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Synthesis of glycosyl triazoles and their identification by spectroscopic data and analysis

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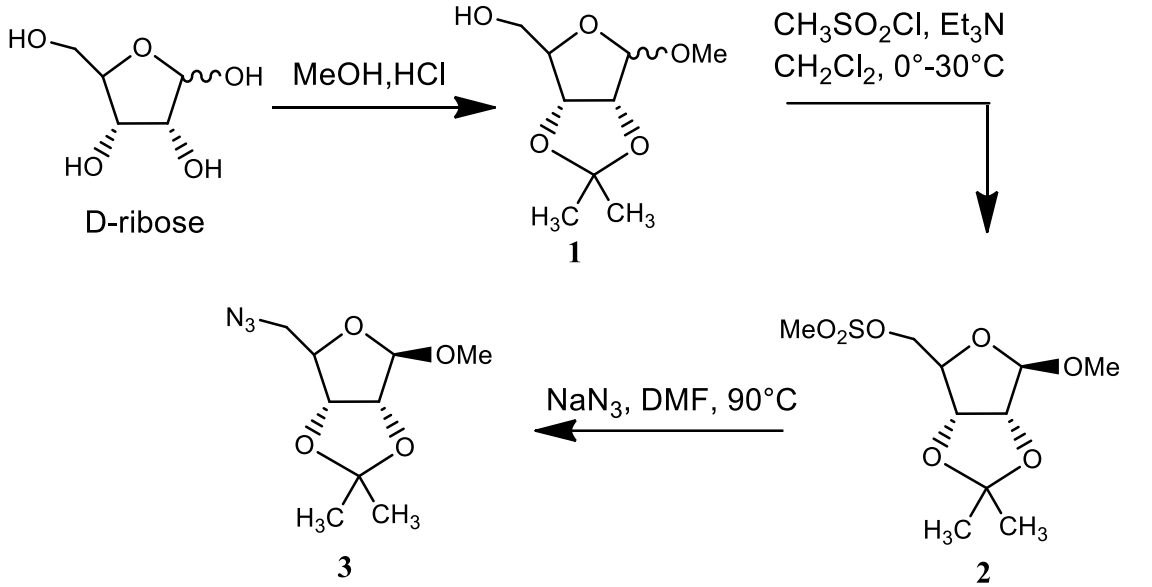
Abstract: *Triazoles specially 1,2,3-triazoles have wide range of applications and biological activities. Several reports show that triazoles have anti-inflammatory,[1,2] anti-platelets,[3] inhibition of histidine biosynthesis,[4] anti-HIV,[5] antibacterial,[6] antiallergic,[7] antimicrobial[8-10] and anticonvulsants [11] activities. Besides it has numerous application such as in bioconjugation, drug discovery,[12] material science,[13-16] local anaesthetic,[17] combinatorial chemistry,[18-21] antineoplastic,[22] dopamine D-2 receptor ligands.[23] Glycosyl and aglycosyl triazoles are important medicinal compounds. The azido sugars are the versatile starting materials in accessing numerous biologically active compounds which include amino sugars, nucleosides and many more glycosylated heterocycles.[24-26] Carbohydrate based triazoles are endowed with many biological activities.*

Key Words: *glycosyl and aglycosyl triazoles, azido sugars.*

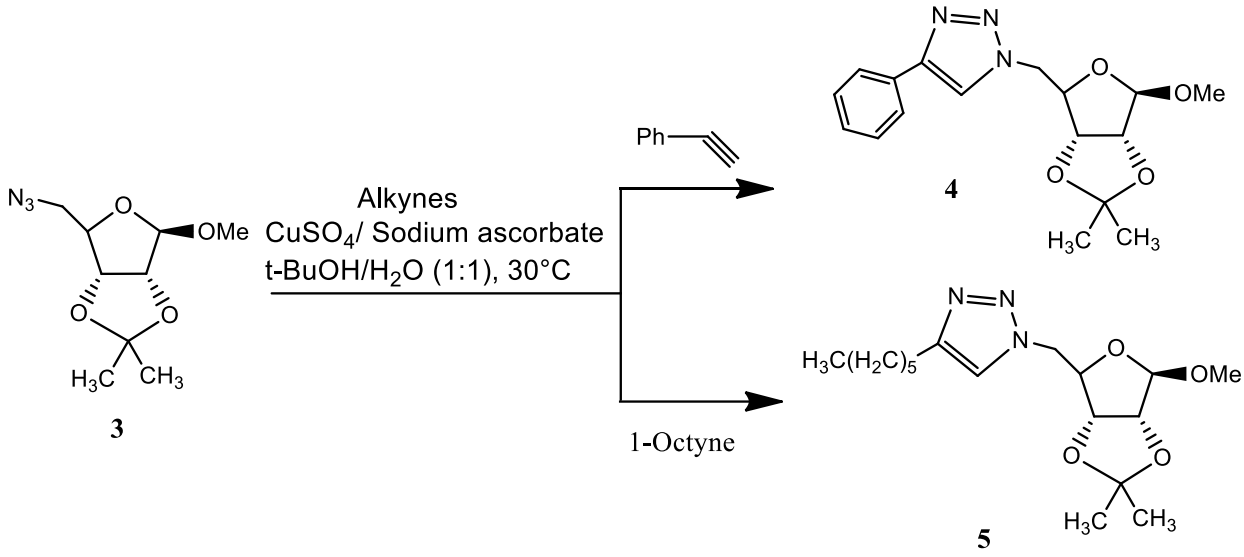
1. Synthesis of glycosyl triazoles:

