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Full Length Research Paper

1,3,4-oxadiazole and selenadiazole derivatives as new *C*-Glycosyl analogs with MAO-B, antibacterial and antifungal activities

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Abstract

Acetylation of the carbohydrazide derivative 3 afforded the di-*N*-acetyl-tetra-*O*-acetyl derivative 4, while condensation with a variety of aldehydes gave the corresponding carbohydrazones 6a-e. Oxidative cyclization of the *O*-acetylated hydrazones 7a,b afforded the 1,3,4-oxadiazoles 8a,b respectively. Periodate oxidation of 6a-d gave the corresponding formyl derivatives 10a-d, which on reduction resulted in the hydroxymethyl derivatives 11a-d respectively. Prolonged acetylation of the carbohydrazone 11b resulted in 2,3-dihydro-1,3,4-oxadiazole-*O*-acetyl derivative 12b$_2$. Cyclization of the semicarbazone 14 afforded 1,2,3-selenadiazole 16. Methylation of compound 17 afforded 1-[5-(2',3'-dimethoxytetrahydrofuran-1'-yl)-1,2-dimethyl-1H-pyrol-3-yl]ethanone 18. Furthermore, two of the prepared compounds were examined to show the ability to activate MAO-B with different values. In addition a number of prepared compounds showed antibacterial and antifungal activities.

Keywords: Carbohydrazone, Triazole, Oxadiazole, Selenadiazole, Pyrrole, and Mono-amine oxidase-B.

1. INTRODUCTION

1,3,4-oxadiazole are associated with diverse biological activities. Various biological activities like antimicrobial, anti-tubercular, anti-inflammatory, anticonvulsant, hypnotic, anesthetic activity.1,3,4-oxadiazoles showed antibacterial properties similar to those of well known sulfonamide drugs (Sahu et al., 2011).

Heterocyclic compounds containing Selenium are of interest due to their biological and synthetic applications. Many substituted 1,2,3-selenadiazoles have been prepared to-date and some of them show high antimicrobial activity (Al-Smadi and Al-Momani 2008). The antifungal activity of other substituted 1,2,3-selenadiazoles has also been determined (Moawad et al., 1989).

Furthermore, monoamine oxidase plays an important physiological role in the central nervous system by regulating the levels of classical neurotransmitters (Setini et al., 2005), therefore, it used as antidepressants (Edmondson et al., 2007). Low platelet MAO activity has been associated with different psychiatric disorders like schizophrenia (Arrojo et al., 2007), alcoholism (Arrojo et al., 2007), borderline personality disorder (Arrojo et al., 2007) (Paris et al., 2004), bulimia (Arrojo et al., 2007) (Carrasco et al., 2000), and aggression (Garpenstrand et al., 2002). Whereas, increasing MAO activity is associated with cancer (Gabilondo et al., 2008) (Toninello et al., 2006) (Gabilondo et al., 2008) (Toninello et al., 2006), obesity and type-2 diabetes (Carpéné et al., 2006), Alzheimer's disease, and Parkinson's disease (Vindis et al., 2000, Parsian et al., 2004, Petzer et al., 2009 and Rommelspacher et al., 2002) study aimed to evaluate the

effect of the newly prepared compounds on MAO-B and its activity against bacteria and fungi.

2. Experimental Section

2.1. Chemistry

General Procedures. Melting points were determined with a Melt-temp. apparatus and are uncorrected. TLC was performed on Baker-Flex silica gel 1B-F plates and the spots were detected by UV light absorption. IR spectra were recorded on Perkin Elmer. USA Spectrometer. ^{1}H NMR, ^{13}C NMR, and 2D ^{1}H NMR were recorded on JEOL JNM ECA 500 MHz using tetramethylsilane as an internal standard. Mass spectra were recorded on GCMS solution DI Analysis Shimadzu Qp-2010 Plus. Optical rotation was obtained at 22 ºC with a Perkin-Elmer Model 241 Polarimeter 10 cm, 1 mL microcell. Microanalyses were performed at the faculty of Science, Cairo University, Cairo, Egypt. Solutions were evaporated under diminished pressure unless otherwise stated. The ChemDraw-Ultra-8.0 has been used in the nomenclature of the prepared compounds.

2.1.1. Reaction of Compounds **1a** (González et al., 1956) and **3** (El Sadek and Zagzoug 1991) with Acetic Anhydride.

General Methods. A solution of sugar derivative (6.15 mmol) in dry pyridine (20 mL) was treated with acetic anhydride (20 mL), and the mixture was kept overnight with occasional shaking at room temperature. Then it was poured onto crushed ice, the acetyl derivative that separated out, was filtered off, washed with water and dried.

1'-(3-Acetyl-2-methylfuran-5-yl)butane-1',2',3',4'-tetrayl tetraacetate **2a**. It was obtained from **1a**. Recrystallized from ethanol as colourless needles; mp 86-88 ºC (Lit. (González and Sánchez 1965) 86-88 ºC); ^{1}H NMR ($CDCl_3$); δ: 2.03, 2.04, 2.08, 2.09 (4s, 12H, 4OAc), 2.56 (s, 3H, $CH_{3(furan)}$), 4.05 (dd, 1H, H-4'a, $J_{3',4'a}$ 5.35 Hz, $J_{4'b,4'a}$ 12.25 Hz), 4.16 (dd, 1H, H-4'b, $J_{3',4'b}$ 3.05 Hz), 5.08 (m, 1H, H-3'), 5.51 (d, 1H, H-2', $J_{2',3'}$ 7.65 Hz), 5.94 (d, 1H, H-1', $J_{1',2'}$ 4.60 Hz), 6.60 (s, 1H, $CH_{(furan)}$).

1'-[3-(2,2-Diacetylhydrazinecarbonyl)-2-methylfuran-5-yl]butane-1',2',3',4'-tetrayl tetra-acetate **4**. It was obtained from **3**, yield (50.85 %). Recrystallized from dilute ethanol as colourless needles; R_f: 0.1 (hexane: ethyl acetate, 2:1, V/V); mp 140-141 ºC; $[\alpha]_D^{20}$ -29.55; IR (KBr)/cm^{-1}: 3286 (NH), 1749 (OAc), 1728 (N-Ac), 1673 cm^{-1} (CO-NH); ^{1}H NMR ($CDCl_3$); δ: 2.03, 2.04, 2.08, 2.09 (4s, 12H, 4OAc), 2.42 (s, 6H, 2N-$COCH_3$), 2.56 (s, 3H, $CH_{3(furan)}$), 4.11 (dd, 1H, H-4'a, $J_{3',4'a}$ 5.35 Hz, $J_{4'b,4'a}$ 12.25 Hz), 4.26 (dd, 1H, H-4'b, $J_{3',4'b}$ 3.05 Hz), 5.12 (m, 1H, H-3'), 5.61 (m, 1H, H-2'), 6.01 (d, 1H, H-1', $J_{1',2'}$ 5.35 Hz), 6.60 (s, 1H, $CH_{(furan)}$), 8.06 (s, 1H, NH): Anal. Calcd for $C_{22}H_{28}N_2O_{12}$: C, 51.53; H, 5.50; N, 5.45%; found: C, 51.56; H, 5.51; N, 5.47 %.

