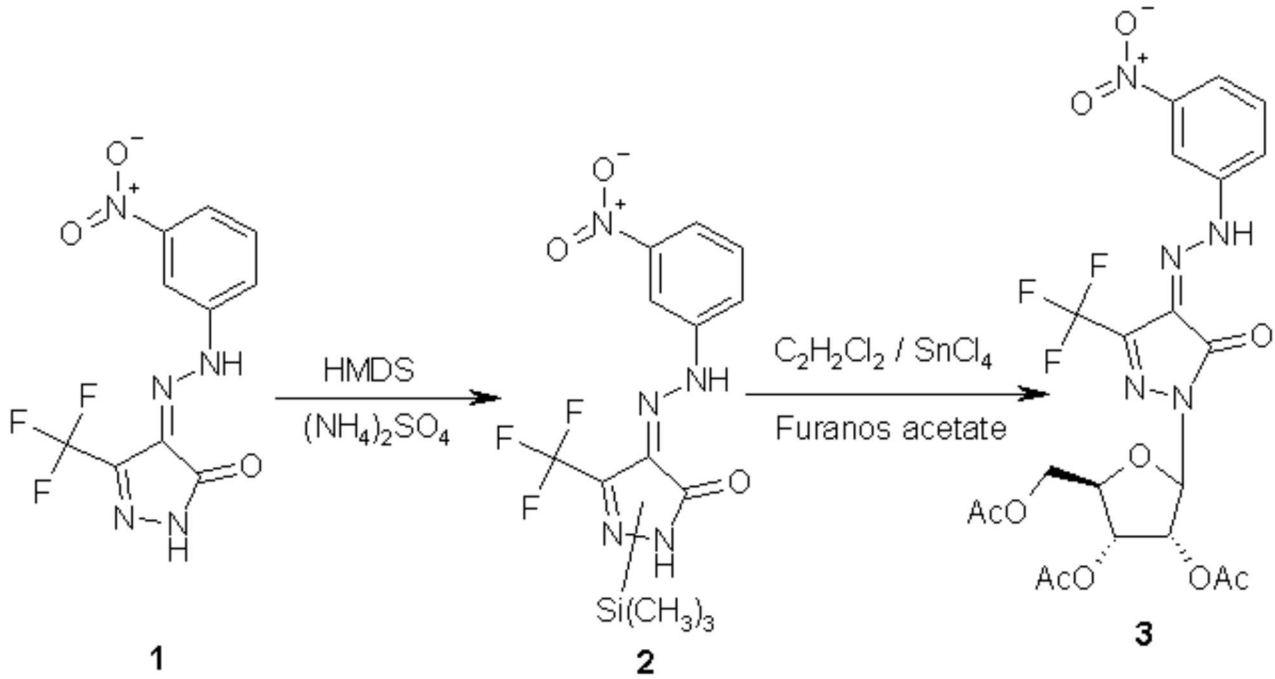

To a solution of 4-(3-nitrophenylazo)-5-trifluoromethyl-2,4-dihydropyrazol-3-one **1** [1] (0.903 g, 3 mmol) in hexamethyldisilazine (HMDS) (25 ml) was added few crystals of anhydrous ammonium sulfate [2]. The mixture was refluxed for three hours, then it was evaporated under vacuum to dryness. The residue was mixed with anhydrous xylene (30 ml) and the resulted solution was re-evaporated under vacuum to dryness to remove the remaining traces of HMDS. To a solution of the residue in anhydrous 1,2-dichloromethane (25 ml) was added 1,2,3,5-tetra-*O*-acetyl-b-*D*-ribofuranose (0.954g, 3 mmol). The mixture was treated with $SnCl_4$ (1.1 mmol) [2] and was then stirred at room temperature for two hours (tlc). The reaction mixture was diluted with dichloromethane (25 ml), washed with saturated aqueous solution of sodium bicarbonate (50 ml) and water (3x30 ml). The organic layer was dried over anhydrous sodium sulfate, filtered, evaporated to a small volume and chromatographed over silica gel column using ethyl acetate / n-hexane (4:6 v/v) to give 1.34g (80%) of **3** as yellow powder.

R_f (ethyl acetate/n-hexane, 50/50, v/v): 0.3.

UV (l_{max} , 95% ethanol): 266, 404.

MS (m/z): 559.

^{1}H-NMR (250 MHz, $CDCl_3$): 2.07(s, 9H, $COCH_3$); 4.07-4.13(dd, 1H, H-5``); 4.32-4.27(m, 1H, H-4`); 4.36-4.41(dd, 1H, H-5`, $J_{5`,4`}$=3.3, $J_{5`,5``}$=12.27); 5.44-5.48(t, 1H, H-3`, $J_{3`,4`}$=5.31); 5.62-5.65(dd, 1H, H-2`, $J_{2`,3`}$=5.67); 5.95(d, 1H, H-1`, $J_{1`,2`}$=3.84); 7.56-8.22(m, 4H, aromatic CH).

^{13}C-NMR (75 MHz, $CDCl_3$): 70.65(C-5`), 72.48(C-3`); 76.99(C-2`); 79.75(C-4`); 84.44(C-1`); 111.52,

117.81, 120.36, 122.85, 124.50, 131.11(6 aromatic carbons); 136.60(q, CF_3); 142.74(C-4); 148.56(C-5); 157.68(C-3); 169.40, 169.60, 170.63(3 CO).

References

1. Zohdi, H.F.; Rateb, N.M.; Haikal, A. *Molecules* **2001**, *6,* M261.
2. Vorbruggen, H.; Bennua, B. *Chem. Ber*. **1981**, *114*, 1279.

Sample Availability: Available from the authors and from MDPI.