The synthesis of glycosyl triazoles started from various type of sugar like D-ribose. D-ribose is treated with methanolic HCl to form 1-O-methyl-2,3-O-isopropylidene-β-D-ribofuranose **(1)**[27] Later on the protected ribose is treated with methanesulfonyl chloride to form 5-O-methanesulfonyl ribofuranose derivative **(2)**[28] Now the mesylated sugar on subsequent treatment with NaN_3 in DMF at 90°C led to the preparation of 5-azido-5-deoxy-2,3-isopropylidene-β-D-ribofuranoside **(3)** [28-29] in 89% yield. The structure of azido compound **(3)** was established with the help of spectroscopic data. IR spectrum of this compound show the characteristic azido peak at 2101 cm^{-1}. ESIMS spectrum showed a peak at m/z 230 $(M+H)^+$ which correspond to molecular weight of the compound. ^1H NMR spectrum show the two methyl of the isopropylidene group as singlet each at δ 1.29 and 1.46 respectively. Anomeric -OMe group appeared as singlet at δ 3.36. H-1 of the furanose ring appeared as singlet at δ 4.95, while H-2 and H-3 both as two proton singlet at δ 4.55 and H-4 as a triplet at δ 4.26 (J= 7.2 Hz). Where as each proton of H-5 were observed at two different field strengths as multiplets at δ 3.20 and 3.39. In ^{13}C NMR spectrum two methyl of isopropylidene group show peaks at δ 25.3 and 26.8 where as quaternary carbon of isopropylidene group appeared at δ 112.9. The OMe group appeared at 55.5 and C-1, C-2, C-3, C-4 and C-5 observed at δ 110.2, 82.4, 85.5, 85.7 and 54.3 respectively. 1, 3-Cycloaddition of the ribofuranosyl azide and alkynes was carried out at ambient temperature in presence of $CuSO_4$ and sodium ascorbate in a mixture of 1:1 $Bu^tOH:H_2O$ as reported by K. B. Sharpless and coworkers[30]. **(Scheme 1).** The cycloaddition of azido ribofuranose **(3)** with phenyl acetylene and 1-octyne in the presence of $CuSO_4$ (2 mol%) and sodium ascorbate (5 mol%) resulted in respective ribofuranosyl triazole 1-(methyl-5-deoxy-2,3-O-isopropylidene-β-D-ribofuranosid-5-yl)-4-phenyl-1H-1,2,3-triazole **(4)** in 70% yield and 4-n-hexyl-1-(methyl-5-deoxy-2,3-O-isopropylidene-β-D-ribofuranosid-5-yl)-1H-1,2,3-triazole **(5)** in 82% yield respectively.

The structure of 1-(methyl-5-deoxy-2,3-O-isopropylidene-β-D-ribofuranosid-5-yl)-4-phenyl-1H-1,2,3-triazole **(4)** was confirmed on the basis of spectroscopic data and analysis. ESIMS spectrum showed a peak at m/z 332 $(M+H)^+$ corresponding to its molecular weight. In ^1H NMR spectrum two of the methyl of isopropylidene group appeared as singlet each at δ 1.29 and 1.43 respectively, where as anomeric OMe group appeared as singlet at δ 3.38. H-1 of furanose ring observed as singlet at δ 4.48 while H-2 and H-3 appeared as doublet each at δ 4.73 and 4.65 having coupling constant J= 5.9 Hz each. H-4 and H-5 appeared as multiplet in the range of δ 4.42-4.61. The triazolyl proton observed as multiplet with two aromatic protons in the range of δ 7.78-7.83. The rest of the aromatic protons appeared as multiplet in the range of δ 7.28-7.42. In ^{13}C NMR spectrum two of the methyl of isopropylidene appeared at δ 25.2 and 26.6 while quaternary carbon of isopropylidene group at 112.8. C-1, C-2, C-3, C-4 and C-5 appeared at 110.2, 82.0, 85.1, 85.3 and 53.2 where as OMe group at C-1 appeared at δ 55.7.The characteristic C-4 and C-5 of the 1,4-regioisomers in the triazolyl ring appeared at δ 148.1 and δ 119.7 respectively. **(Scheme 2)**.