2.1.2. N,N-Diacetyl-2-methyl-5(1',2',3',4'tetrahydro xy)furan-3-carbohydrazide **5**. A solution of *O*-acetyl sugar **4** (0.0402 mmol) in a mixture of methanol (4 mL) and ammonia (2 mL) was kept overnight at room temperature. The excess methanolic ammonia was evaporated in a current of air, crystalline mass was obtained, yield (97.98%). Recrystallized from ethanol as colourless needles; R_f: 0.54 (chloroform: methanol, 3:1, V/V); mp 170-172 ºC; IR (KBr)/cm^{-1}: 3363-3236 (OH), 3216 (NH), 1673 (N-$COCH_3$), 1642 (CO-N), 1591 cm^{-1} (C=N); Anal. Calcd for $C_{14}H_{20}N_2O_8$: C, 48.81; H, 5.84; N, 8.16 %; found: C, 48.84; H, 5.85; N, 8.14 %.

2.1.3. Reaction of Compound **3** with a Number of Aldehydes.

General Method. A solution of **3** (3.0 g, 11.54 mmol) in ethanol (10 mL) containing acetic acid (0.1 mL) was boiled with the corresponding aldehyde (11.54 mmol), and the reaction mixture was refluxed on water bath for 30 minutes. After cooling 3-carbohydrazone that separated out, was filtered off, washed with little ethanol, and dried.

2-Methyl-N-[(2-phenyl-2H-1,2,3-triazol-4-yl)methylene]-5-(1',2',3',4'-tetrahydroxybutyl)-furan-3-carbohydrazone **6a.** It was obtained from **3**, yield (83.54 %). Recrystallized from ethanol as colourless needles; R_f: 0.52 (chloroform: methanol, 5:1, V/V); mp 213-215 ºC; $[\alpha]_D^{20}$ -5.7; IR (KBr)/cm^{-1}: 3352 (OH), 3266 (NH), 1654 (CO-amide), 1626 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 2.50 (s, 3H, $CH_{3(furan)}$), 3.50 (m, 3H, H-4'a, H-3', H-4'b), 3.64 (m, 5H, H-1', H-2', 2'-OH, 3'-OH, 4'-OH), 4.81 (s, 1H, 1'-OH), 6.78 (s, 1H, $CH_{(furan)}$), phenyl protons: 7.32 (t, 1H, *p*-H), 7.45 (t, 2H, *o*-H), 7.97 (m, 2H, *m*-H), 8.22 (s, 1H, CH=N), 8.53 (s, 1H, $CH_{(triazole)}$), 11.57 (s, 1H, NH); MS: m/z (%), 415 (0.68, M^+), 397 (1.72, M^+-H_2O), 379 (0.5, M^+-$2H_2O$), 337 (2.06, M^+-$2H_2O$-CH_2=C=O), 325 (17.73), 324 (19.68), 294 (0.96), 229 (10.15), 211 (3.03), 170 (9.83), 155 (40.16), 154 (18.18), 153 (4.79), 152 (2.03), 151(12.33), 144 (0.25), 139 (7.23), 138 (11.89), 137 (100), 110 (14.90), 91 (20.24), 77 (24.53), 43 (57.83); Anal. Calcd for $C_{19}H_{21}N_5O_6$: C, 54.96; H, 5.08; N, 16.86%; found: C, 54.94; H, 5.10; N, 16.86%.

N-[(2-(*p*-Bromophenyl)-2H-1,2,3-triazol-4 yl)methylene]-2-methyl-5-(1',2',3',4'-tetrahydroxybutyl)furan-3-carbohydrazone **6b.** It was obtained from **3**, yield (91.63 %). Recrystallized from ethanol as colourless needles; R_f: 0.76 (chloroform: methanol,

5:1, V/V); mp 218-220 °C; $[\alpha]_D^{20}$ -4.21; IR (KBr)/cm^{-1}: 3379 (OH), 3242 (NH), 1671 (CO-amide), 1650 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 2.51 (s, 3H, $CH_{3(furan)}$), 3.41 (m, 1H, H-4'a), 3.55 (m, 3H, H-3', H-4'b, H-2'), 4.39 (m, 1H, 4'-OH), 4.52 (d, 1H, 3'-OH, *J* 7.65 Hz), 4.65 (d, 1H, 2'-OH, *J* 5.35 Hz), 4.78 (d, 1H, H-1', *J* 6.85 Hz), 5.22 (d, 1H, 1'-OH, *J* 6.85 Hz), 6.78 (s, 1H, $CH_{(furan)}$), *p*-bromophenyl protons: 7.75 (d, 2H, *m*-H), 7.96 (d, 2H, *o*-H), 8.41 (s, 1H, CH=N), 8.55 (s, 1H, $CH_{(triazole)}$), 11.63 (s, 1H, NH). After shaking with D_2O, the NH proton and the four OH protons disappeared; MS: m/z (%), 495/493 (0.18, 0.17, M^+), 477/475 (3.09, 3.07, M^+-H_2O), 459/457 (0.25, 0.17, M^+-2H_2O), 417/415 (1.11, 1.25, M^+-2H_2O-CH_2-C=O), 405/403 (8.56, 6.38), 404/402 (24.68, 23.25), 388/386 (0.75, 0.64), 374/372 (0.55, 0.9, M^+-2H_2O-CH_2-C=O-CH_3CO), 267/265 (13.80, 14.18), 250/248 (9.08, 7.63), 229 (14.49), 211 (34.32), 170 (10.92), 169 (19.60), 155 (23.48), 154 (27.70), 153 (9.24), 152 (3.09), 151 (14.57), 139 (6.11), 138 (15.87), 137 (100), 110 (17.07), 90 (16.23), 63 (11.84), 61 (8.20), 55 (11.63), 53 (12.73); Anal. Calcd for $C_{19}H_{20}BrN_5O_6$: C, 46.15; H, 4.07; N, 14.15; Br, 16.18%; found: C, 46.17; H, 4.08; N, 14.17; Br, 16.17%.