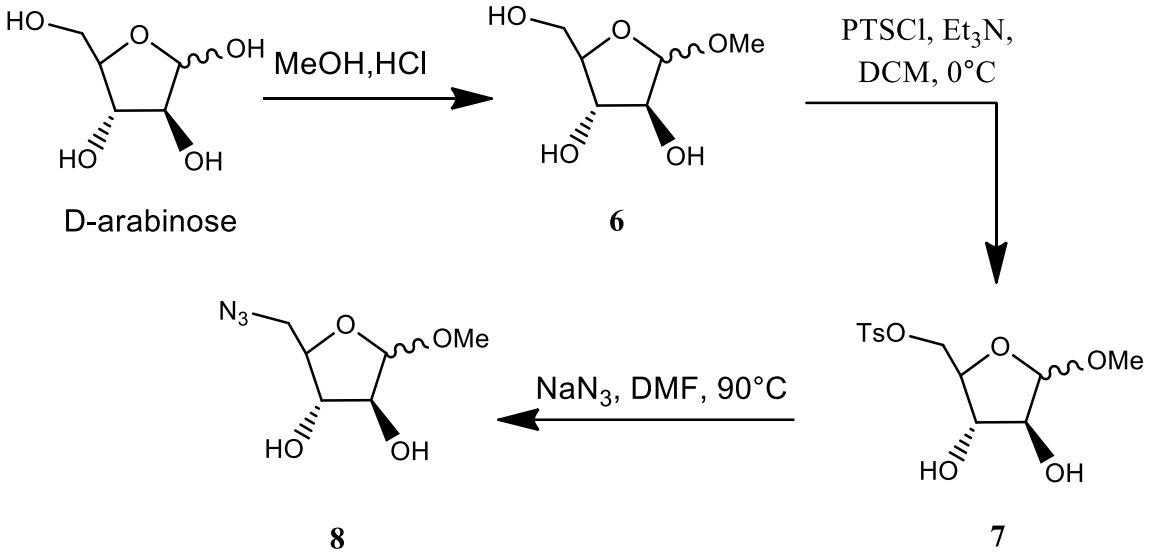


(SCHEME 1)



(SCHEME 2)

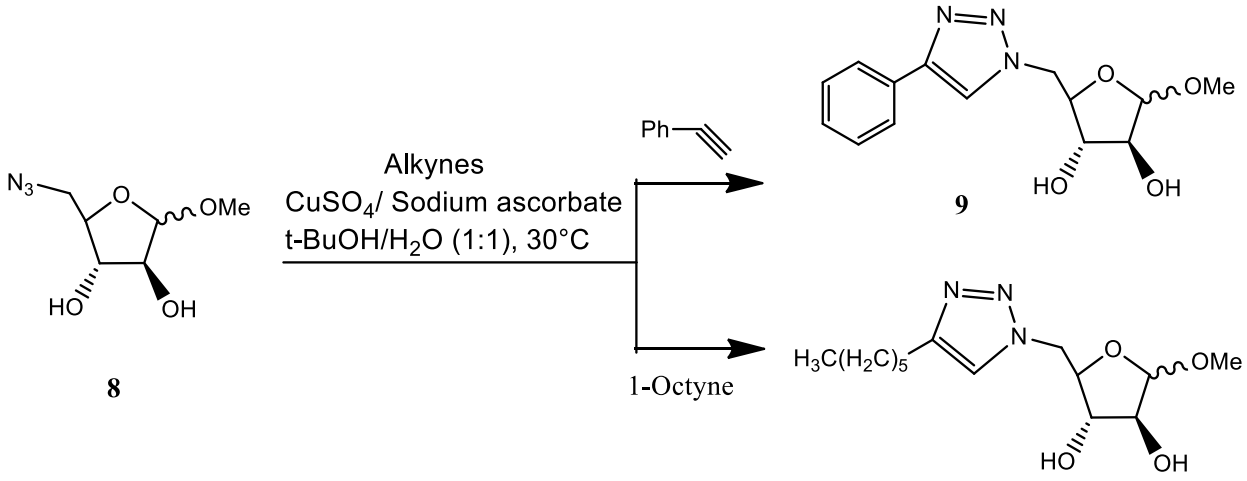
D-arabinose on treatment with methanolic HCl form methyl D-arabinofuranoside **(6)**[31] Later on reaction with p-toluenesulfonyl chloride at low temperature gave predominantly the methyl 5-O-(p-toluenesulfonyl)-D-arabinofuranoside **(7)**[32] Now this compound on treatment with sodium azide in DMF at 90°C gave the desired intermediate 5-azido-5-deoxy-D-arabinofuranoside **(8)** in good yield. The molecular structure of the compound was confirmed by spectroscopic data and analysis. IR spectrum of the compound showed the stretching frequencies at 3430, 2105 and 1101 cm^{-1} for the presence of OH and azido group. ESIMS spectrum showed showed a peak at m/z 190 $(M+H)^+$ corresponding to its molecular formula. In ^1H NMR spectrum H-1 and H-3 appeared as singlet at δ 4.85 and δ 3.90, where as H-2 and H-4 appeared as two proton multiplet in the range of δ 4.04-4.11. H-5 proton showed the multiplet in the range of δ 3.44-3.55 and the OCH_3 group placed at anomeric position as singlet at δ 3.40. ^{13}C NMR spectrum showed C-1, C-2, C-3 and C-4 at δ 109.1, 78.6, 81.6 and 83.1 respectively while C-5 and OCH_3 at C-1 observed at δ 52.4 and δ 55.5. **(Scheme 3)**



(SCHEME 3)

1,3-Cycloaddition of the azido arabinofuranose **(8)** with the phenyl acetylene and 1-octyne in the presence of $CuSO_4$ (2 mol%) and sodium ascorbate (5 mol%) in a mixture of 1:1 *t*-BuOH:H_2O led to the formation of 1-(methyl-5-deoxy-α-D-arabinofuranosid-5-yl)-4-phenyl-1H-1,2,3-triazole **(9)** in 60 % yield and 4-n-hexyl-1-(methyl-5-deoxy-α-D-arabinofuranosid-5-yl)-1H-1,2,3-triazole **(10)** in 65% yield respectively.

Identification of the synthesized triazole compound was established on the basis of spectroscopic data and analysis. IR spectrum of the compound showed stretching frequencies at 3396 and 1652 cm^{-1} for the characteristic of OH group and C=C. ESIMS spectrum observed a molecular peak at m/z 292 $(M+H)^+$ In ^1H NMR spectrum all the three proton of H-1, H-2 and H-3 appeared as singlet at δ 4.76, 4.11 and 3.86 whereas H-4 and H-5 form doublet and multiplet at δ 4.22 having coupling constant J= 4.1 Hz and δ 4.48-4.65 respectively. Methoxy group (OMe) showed a singlet at δ 3.22 and triazolyl proton appeared as singlet at δ 7.85 while OH proton showed broad singlet at δ 5.0. Aromatic protons of phenyl ring appeared at two different field strengths as multiplets at δ 7.16 and δ 7.65 respectively. In ^{13}C NMR spectrum C-1, C-2, C-3 and C-4 observed at δ 109.2, 78.3, 80.0 and 81.9 where as carbon of OMe and C-5 appeared at δ 55.3 and 52.0 respectively. 1,4-regioisomers of triazolyl ring have characteristic C-4 and C-5 appeared at δ 147.2 and δ 122.3. Aromatic carbons observed in the range of δ 126.1-130.5. **(Scheme 4)**

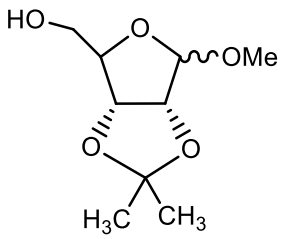


(SCHEME 4)

2. EXPERIMENTAL SECTION:

Chemistry: Commercially available reagent grade chemicals were used as received. All reactions were followed by TLC on E. Merck Kieselgel 60 F254, with detection by UV light and/or spraying a 5% H_2SO_4 in EtOH solution. Column chromatography was performed on silica gel (60–120 mesh E. Merck). IR spectra were recorded as thin films (KBr) or neat $CHCl_3$ solution with a Perkin Elmer Spectrum RX-1 (4000–450 cm^{-1}) spectrophotometer. ^1H and ^{13}C NMR spectra were recorded on a Brucker DRX-200 MHz and 50 MHz, respectively, in $CDCl_3$, and DMSO. Chemical shift values are reported in ppm relative to TMS (tetramethylsilane) as internal reference, unless otherwise stated; bs (broad singlet), s (singlet), d (doublet), t (triplet), m (multiplet); J in hertz. ESI mass spectra were performed using Quattro II (Micromass). Melting points were determined by open capillary method and uncorrected. Elemental analyses were performed on a Perkin–Elmer 2400 II elemental analyzer.

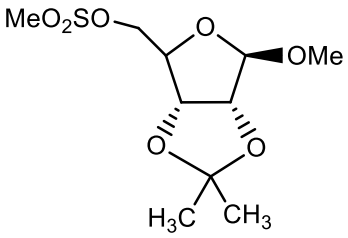
1. PROCEDURE FOR SYNTHESIS OF METHYL 2, 3-*O*-ISOPROPYLIDENE-β-D-RIBOFURANOSIDE (1):



1

To a magnetically stirred reaction D-ribose (8.24 g, 54.9 mmol) was dissolved in methanolic HCl and it was allowed at room temperature till the completion of reaction. When a clear solution was obtained, it was neutralized by triethylamine at 0°C. The reaction product was concentrated under reduced pressure and temperature. The latter was chromatographed over silica gel (60-120 mesh) using a gradient of hexane-EtOAc (8:2) as eluent to give methyl 2,3-*O*-isopropylidene-β-D-ribofuranoside (9.52 g, 85%).