Ethyl2-methyl-5-[(2-(2-methyl-5-(1',2',3',4'-tetrahydroxybutyl)furan-3-carbonyl)-hydrazono)methyl]furan-3-carboxylate **6c.** It was obtained from **3**, yield (89.64 %). Recrystallized from ethanol as colourless needles; R_f: 0.42 (chloroform: methanol, 7:1, V/V); mp 211-213 °C; $[\alpha]_D^{20}$ -29.55; IR (KBr)/cm^{-1}: 3476 (OH), 3247 (NH), 1696 (CO-ester), 1662 (CO-amide), 1623 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 1.25 (t, 3H, $CH_{3(ester)}$, *J* 6.85 Hz), 2.48 (s, 3H, $CH_{3(furan-1)}$), 2.58 (s, 3H, $CH_{3(furan-2)}$, 3.51 (m, 3H, H-4'a, H-3', H-4'b), 4.06 (s, 1H, H-2'), 4.21 (q, 2H, $CH_{2(ester)}$, *J* 6.85 Hz), 4.38 (t, 1H, 4'-OH, *J* 5.35 Hz), 4.50 (d, 1H, 3'-OH, *J* 7.65 Hz), 4.64 (d, 1H, 2'-OH, *J* 5.35 Hz), 4.75 (d, 1H, H-1', *J* 6.10 Hz), 5.19 (d, 1H, 1'-OH, *J* 6.90 Hz), 6.74 (s, 1H, $CH_{(furan-1)}$), 7.04 (s, 1H, $CH_{(furan-2)}$), 8.17 (s, 1H, CH=N), 11.42 (s, 1H, NH). After shaking with D_2O, the NH proton and the four hydroxyl protons disappeared; MS: m/z (%), 424 (0.58, M^+), 406 (2.93, M^+-H_2O), 388(0.44, M^+-2H_2O), 361 (1.39, M^+-H_2O-OEt), 346 (1.40, M^+-2H_2O-CH_2=C=O), 334 (9.91), 333 (30.43), 332 (11.71), 317 (0.66), 303 (1.66, M^+-2H_2O-CH_2=C=O-$COCH_3$), 287 (41.70), 229 (26.45), 211 (25.67), 155 (6.39), 154 (19.37), 153 (14.25), 152 (7.74), 139 (6.81), 138 (12.33), 137 (58.13), 124 (2.08), 123 (8.89), 110 (15.21), 109 (8.06), 107 (2.61), 93 (4.52), 92 (2.24), 81 (10.65), 79 (11.60), 68 (3.00), 43 (100, $COCH_3$); Anal. Calcd for $C_{19}H_{24}N_2O_9$: C, 53.76; H, 5.70; N, 6.61%; found: C, 53.77; H, 5.70; N, 6.60%.

2.1.4. Reaction of Compounds of **6a-c** with Acetic Anhydride

General Methods**.** A solution of sugar derivative **6a-c** (6.15 mmol) in dry pyridine (20 mL) was treated with acetic anhydride (20 mL), and the mixture was kept overnight with occasional shaking at room temperature. Then it was poured onto crushed ice, the acetyl derivative that separated out, was filtered off, washed with water and dried.

1'-[2-Methyl-3-(2-((2-phenyl-2H-1,2,3-triazol-4-yl)methylene)hydrazinecarbonyl)-furan-5-yl]butane-1',2',3',4'-tetrayl tetraacetate **7a**. It was obtained from **6a**, yield (98 %). Recrystallized from dilute ethanol as colourless needles; R_f: 0.58 (hexane: ethyl acetate, 2:1, V/V); mp 161-162 °C; $[\alpha]_D^{20}$ -27.03; IR (KBr)/cm^{-1}: 3163 (NH), 1752 (OAc), 1665 (CO-amide), 1610 cm^{-1} (C=N); ^{1}H NMR ($CDCl_3$); δ: 2.06 (s, 12H, 4OAc), 2.68 (s, 3H, $CH_{3(furan)}$), 4.15 (dd, 1H, H-4'a, $J_{3',4'a}$ 5.35 Hz), 4.25 (dd, 1H, H-4'b, $J_{3',4'b}$ 3.05, $J_{4'b,4'a}$ 12.20 Hz), 5.23 (m, 1H, H-3'), 5.64 (m, 1H, H-2'), 6.07 (d, 1H, H-1', $J_{1',2'}$ 3.85 Hz), 6.74 (s, 1H, $CH_{(furan)}$), phenyl protons: 7.67-7.44 (m, 3H, *p*-H, *o*-H), 8.05 (m, 2H, *m*-H), 8.11 (s, 1H, CH=N), 8.12 (s, 1H, $CH_{(triazole)}$, 12.19 (s, 1H, NH); MS: m/z (%), 585 (1.06, M^++2), 584 (4.87, M^++1), 583 (12.59, M^+), 523 (24.33, M^+-AcOH), 463 (19.66), 438 (0.98), 422 (22.51), 421 (63.58), 408 (13.66), 403 (12.75), 398 (11.57), 397 (55.58), 379 (19.77), 378 (8.54), 366 (16.56), 362 (18.55), 361 (35.27), 337 (9.89), 336 (9.79), 325 (20.22), 324 (100), 295 (8.94), 253 (2.74), 251 (10.06), 250 (25.70), 235 (25.78), 234 (12.34), 209 (15.02), 294 (5.09), 208 (12.61), 207 (17.98), 193 (62.65), 192 (19.99), 191 (14.76), 188 (12.84), 175 (19.17), 151 (18.90), 138 (15.27), 137 (84.61), 77 (20.22) ; Anal. Calcd for $C_{27}H_{29}N_5O_{10}$: C, 55.57; H, 5.03; N, 12.02%; found: C, 55.57; H, 5.01; N, 12.00%.

1'-[3-(2-((2-(*p*-Bromophenyl)-2H-1,2,3-triazol-4-yl)methylene)hydrazinecarbonyl)-2-methylfuran-5-yl]butane-1',2',3',4'-tetrayl tetraacetate **7b**. It was obtained from **6b**, yield (82.09 %). Recrystallized from dilute ethanol as colourless needles; R_f: 0.59 (hexane: ethyl acetate, 2:1, V/V); mp 158-160 °C; $[\alpha]_D^{20}$ -75.33; IR (KBr)/cm^{-1}: 3203 (NH), 1746 (OAc), 1652 (CO-amide), 1629 cm^{-1} (C=N); ^{1}H NMR ($CDCl_3$); δ: 2.08, 2.09, 2.10, and 2.11 (4s, 12H, 4OAc), 2.67 (s, 3H, $CH_{3(furan)}$), 4.17 (m, 1H, H-4'a, $J_{4'b,4'a}$12.25 Hz), 4.26 (dd, 1H, H-4'b, $J_{3',4'b}$ 2.30 Hz), 5.22 (m, 1H, H-3'), 5.66 (dd, 1H, H-2', $J_{1',2'}$ 4.60 Hz, $J_{2',3'}$ 8.45 Hz), 6.06 (m, 1H, H-1'), 6.70 (s, 1H, $CH_{(furan)}$), *p*-bromophenyl protons: 7.71 (m, 2H, *m*-H), 8.01 (m, 4H, *o*-H, CH=N, $CH_{(triazole)}$), 12.11 (bs, 1H, NH); MS: m/z (%), 664/662 (1.17, 1.19, M^++1), 663/661, (3.44, 3.29, M^+),

620/618 (0.43, 0.42), 603/601 (7.57, 7.01), 532/530 (0.21, 0.31), 518/516 (0.66, 0.62), 501/499 (19.64, 19.71), 459/457 (8.01, 7.76), 446/444 (5.01, 5.40), 442/440 (6.94, 7.63), 441/439 (12.87, 11.68), 404/402 (25.09, 26.00), 397 (46.60), 375/373 (0.51, 0.60), 374/372 (1.20, 1.37), 324 (0.49), 253 (12.32), 250 (22.27), 235 (21.74), 234 (10.32), 209 (11.89), 207 (16.85), 193 (56.92), 192 (20.46), 191 (13.68), 175 (17.91), 155 (11.41), 153 (10.06), 151 (33.66), 137 (100), 123 (9.03), 121 (11.27), 115 (16.48), 110 (12.89), 95 (9.48), 60 (15.40), 53 (8.00); Anal. Calcd for $C_{27}H_{28}BrN_5O_{10}$: C, 48.93; H, 4.25; N, 10.55; Br, 12.07%; found: C, 48.95; H, 4.26; N, 10.57; Br, 12.06%.

1'-[3-(2-((3-(Ethoxycarbonyl)-2-methylfuran-2-yl)methylene)hydrazinecarbonyl)-2-methylfuran-5-yl]butane-1',2',3',4'-tetrayl tetraacetate **7c**. It was obtained from **6c**, yield (71.63 %). Recrystallized from dilute ethanol as a colourless semi-solid material; R_f: 0.24 (hexane:ethyl acetate, 2:1, V/V); mp 74-75 ºC; IR (KBr)/cm^{-1}: 3273 (NH), 1752 (OAc), 1700(CO-ester), 1685 (CO-amide), 1602 cm^{-1} (C=N); Anal. Calcd for $C_{27}H_{32}N_2O_{13}$: C, 54.74; H, 5.43; N, 4.75%; found: C, 54.73; H, 5.44; N, 4.73 %.

2.1.5. Reaction of compounds **7a,b** with Yellow Mercuric Oxide.

General Methods. A solution of carbohydrazide (5.523 mmol) in dry ether (75 mL) was stirred with yellow mercuric oxide (4.8 g), magnesium oxide (0.48 g), and iodine (4.0 g) at room temperature for 48 hours under anhydrous conditions. The reaction mixture was filtered off, and the filtrate washed with potassium iodide solution, sodium thiosulphate, and water respectively, and dried over anhydrous sodium sulphate. On evaporation of the dried filtrate, a colourless crystalline mass was obtained. An additional crop was obtained by extracting the inorganic residue with chloroform which upon concentration yielded the same product.

1'-[2-Methyl-3-(5-(2-phenyl-2H-1,2,3-triazol-4-yl)1,3,4-oxadiazol-2-yl)furan-5-yl]butane-1',2',-3',4'-tetrayl tetraacetate **8a.** It was obtained from **7a**, yield (50.8 %). Recrystallized from ethanol as colourless needles; R_f: 0.6 (hexane: ethyl acetate, 2:1, V/V); mp 180-181 ºC; $[\alpha]_D^{20}$ -13.46; IR (KBr)/cm^{-1}: 1746 (OAc), 1632, 1612 cm^{-1} (C=N); ^{1}H NMR ($CDCl_3$); δ: 2.05, 2.08, 2.10, 2.12 (4s, 12H, 4OAc), 2.73 (s, 3H, $CH_{3(furan)}$), 4.15 (dd, 1H, H-4'a, $J_{3',4'a}$ 5.35 Hz, $J_{4'b,4'a}$ 12.60 Hz), 4.26 (dd, 1H, H-4'b, $J_{3',4'b}$ 3.05 Hz), 5.22 (m, 1H, H-3'), 5.63 (dd, 1H, H-2', $J_{1',2'}$ 4.55 Hz, $J_{2',3'}$ 7.65 Hz), 6.09 (d, 1H, H-1', $J_{1',2'}$ 4.55 Hz), 6.87(s, 1H, $CH_{(furan)}$), phenyl protons: 7.424 (t, 1H, *p*-H), 7.528 (t, 2H, *o*-H), 8.164 (d, 2H, *m*-H), 8.398 (s, 1H, $CH_{(triazole)}$); MS: m/z (%), 582 (1.66, M^++1), 581 (4.97, M^+), 521 (6.28), 479 (4.48), 436 (2.34), 420 (20.67), 419 (48.07), 406 (8.29), 402 (2.80), 378 (19.78), 377 (71.02), 365 (8.84), 364 (17.59), 360 (28.05), 334 (7.28), 323 (21.38), 322 (100), 321 (9.31), 293 (2.21), 279 (7.08), 188 (2.72), 172 (13.59), 137 (10.14), 115 (17.79), 105 (2.30), 91 (9.98), 77 (13.94), 55 (7.09); Anal. Calcd for $C_{27}H_{27}N_5O_{10}$: C, 55.77; H, 4.68; N, 12.03%; found: C, 55.76; H, 4.68; N, 12.04 %.

1'-[3-(5-(2-(*p*-Bromophenyl)-2H-1,2,3-triazol-4-yl)-1,3,4-oxadiazol-2-yl)-2-methyl-furan-5-yl]butane-1',2',3',4'-tetrayl tetraacetate **8b.** It was obtained from **7b**, yield (84.31 %). Recrystallized from ethanol as colourless needles; R_f: 0.56 (hexane: ethyl acetate, 3:1, V/V); mp 140-142 ºC; $[\alpha]_D^{20}$ -13.86; IR (KBr)/cm^{-1}: 1745 (OAc), 1632 cm^{-1} (C=N); ^{1}H NMR ($CDCl_3$); δ: 2.06, 2.08, 2.10, 2.13 (4s, 12H, 4OAc), 2.74 (s, 3H, $CH_{3(furan)}$), 4.15 (dd, 1H, H-4'a, $J_{3',4'a}$ 5.35 Hz, $J_{4'b,4'a}$ 12.25 Hz), 4.27 (dd, 1H, H-4'b, $J_{3',4'b}$ 3.05 Hz), 5.22 (m, 1H, H-3'), 5.64 (m, 1H, H-2'), 6.09 (d, 1H, H-1', $J_{1',2'}$ 4.60 Hz), 6.87 (s, 1H, $CH_{(furan)}$), *p*-bromophenyl protons: 7.66 (d, 2H, *m*-H), 8.07 (d, 2H, *o*-H), 8.41 (s, 1H, $CH_{(triazole)}$); MS: m/z (%), 662/660 (0.17, 0.18, M^++1), 661/659 (0.34, 0.36, M^+), 601/599 (0.74, 0.81, M^+-AcOH), 559/557 (0.54, 0.48, M^+-AcOH-CH_2=C=O), 530/528 (0.22, 0.25, M^+-AcOH-CH_2=C=O-CHO), 516/514 (0.33, 0.25, M^+-AcOH-CH_2=C=O-CHO-CH_2), 457/455 (7.77, 8.45, M^+-AcOH-CH_2=C=O-CHO-CH_2-OAc), 444/442 (2.16, 2.31), 414/412 (1.35, 1.16, M^+-AcOH-CH_2=C=O-CHO-CH_2-OAc-Ac), 402/400 (9.43, 8.97), 373/371 (0.31, 0.13), 253 (12.42), 251 (12.57), 169 (13.22), 155 (11.64), 141 (13.46), 127 (17.33), 125 (13.72), 113 (21.79), 111 (23.91), 99 (31.66), 98 (9.05), 97 (40.88), 96 (15.10), 85 (66.38), 84 (11.55), 83 (33.14), 82 (20.47), 71 (85.10), 70 (16.37), 69 (26.15), 57 (100), 56 (16.02), 55 (25.65); Anal. Calcd for $C_{27}H_{26}BrN_5O_{10}$: C, 49.11; H, 3.95; N, 10.61; Br, 12.11%; found: C, 49.10; H, 3.97; N, 10.60; Br, 12.10%.