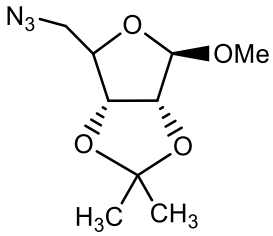
2. PROCEDURE FOR SYNTHESIS OF METHYL 5-*O*-METHANESULPHONYL-2, 3-*O*-ISOPROPYLIDENE-β-D-RIBOFURANOSIDE (2):



2

To a magnetically stirred solution of methyl 2,3-*O*-isopropylidene-ribofuranoside **(1)** (8.7 g, 42.6 mmol) in dry dichloromethane, containing triethylamine (6.07 ml, 60.1 mmol) at 0°C methane sulphonyl chloride (7.35 ml, 64.2 mmol) added dropwise. Stirring was continued at 0°C followed by at ambient temperature for 1 h. The reaction mixture was extracted with saturated sodium bicarbonate (2 x 50 ml) and water (2 x 50 ml) and the organic layer was dried (anhyd. Na_2SO_4), and evaporated under reduced pressure to give crude mass. The latter was chromatographed over silica gel (60-120 mesh) using a gradient of hexane- EtOAc (8:2) as eluent to give methyl 5-*O*-methanesulphonyl-2,3-*O*-isopropylidene-β-D-ribofuranoside **(2)** (10.8 g, 90%) as white solid; m.p.70°C; IR (KBr) cm^{-1}: 2940, 1450, 1184; ^1H NMR (200 MHz, $CDCl_3$): δ 1.29 (s, 3H, C*H*$_3$ of CMe_2), 1.43 (s, 3H, C*H*$_3$ of CMe_2), 3.11 (s, 3H, SO_2CH_3), 3.30 (s, 3H, OCH_3), 4.17 (d, 2H, *J* = 7.0 Hz, H-5), 4.36 (t, 1H, *J* = 7.0 Hz, H-4), 4.54 (d, 1H, *J* = 5.9 Hz, H-3), 4.63 (d, 1H, *J* = 5.9 Hz, H-2), 4.95 (s, 1H, H-5); ^{13}C NMR (50 MHz, $CDCl_3$): δ 25.1 (CH_3 of isopropylidene), 26.6 (CH_3 of isopropylidene), 38.2 (SO_2CH_3), 55.4 (OCH_3), 68.9 (C-5), 81.7 (C-2), 84.3 (C-3), 85.3 (C-4), 109.9 (C-1), 113.2 (C- of isopropylidene gp.)

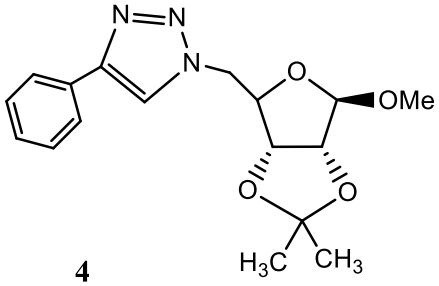
3. PROCEDURE FOR SYNTHESIS OF METHYL 5-AZIDO-5-DEOXY-2,3-*O*-ISOPROPYLIDENE- β -D-RIBOFURANOSIDE (3):



3

To a magnetically stirred solution of the methyl 5-*O*-methanesulphonyl-2,3-*O*-isopropylidene-β-D-ribofuranoside **(2)** (9.3 g, 32.9 mmol) in DMF, NaN_3 (3.13 g, 48.2 mmol) was cautiously added and reaction mixture was stirred for 3-4 h at 90°C. The reaction mixture was diluted with water and extracted by ethyl acetate; organic layer was washed by water, dried (anhyd. Na_2SO_4) and evaporated under reduced pressure to get crude mass. The latter was chromatographed over silica gel (60-120 mesh) using a gradient of hexane-EtOAc (9:1) as eluent to give methyl 5-azido-5-deoxy-2,3-*O*-isopropylidene-β-D-ribofuranoside **(3)** as a colorless liquid (6.7 g, 89%) ; IR (neat) cm^{-1}: 2938, 2101, 1102 ; ^1H NMR (300 MHz, $CDCl_3$): δ 1.29 (s, 3H, C*H*$_3$ of CMe_2), 1.46 (s, 3H, C*H*$_3$ of CMe_2), 3.20-3.26 (m, 1H of H-5), 3.36 (s, 3H, OCH_3), 3.39-3.46 (m, 1H, of H-5), 4.26 (t, 1H, *J* = 7.2 Hz, H-4), 4.55 (s, 2H, H-2 & H-3), 4.95 (s, 1H, H-1); ^{13}C NMR (50 MHz, $CDCl_3$): δ 25.3 (CH_3 of isopropylidene), 26.8 (CH_3 of isopropylidene), 54.3 (C-5), 55.5 (OCH_3), 82.4 (C-2), 85.5 (C-3), 85.7 (C-4), 110.2 (C-1), 112.9 (C- of isopropylidene gp.); ESIMS: 230 $(M+H)^+$.