2.1.6. Deacetylation of Compounds **8a,b.**

General Methods. A solution of compound 8a,b (0.0402 mmol) in a mixture of methanol (4 mL) and ammonia (2 mL) was kept overnight at room temperature. The excess methanolic ammonia was evaporated in a current of air, crystalline mass was obtained.

1'-[2-Methyl-3-(5-(2-phenyl-2H-1,2,3-triazol-4-yl)-1,3,4-oxadiazol-2-yl)furan-5-yl]-butane-1',2',3',4'-tetraol **9a.** It was obtained from **8a**, yield (97.5 %). Recrystallized from ethanol as colourless needles; mp 216-217 ºC; $[\alpha]_D^{20}$ -5.98; IR (KBr)/cm^{-1}: 3337-3272 (OH), 1621 cm^{-1} (C=N); MS: m/z (%), 414 (0.35, M^++1), 413 (1.12, M^+), 395 (1.84, M^+-H_2O), 324 (4.29), 323 (28.01), 322 (100), 321 (13.58), 306 (4.70), 172 (8.81), 137 (6.52), 136 (5.38), 117 (3.17), 110 (3.58), 91 (8.13), 77 (11.69), 74 (6.33), 61 (6.81), 56 (6.57), 53 (11.26), 51 (5.36); Anal. Calcd for $C_{19}H_{19}N_5O_6$:

C, 55.21; H, 4.65; N, 16.96%; found: C, 55.20; H, 4.63; N, 16.94%.

1'-[3-(5-(2-(*p*-Bromophenyl)-2H-1,2,3-triazol-4-yl)-1,3,4-oxadiazol-2-yl)-2-methyl-furan-5-yl]butane-1',2',3',4'-tetraol **9b.** It was obtained from **8b**, yield (97.31 %). Recrystallized from ethanol as colourless needles; mp 224-225 ºC; $[\alpha]_D^{20}$ -6.62; IR (KBr)/cm^{-1}: 3354-3280 (OH), 1615 cm^{-1} (C=N); MS: m/z (%), 493/491 (0.75, 0.72, M$^+$), 475/473 (3.67, 3.48), 444/442 (1.67, 1.54), 415/413 (2.95, 2.86), 403 (28.58), 402/400 (100, 99.71), 386/384 (5.67, 5.88), 359/357 (2.69, 2.84), 322 (7.02), 321 (6.38), 250 (8.77), 137 (15.66), 136 (11.31), 115 (5.81), 108 (3.22), 105 (3.62), 90 (11.52), 74 (16.91), 63 (9.19), 61 (15.45), 60 (4.93), 56 (15.36), 55 (8.20), 53 (22.29); Anal. Calcd for $C_{19}H_{18}BrN_5O_6$: C, 46.35; H, 3.67; N, 14.23; Br, 16.22%; found: C, 46.36; H, 3.69; N, 14.23; Br, 16.23%.

2.1.7. Reduction of Compounds **6a-d** with Sodium Borohydride.

General Method. A solution of 5-(1',2',3',4'-tetrahydroxybutyl)furan derivative **6a-d** (11.44 mmol) in distilled water (20 mL) was treated with a solution of sodium metaperiodate (7.343 g, 34.33 mmol) in distilled water (20 mL) dropwise with continuous stirring for 3 hours, the formyl derivative that separated out, was filtered off, washed with water, and dried.

5-Formyl-2-methyl-N-[(2-phenyl-2H-1,2,3-ts1riazol-4-yl)methylene]furan-3-carbohydraz-one **10a.** It was obtained from **6a**, yield (97 %). Recrystallized from ethanol as colourless needles; R_f: 0.8 (chloroform: methanol, 10:1, V/V); mp 208-210 ºC; IR (KBr)/cm^{-1}: 3218 (NH), 1690 (CO-aldehyde), 1650 (CO-amide), 1590 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 2.65 (s, 3H, $CH_{3(furan)}$), 6.90 (s, 1H, $CH_{(furan)}$), phenyl protons; 7.44 (t, 1H, *p*-H), 7.57 (m, 2H, *o*-H), 8.02 (d, 2H, *m*-H), 8.43 (s, 1H, CH=N), 8.54 (m, 1H, $CH_{(triazole)}$), 9.59 (s, 1H, CHO), 11.90 (s, 1H, NH). ^{13}C NMR (DMSO-d_6); δ: 14.45 (C-14), 117.52 (C-13), 119.03 (C-12), 128.76 (C-11), 130.40 (C-10), 130.58 (C-9), 134.98 (C-8), 138.82 (C-7), 139.35 (C-6), 145.93 (C-5), 150.39 (C-4), 158.77 (C-3), 164.11 (C-2), 179.00 (C-1); MS: m/z (%), 324 (3.22, M$^+$+1), 323 (15.30, M$^+$), 167 (10.89), 153 (36.26), 149 (28.39), 138 (12.44), 137 (100), 136 (15.58), 129 (20.89), 115 (14.32), 111 (16.09), 109 (14.98), 105 (13.42), 101 (12.18), 99 (11.64), 98 (19.16), 97 (29.19), 96 (16.83), 95 (35), 91 (23.69), 87 (20.22), 85 (29.80), 84 (22.75), 83 (39.58), 82 (17.37), 81 (25.93), 79 (13.02), 77 (32.62), 74 (17.29), 73 (67.05), 72 (12.06), 71 (48.42), 70 (27.25), 69 (55.96), 68 (14.05), 67 (24.81), 61 (16.60), 60 (87.29), 59 (18.84), 57 (97.54), 56 (32.25), 55 (88.24), 54 (11.70), 53 (15.46), 51 (16.01); Anal. Calcd for $C_{16}H_{13}N_5O_3$: C, 59.42; H, 4.02; N, 21.64%; found: C, 59.44; H, 4.05; N, 21.66%.

5-Formyl-2-methyl-N-[(2-(*p*-bromophenyl)-2H-1,2,3-triazol-4-yl)methylene]furan-3-carbohydrazone **10b**. It was obtained from **6b**, yield (97.21 %). Recrystallized from ethanol as colourless needles; R_f: 0.46 (chloroform: methanol, 30:1, V/V); mp 230-232 ºC; IR (KBr)/cm^{-1}: 3312 (NH), 1689 (CO-aldehyde), 1670 (CO-amide), 1587 cm^{-1} (C=N); MS: m/z (%), 404/402 (1.69, 2.24, M$^+$+1), 403/401 (5.48, 5.39, M$^+$), 250 (2.43), 248 (2.78), 185 (2.14), 155 (5.13), 153 (25.79), 138 (8.90), 137 (100), 136 (10.87), 95 (17.31), 76 (5.19), 75 (4.47), 63 (4.37), 53 (6.52); Anal. Calcd for $C_{16}H_{12}BrN_5O_3$: C, 74.77; H, 3.03; N, 17.40; Br, 19.88%; found: C, 47.78; H, 3.01; N, 17.41; Br, 19.87%.

Ethyl 5-[(2-(5-formyl-2-methylfuran-3-carbonyl)hydrazono)methyl]-2-methylfuran-3-carboxylate **10c.** It was obtained from **6c**, yield (95.24 %). Recrystallized from ethanol as colourless needles; R_f: 0.61 (chloroform: methanol, 25: 1, V/V); mp 160 ºC (dec); IR (KBr)/cm^{-1}: 3218 (NH), 1708 (CO-ester), 1698 (CO-aldehyde), 1688 (CO-amide), 1627 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 1.25 (t, 3H, $CH_{3(ester)}$, *J* 6.90 Hz), 2.59 (s, 3H, $CH_{3(furan-2)}$), 2.63 (s, 3H, $CH_{3(furan-1)}$), 4.21 (q, 2H, $CH_{2(ester)}$, *J* 6.90 Hz), 7.12 (s, 1H, $CH_{(furan-2)}$), 7.88 (s, 1H, $CH_{(furan-1)}$), 8.15 (s, 1H, CH=N), 9.56 (s, 1H, CHO), 11.70 (s, 1H, NH); MS: m/z (%), 333 (2.76, M$^+$+1), 332 (13.66, M$^+$), 153 (35.06), 138 (9.89), 137 (100), 136 (14.94), 129 (8.15), 98 (8.40), 97 (12.48), 96 (8.50), 95 (22.71), 85 (11.54), 84 (9.35), 83 (14.90), 81 (12.33), 79 (10.60), 73 (17.29), 71 (16.69), 70 (9.91), 69 (22.87), 68 (7.69), 67 (13.39), 60 (29.41), 57 (29.78), 56 (1.96), 55 (34.71), 53 (9.85), 51 (8.34); Anal. Calcd for $C_{16}H_{16}N_2O_6$: C, 57.82; H, 4.82; N, 8.41%; found: C, 57.83; H, 4.85; N, 8.43%.

2.1.8. Reduction of compounds **10a-d** with Sodium Borohydride.

General Method. A suspension of 5-formyl derivative **10a-d** (15.177 mmol) in distilled water (20 mL) was treated with a solution of sodium borohydride (1.82 g) in distilled water (20 mL), and the reaction mixture kept overnight at room temperature with occasional stirring, the 5-hydroxymethyl derivative that separated out, was filtered off, washed with water, and dried.

5-(Hydroxymethyl)-2-methyl-N-[(2-phenyl-2*H*-1,2,3-triazol-4-yl)methylene]furan-3-carbohydrazone **11a**. It was obtained from **10a**, yield (94.53 %). Recrystallized from N,N-dimethylsulfoxide as colourless needles; R_f: 0.36 (chloroform: methanol, 20:1, V/V); mp 230-232 ºC; IR (KBr)/cm^{-1}: 3371 (OH), 3225 (NH), 1656 (CO-amide), 1639 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 2.55 (s, 3H, $CH_{3(furan)}$), 4.36 (s, 2H, CH_2), 5.33 (bs, 1H, OH), 6.74 (s, 1H, $CH_{(furan)}$), phenyl protons: 7.42 (t, 1H, *p*-H), 7.56 (t, 2H, *o*-H), 8.01 (d, 2H, *m*-H), 8.39 (s, 1H, CH=N), 8.54 (s, 1H, $CH_{(triazole)}$), 11.70 (bs, 1H, NH); MS: m/z (%), 327

(1.91, M^++2), 326 (13.96, M^++1), 325 (71.43, M^+), 294 (11.30), 173 (5.17), 155 (51.64), 140 (32.24), 139 (100), 138 (48.91), 137 (1.01), 121 (7.21), 112 (9.45), 110 (10.17), 97 (21.53), 77 (49.82), 69 (28.88), 65 (8.05), 64 (9.02), 55 (6.59), 53 (11.14), 52 (12.78), 51 (19.44); Anal. Calcd for $C_{16}H_{15}N_5O_3$: C, 59.05; H, 4.65; N, 21.50%; found: C, 59.07; H, 4.65; N, 21.53%.

5-(Hydroxymethyl)-2-methyl-N-[(2-(*p*-bromophenyl)-2H-1,2,3-triazol-4-yl)methyl-ene]furan-3-carbohydrazone **11b**. It was obtained from **10b**, yield (78.24 %). Recrystallized from N,N-dimethylsulfoxide as colourless needles; R_f: 0.35 (chloroform: methanol, 20:1, V/V); mp 248-250 ºC; IR (KBr)/cm^{-1}: 3289 (OH), 3219 (NH), 1652 (CO-amide), 1604 cm^{-1} (C=N); MS: m/z (%), 406/404 (6.06, 7.30, M^++1), 405/403 (33.71, 34.72, M^+), 171/169 (5.75, 6.53), 157 (15.91), 156 (5.73), 155 (64.45), 140 (46.19), 139 (100), 138 (53.02), 137 (15.34), 112 (8.77), 110 (9.33), 97 (20.29), 90.05 (15.08), 80 (9.32), 79 (8.28), 76 (11.76), 75 (9.99), 69 (27.60), 63 (9.96), 55 (10.47), 53 (13.26), 52 (25.22), 51 (16.03), 50 (12.55); Anal. Calcd for $C_{16}H_{14}BrN_5O_3$: C, 47.53; H, 3.48; N, 17.31; Br, 19.78%; found: C, 47.54; H, 3.49; N, 17.33; Br, 19.77%.

Ethyl 5-[(2-(5-(hydroxymethyl)-2-methylfuran-3-carbonyl)hydrazono)methyl]-2-methyl-furan-3-carboxylate **11c**. It was obtained from **10c**, yield (96.72 %). Recrystallized from ethanol as colourless needles; R_f: 0.5 (chloroform: methanol, 15:1, V/V); mp 209-211 ºC; IR (KBr)/cm^{-1}: 3371 (OH), 3316 (NH), 1694 (CO-ester), 1662 (CO-amide), 1624 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 1.25 (t, 3H, $CH_{3(ester)}$, *J* 7.65 Hz), 2.49 (s, 3H, $CH_{3(furan-1)}$), 2.58 (s, 3H, $CH_{3(furan-2)}$), 4.21 (q, 2H, $CH_{2(ester)}$, *J* 7.65 Hz), 4.33 (s, 2H, CH_2), 5.31 (s, 1H, OH), 6.71 (s, 1H, $CH_{(furan-1)}$, 7.05 (s, 1H, $CH_{(furan-2)}$), 8.17 (s, 1H, CH=N), 11.38 (s, 1H, NH). After shaking with D_2O, the NH proton and the OH proton disappeared; MS: (m/z), 335 (2.98, M^++1), 334 (13.81, M^+), 315 (14.48), 314 (12.45), 304 (10.12), 303 (20.57), 287 (4.50), 276 (9.24), 275 (31.03), 244 (5.81), 230 (5.88), 216 (5.57), 215 (5.40), 186 (3.89), 155 (20.22), 153 (9.24), 152 (13.37), 140 (8.83), 139 (100), 138 (9.15), 137 (24.87), 129 (12.91), 123 (12.02), 115 (9.88), 108 (7.73), 98 (7.10), 97 (11.66), 96 (7.10), 95 (9.09), 81 (9.58), 79 (21.77), 77 (6.65), 73 (25.03), 69 (22.15), 60 (22.99), 57 (24.20), 55 (24.87), 53 (14.91), 52 (18.57), 51 (14.92); Anal. Calcd for $C_{16}H_{18}N_2O_6$: C, 57.48; H, 5.41; N, 8.39%; found: C, 57.48; H, 5.43; N, 8.38%.