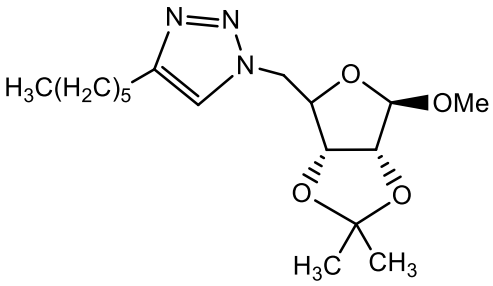
4. PROCEDURE FOR SYNTHESIS OF 1-(METHYL-5-DEOXY-2,3-O-ISOPROPYLIDENE-β-D-RIBOFURANOSID-5-YL)-4-PHENYL-1H-1,2,3-TRIAZOLE (4):



4

Methyl 5-azido-5-deoxy-2,3-*O*-isopropylidene-β-D-ribofuranoside **(3)** (0.54 g, 2.35 mmol) and phenyl acetylene (0.23 ml, 2.35 mmol) were suspended in a 1:1 mixture of tert-butyl alcohol and water (8 ml). Now in the reaction mixture sodium ascorbate (0.028 g, 5 mol%, freshly prepared in water) and copper sulphate (0.014 g, 2 mol%, freshly prepared in water) was added and heterogenous mixture was stirred for 5-6 h at room temperature. After completion of the reaction ice cold water was added which form white precipitate, it on filtration and dried under vacuum give crude mass which was purified by a short column of silica gel (60-120 mesh) and using hexane-EtOAc (2.5:1) as eluent to give 1-(methyl-5-deoxy-2,3-O-isopropylidene-β-D-ribofuranosid-5-yl)-4-phenyl-1H-1,2,3-triazole **(4)** Yield (0.54 g, 70%) m.p.134°C; IR (KBr) cm^{-1}: 3084, 2942, 1655; ^1H NMR (200 MHz, $CDCl_3$ + CCl_4): δ 1.28 (s, 3H, C*H*$_3$ of CMe_2), 1.43 (s, 3H, C*H*$_3$ of CMe_2), 3.39 (s, 3H, OCH_3), 4.42-4.61 (m, 3H, H-4 & H-5), 4.65 (d, 1H, *J* = 5.9 Hz, H-3), 4.74 (d, 1H, *J* = 5.9 Hz, H-2), 5.01 (s, 1H, H-1), 7.29-7.45 (m, 3H, ArH), 7.80-7.85 (m, 3H, ArH & triazolyl H); ^{13}C NMR (50 MHz, $CDCl_3$ + CCl_4): δ 25.3 (CH_3 of isopropylidene), 26.8 (CH_3 of isopropylidene), 53.2 (C-5), 55.4 (OCH_3), 82.1 (C-2), 85.3(C-3) , 85.5 (C-4), 109.7 (C-1), 113.2 (C- of isopropylidene), 120.0 (C-5 triazole), 126.1 (ArH), 128.5 (ArH), 129.1 (ArH), 130.9 (ArH), 148.3 (C-4 triazole); ESIMS: 332 $(M+H)^+$;

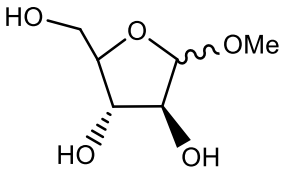
5. PROCEDURE FOR SYNTHESIS OF 4-*n*-HEXYL-1-(METHYL 5-DEOXY-2,3-*O*-ISOPROPYLIDENE-β-D-RIBOFURANOSID-5-YL)-1*H*-1,2,3-TRIAZOLE (5):



5

Methyl 5-azido-5-deoxy-2,3-*O*-isopropylidene-β-D-ribofuranoside **(3)** (0.66 g, 2.88 mmol) and 1-octyne (0.31 ml, 2.88 mmol) were suspended in a 1:1 mixture of tert-butyl alcohol and water (8 ml). Now in the reaction mixture sodium ascorbate (5 mol%, freshly prepared in water) and copper sulphate (2 mol%, freshly prepared in water) was added and heterogenous mixture was stirred for 5-6 h at room temperature. After completion of the reaction ice cold water was added which form white precipitate, it on filtration and dried under vaccum give crude mass which was purified by a short column of silica gel (60-120 mesh) and using hexane-EtOAc (2.5:1) as eluent to give 4-n-hexyl-1-(methyl-5-deoxy-2,3-O-isopropylidene-β-D-ribofuranosid-5-yl)-1H-1,2,3-triazole Yield (0.25g, 82%); m.p.50°C; IR (KBr) cm^{-1}: 3084, 2944, 1651; ^{1}H NMR (300 MHz, $CDCl_3$ + CCl_4): δ 0.90 (t, 3H, *J* = 6.5 Hz, CH_3), 1.23-1.45 (m, 9H, C*H*$_3$ of CMe_2 & 3 x CH_2), 1.50 (s, 3H, C*H*$_3$ of CMe_2), 1.63-1.73 (m, 2H, CH_2), 2.71 (t, 2H, *J* = 7.6 Hz, CH_2), 3.39 (s, 3H, OCH_3), 4.29-4.39 (m, 1H, H-4), 4.46-4.54 (m, 2H, H-5), 4.63 (d, 1H, *J* = 5.9 Hz, H-3), 4.72 (d, 1H, *J* = 5.9 Hz, H-2), 4.99 (s, 1H, H-1), 7.31 (s, 1H, triazolyl *H*); ^{13}C NMR (50 MHz, $CDCl_3$ + CCl_4): δ14.5 (CH_3), 22.7 (CH_2), 25.3 (CH_3 of isopropylidene), 26.0 (CH_2), 26.8 (CH_3 of isopropylidene), 29.2 (CH_2), 29.6 (CH_2), 31.9 (CH_2), 53.1 (C-5), 55.7 (OCH_3), 82.1 (C-2), 85.3 (C-3), 85.5 (C-4), 110.3 (C-1), 113.0 (C- of isopropylidene), 120.8 (C-5 triazole), 148.7 (C-4 triazole); ESIMS: 340 $(M+H)^+$;

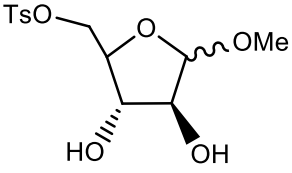
6. PROCEDURE FOR SYNTHESIS OF METHYL D-ARABINOFURANOSIDE (6):



6

To a magnetically stirred reaction D-arabinose (10 g, 66.67 mmol) was dissolved in methanolic HCl and it was allowed at room temperature till the completion of reaction. When a clear solution was obtained, it was neutralized by triethylamine at 0°C. The reaction product was concentrated under reduced pressure and temperature. The latter was chromatographed over silica gel (60-120 mesh) using a gradient of chloroform-methanol (4:1) as eluent to give methyl D-arabinofuranoside (10.1 g, 92.7%). ^{1}H NMR (200 MHz, $CDCl_3$ + DMSO-d_6) δ 3.41 (s, 3H, OCH_3), 3.42-4.23 (m, 3H, H-2, H-4 and H-3), 4.25-4.29 (m, 2H, H-5), 4.97 (s, 1H, H-1).

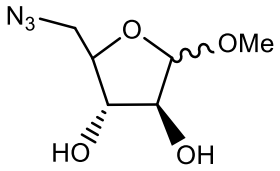
7. PROCEDURE FOR SYNTHESIS OF METHYL 5-O-(p-TOLUENESULPHONYL)-D-ARABINOFURANOSIDE (7):



7

To a magnetically stirred solution of methyl-D-arabinofuranoside **(6)** (6.61 g, 40.3 mmol) in dry dichloromethane (DCM) and triethylamine (5.65 ml, 40.3 mmol) at 0°C, p-toluenesulfonylchloride (7.65 g, 40.3 mmol) was added slowly at 0°C and the stirring was continued for 6 h. The reaction mixture was poured in crushed ice and extracted with DCM. Organic layer was dried (anhyd. Na_2SO_4) and concentrated at reduced pressure and temperature to give crude mass which was chromatographed over silica gel (60-120 mesh) using a gradient of chloroform-methanol (9:1) as eluent to give methyl 5-O-(p-toluenesulphonyl)-D-arabinofuranoside **(7)** (7.1 g, 58%) as colorless oil. IR (neat) cm^{-1}: 3449, 2369, 1654, 1356, 1177; ^1H NMR (200 MHz, $CDCl_3$ + DMSO-d_6) δ 2.42 (s, 3H, CH_3Ph)), 3.32 (s, 3H, OCH_3), 3.58 (s, 1H, OH), 3.91-4.11 (m, 3H, H-3, H-4, H-2) 4.17-4.20 (m, 2H, H-5), 4.78 (s, 1H, H-1) 7.33 (d, 2H, *J*= 8 Hz, ArH), 7.79 (d, 2H, *J*= 8.3 Hz, ArH).

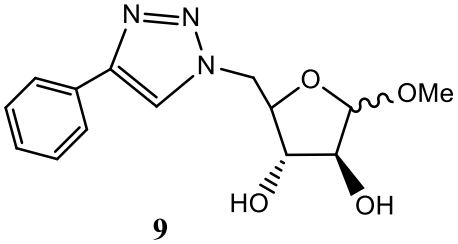
8. PROCEDURE FOR SYNTHESIS OF METHYL 5-AZIDO-5-DEOXY-α-D-ARABINOFURANOSIDE (8):



8

To a magnetically stirred solution of the methyl 5-O-(p-toluenesulphonyl)-D-arabinofuranoside **(7)** (11.0 g, 34.5 mmol) in DMF, NaN_3 (2.69 g, 41.3 mmol) was cautiously added and reaction mixture was stirred for 6-8 h at 90°C-100°C. The solvent was removed by liq. nitrogen under reduced pressure and dry Et_2O (150 ml) was added to remove the insoluble salts by fitration. The crude was chromatographed on a silica gel (60-120 mess) column with $CHCl_3$ and MeOH (95:5) to obtain product (5.87 g, 90%) as a yellow oil; IR (neat) cm^{-1}: 3430, 2105, 1101; ^1H NMR (200 MHz, $CDCl_3$ + DMSO-d_6) δ 3.40 (s, 3H, OCH_3), 3.44-3.55 (m, 2H, H-5), 3.90 (s, 1H, H-3), 4.04-4.11 (m, 2H, H-2 & H-4), 4,85 (s, 1H, H-1); ^{13}C NMR (50 MHz,$CDCl_3$ + DMSO-d_6) δ 52.4 (C-5), 55.5 (OCH_3), 78.6 (C-2), 81.6 (C-3), 83.1 (C-4), 109.1 (C-1); ESIMS: 190 $(M+H)^{+.}$