5-(Hydroxymethyl)-2-methyl-N-(3-phenylallylidene)furan-3-carbohydrazone **11d.** It was obtained from **10d**, yield (81.21 %). Recrystallized from ethanol as pale yellow needles; R_f: 0.33 (chloroform: methanol, 20:1, V/V); mp 129-130 ºC; IR (KBr)/cm^{-1}: 3402 (OH), 3241 (NH), 1624 (CO-amide), 1581 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 2.49 (s, 3H, $CH_{3(furan)}$), 4.34 (d, 2H, CH_2, *J* 6.10 Hz), 5.31 (t, 1H, OH, *J* 6.10 Hz), 6.73 (s, 1H, $CH_{(furan)}$), 7.00 (d, 2H, CH=CH, *J* 6.85 Hz), phenyl protons: 7.29 (t, 1H, *p*-H), 7.36 (t, 2H, *o*-H), 7.59 (d, 2H, *m*-H), 8.13 (d, 1H, CH=N, *J* 6.90 Hz), 11.23 (s, 1H, NH); MS: m/z (%), 415 (0.39, M^++1), 414 (0.81, M^+), 354 (13.73), 297 (2.84), 296 (18.04), 295 (100), 235 (2.83), 209 (1.22), 193 (6.36), 192 (17.16), 175 (7.87), 151 (5.27), 147 (3.39), 137 (8.71), 121 (2.58), 115 (12.23), 104 (2.25), 77 (2.61); Anal. Calcd for $C_{16}H_{16}N_2O_3$: C, 67.57; H, 5.66; N, 9.83%; found: C, 67.59; H, 5.63; N, 9.85%.

2.1.12. Acetylation of Compounds **11a,c.**

General Methods. A solution of sugar derivative **11a,c** (6.15 mmol) in dry pyridine (20 mL) was treated with acetic anhydride (20 mL), and the mixture was kept overnight with occasional shaking at room temperature. Then it was poured onto crushed ice, the acetyl derivative that separated out, was filtered off, washed with water and dried.

[2-Methyl-3-(2-((2-phenyl-2H-1,2,3-triazol-4-yl)methylene)hydrazinecarbonyl)furan-5-yl]methyl acetate **12a**. It was obtained from **11a**, yield (94.09 %). Recrystallized from ethanol as colourless needles; R_f: 0.35 (hexane: ethylacetate, 2:1, V/V); mp 165 ºC; IR (KBr)/cm^{-1}: 3136 (NH), 1743 (OAc), 1632 (CO-amide), 1576 cm^{-1} (C=N); ^{1}H NMR ($CDCl_3$); δ: 2.10 (s, 3H, OAc), 2.69 (s, 3H, $CH_{3(furan)}$), 5.04 (s, 2H, CH_2), 6.76 (s, 1H, $CH_{(furan)}$), phenyl protons: 7.34 (m, 1H, *p*-H), 7.47 (m, 2H, *o*-H), 7.60 (m, 2H, *m*-H), 8.01 (s, 1H, CH=N), 8.10 (s, 1H, $CH_{(triazole)}$), 12.21 (s, 1H, NH); Anal. Calcd for $C_{18}H_{17}N_5O_4$: C, 58.82; H, 4.67; N, 19.07%; found: C, 58.85; H, 4.66; N, 19.06%.

Ethyl 5-[(2-(5-(acetoxymethyl)-2-methylfuran-3-carbonyl)hydrazono)methyl]-2-methyl-furan-3-carboxylate **12c.** It was obtained from **11c**, yield (88.83 %). Recrystallized from dilute ethanol as colourless needles; R_f: 0.52 (hexane: ethyl acetate, 3:1, V/V); mp 177-179 ºC; IR (KBr)/cm^{-1}: 3225 (NH), 1746 (OAc), 1707 (CO-ester), 1662 (CO-amide), 1598 cm^{-1} (C=N); ^{1}H NMR ($CDCl_3$); δ: 1.35 (t, 3H, $CH_{3(ester)}$), 2.09 (s, 3H, OAc), 2.61 (s, 3H, $CH_{3(furan-1)}$), 2.69 (s, 3H, $CH_{3(furan-2)}$), 4.31 (q, 2H, $CH_{2(ester)}$), 4.98 (s, 2H, CH_2), 6.56 (s, 1H, $CH_{(furan-1)}$), 6.65 (s, 1H, $CH_{(furan-2)}$), 8.57 (s, 1H, CH=N), 11.32 (bs, 1H, NH); ^{13}C NMR ($CDCl_3$); δ: 14.47 (C-18), 21.04 (C-17), 57.67 (C-16), 60.83 (C-15), 107.57 (C-14), 109.33 (C-13), 115.89 (C-12), 116.62 (C-11), 122.82 (C-10), 146.77 (C-9), 148.85 (C-8), 150.37 (C-7), 155.10 (C-6), 162.71 (C-5), 163.31 (C-4), 164.90 (C-3), 170.72 (C-2), 177.24 (C-1); MS: m/z (%), 378 (1.18, M^++2), 377 (6.63, M^++1), 376 (30.09, M^+), 317 (9.01), 316 (5.26), 182 (11.48), 181 (100), 155 (9.39), 149 (9.00), 139 (47.05), 138 (16.97),

137 (78.32), 123 (7.06), 122 (6.39), 121 (14.26), 97 (10.09), 94 (14.20), 85 (10.38), 83 (15.54), 81 (6.82), 79 (12.29), 73 (9.24), 71 (8.88), 69 (12.97), 60 (13.42), 57 (16.68), 55 (15.98), 52 (9.61), 51 (6.21); Anal. Calcd for $C_{18}H_{20}N_2O_7$: C, 57.43; H, 5.36; N, 7.45%; found: C, 57.44; H, 5.36; N, 7.44%.