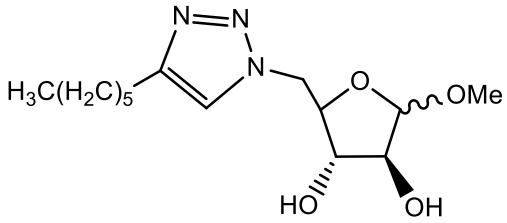
9. PROCEDURE FOR SYNTHESIS OF 1-(METHYL 5-DEOXY-α-D-ARABINOFURANOSID-5-YL)-4-PHENYL-1*H*-1,2,3-TRIAZOLE (9):



9

Methyl 5-azido-5-deoxy-α-D-arabinofuranoside **(8)** (0.45g, 2.30 mmol) and phenyl acetylene (0.26 ml, 2.30 mmol) were suspended in a 1:1 mixture of tert-butyl alcohol and water (8 ml). Now in the reaction mixture sodium ascorbate (5 mol%, freshly prepared in water) and copper sulphate (2 mol%, freshly prepared in water) was added and heterogenous mixture was stirred for 5-6 h at room temperature. After completion of the reaction ice cold water was added which form white precipitate, it on filtration and dried under vaccum give crude mass which was purified by a short column of silica gel (60-120 mesh) and using chloroform-methanol (99:1) as eluent to give 1-(methyl-5-deoxy-α-D-arabinofuranosid-5-yl)-4-phenyl-1H-1,2,3-triazole **(9)** as colorless oil (0.42 g, 62%); IR (Neat) cm^{-1}: 3397, 2927, 2370, 1653, 1100; ^1H NMR (200 MHz, $CDCl_3$) δ 3.23 (s, 3H, OCH_3), 3.87 (s, 1H, H-3), 4.13 (s, 1H, H-2), 4.25 (d, 1H, *J* = 4.2 Hz, H-4), 4.50-4.67 (m, 2H, H-5), 4.79 (s, 1H, H-1), 5.1 (bs, 1H, OH), 7.18-7.32 (m, 3H, ArH), 7.63-7.64 (m, 2H, ArH), 7.86(s, 1H, triazolyl H); ^{13}C NMR (50 MHz,$CDCl_3$) δ 52.1 (C-5), 55.5 (OCH_3), 78.5 (C-2), 80.0 (C-3), 81.9 (C-4), 109.4 (C-1), 122.2(C-5 triazole), 126.0 (ArH), 128.6 (ArH), 129.2 (ArH), 130.4 (ArH), 147.9 (C-4 triazole); ESIMS: 292.2 $(M+H)^+$;

10. PROCEDURE FOR SYNTHESIS OF 4-n-HEXYL-1-(METHYL-5-DEOXY-α-D-ARABINOFURANOSID-5-YL)-1H-1,2,3-TRIAZOLE (10):



10

Methyl 5-azido-5-deoxy-α-D-arabinofuranoside **(8)** (0.52 g, 2.75 mmol) and 1-octyne (0.29 ml, 2.75 mmol) were suspended in a 1:1 mixture of tert-butyl alcohol and water (8 ml). Now in the reaction mixture sodium ascorbate (5 mol%, freshly prepared in water) and copper sulphate (2 mol%, freshly prepared in water) was added and heterogenous mixture was stirred for 5-6 h at room temperature. After completion of the reaction ice cold water was added which form white precipitate, it on filtration and dried under vaccum give crude mass which was purified by a short column of silica gel (60-120 mesh) and using chloroform-methanol (99:1) as eluent to give 4-n-hexyl-1-(methyl-5-deoxy-α-D-arabinofuranosid-5-yl-1H-1,2,3-triazole **(10)** as colorless oil (0.53 g, 65%); IR (neat) cm^{-1}: 3443, 2927, 2271, 1632, 1105, 1034; ^{1}H NMR (200 MHz,$CDCl_3$) δ 0.87 (t, 3H, *J* = 6.3 Hz, CH_3), 1.26-1.33 (m, 4H, 2 x CH_2), 1.57-1.64 (m, 2H, CH_2), 2.62 (t, 2H *J* = 7.8 Hz, CH_2), 3.30 (s, 3H, OCH_3), 3.74 (d, 1H, *J* = 3.4 Hz, H-3), 4.06 (s, 1H, H-2), 4.18-4.23 (m, 1H, H-4), 4.55-4.58 (m, 2H, H-5), 4.75 (s, 1H, H-1), 5.1 (bs, 2H, 2 x OH), 7.47 (s, 1H, triazolyl H); ^{13}C NMR (50 MHz,$CDCl_3$) δ 14.4 (CH_3), 22.7 (CH_2), 25.7 (CH_2), 29.2 (CH_2), 31.7 (CH_2), 51.8 (C-5), 55.3 (OCH_3), 78.5 (C-2), 81.7 (C-3), 81.9 (C-4), 109.5 (C-1), 123.0 (C-5 triazole), 148.3 (C-4 triazole); ESIMS: 286.2 $(M+H)^{+}$;

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