2.1.13. [2-Methyl-3-(5-(2-phenyl-2H-1,2,3-triazol-4-yl)-1,3,4-oxadiazol-2-yl)furan-5-yl]methyl acetate **13**. It was obtained from **12a** as the same procedure for preparation of **12a,c**, yield (34.16 %). Recrystallized from ethanol as colorless needles; R_f: 0.44 (n-hexane: ethyl acetate, 5:1, V/V); mp 155-157 ºC; IR (KBr)/cm^{-1}: 1737 (OAc), 1644 cm^{-1} (C=N); ^{1}H NMR ($CDCL_3$), 2.12 (s, 3H, OAc), 2.76 (s, 3H, $CH_{3(furan)}$), 5.07 (s, 2H, CH_2), 6.92 (s, 1H, $CH_{(furan)}$), phenyl protons: 7.44 (t, 1H, *p*-H), 7.54 (t, 2H, *o*-H), 8.18 (m, 2H, *m*-H), 8.42 (s, 1H, $CH_{(triazole)}$); Anal. Calcd for $C_{18}H_{15}N_5O_4$: C, 59.19; H, 4.16; N, 19.15%; found: C, 59.18; H, 4.14; N, 19.17%.

2.1.14. [3-(3-Acetyl-2-(2-(*p*-bromophenyl)-2H-1,2,3-triazol-4-yl)-2,3-dihydro-1,3,4-oxadiazol-5-yl)-2-methylfuran-5-yl]methyl acetate **12b$_2$**. A solution of **11b** (2.0 g, 4.96 mmol) in a mixture of pyridine (40 mL), and acetic anhydride (40 mL) was kept for two weeks (due to its low solubility in the reaction mixture) at room temperature with occasional stirring. The reaction mixture was filtered off, and the filtrate was poured onto crushed ice; the acetyl derivative that separated out, was filtered off, washed with water, and dried, yield (78.61 %). Recrystallized from ethanol as colourless needles; R_f: 0.85 (hexane: ethyl acetate, 2:1, V/V); mp 129-131 ºC; IR (KBr)/cm^{-1}: 1741 (OAc), 1668 (N-Ac), 1566 cm^{-1} (C=N); ^{1}H NMR ($CDCl_3$); δ: 2.08 (s, 3H, OAc), 2.33 (s, 3H, N-Ac), 2.55 (s, 3H, $CH_{3(furan)}$), 5.00 (s, 2H, CH_2), 6.65 (s, 1H, $CH_{(furan)}$), 7.25 (s, 1H, $CH_{(b)}$), *p*-bromophenyl protons: 7.58 (d, 2H, *m*-H), 7.85 (s, 1H, $CH_{(triazole)}$), 7.94 (m, 2H, *o*-H); MS: m/z (%), 490/488 (1.83, 1.77, M^++1), 489/487 (7.44, 7.61, M^+), 447/ 445 (5.39, 5.54, M^+-CH_2=C=O), 388/386 (3.04, 3.07), 387/385 (5.44, 5.24), 182 (6.42), 181 (63.84), 180 (7.69), 157 (2.42), 155 (3.17), 139 (14.36), 137 (9.12), 121 (6.11), 94 (6.72), 79(3.03), 52 (3.08), 44 (2.66), 43 (100, $COCH_3$); Anal. Calcd for $C_{20}H_{18}BrN_5O_5$: C, 49.18; H, 3.73; N, 14.35; Br, 16.37%; found: C, 49.19; H, 3.72; N, 14.34; Br 16.36 %.

2.1.15.2-[1'-(2-Methyl-5-(1',2',3',4'-tetrahydroxybutyl)furan-3-yl)ethylidene]hydrazine-carboxamide **14**. A solution of **1a** (2.44 g, 0.01 mmol) in ethanol (10 mL) was heated under reflux with a solution of semicarbazide hydrochloride (1.11 g, 0.01 mmol) in distilled water (10 mL) and a solution of sodium acetate (2.5 g, 0.03 mmol) in distilled water (10 mL) for one hour. After cooling, the title compound that separated out, was filtered off, washed with little ethanol, and dried, yield (83.06 %). Recrystallized from ethanol-water as colourless needles; mp 202 ºC (dec.); $[\alpha]_D^{20}$ -7.43; IR (KBr)/cm^{-1}: 3490 (OH), 3390 (NH), 3271 (NH_2), 1751 (CO-amide), 1579 cm^{-1} (C=N); ^{1}H NMR (DMSO-d_6); δ: 2.03 (s, 3H, CH_3), 2.37 (s, 3H, $CH_{3(furan)}$), 3.46 (m, 4H, H-4'a, H-3', H-4'b, H-2'), 4.39 (t, 1H, 4'-OH, $J_{4'b,OH}$ 5.35 Hz, $J_{4'a,OH}$ 5.35 Hz), 4.52 (d, 1H, 3'-OH, $J_{3',OH}$ 7.65 Hz), 4.66 (m, 2H, H-1', 2'-OH), 5.00 (d, 1H, 1'-OH), 6.27 (bs, 2H, NH_2), 6.48 (s, 1H, $CH_{(furan)}$), 9.21 (s, 1H, NH); Anal. Calcd for $C_{12}H_{19}N_3O_6$: C, 47.85; H, 6.37; N, 13.96%; found: C, 47.84; H, 6.36; N, 13.95%.

2.1.16. 5-(7,8-Dihydroxytetrahydrofuran-2-yl)-2,4,5-trioxo-3-(1,2,3-selenadiazol-5-yl)-nonanal **16**. A mixture of **14** (1.5 g, 5 mmol) and selenium dioxide (0.55 g, 5 mmol) in glacial acetic acid (30 mL) was refluxed for 8 hours, the reaction mixture was filtered off twice while hot to remove the red elemental selenium, then water was added to the filterate to obtain the title compound. It was filtered off and dried, yield (27.79 %). Recrystallized from dioxane-water as a brown mass; mp > 360 ºC; $[\alpha]_D^{20}$ -46.44; IR (KBr)/cm^{-1}: 3310 (OH), 1717 cm^{-1} (C=O); ^{1}H NMR (DMSO-d_6); δ: 9.76 (m, 1H, CHO), 7.48 (m, 1H, $CH_{(selenadiazole)}$), 4.37 (m, 7H, sugar moiety), 1.22 (m, 1H, CH); Anal. Calcd for $C_{11}H_{10}N_2O_7Se$: Se, 7.78; found: Se, 7.88%.

2.1.17. 1-[5-(2',3'-Dimethoxytetrahydrofuran-1'-yl)-1,2-dimethyl-1H-pyrol-3-yl]ethanone **18**. A solution of 1-[2-methyl-5-(1',2',3',4'-tetrahydroxybutyl)-1*H*-pyrrol-3-yl]ethanone **17 (González and Sánchez 1965)** (1.0 g, 4.115 mmoL) in acetone (20 mL) was treated with sodium hydroxide (2.22 g, 54.81 mmoL), the mixture was stirred at 45 ºC. Dimethyl sulfate (3.2 mL, 25.40 mmoL) was added, after which the temperature of the mixture was raised spontaneously to the boiling temperature and was left for 3 hours. After cooling, and dilution with water, it was then extracted with chloroform. The chloroform layer was washed with water and dried over anhydrous sodium sulphate. After evaporation of the dried filtrate, a dirty green solid was obtained; yield (0.95g, 86.46%). It was recrystallized from benzene as colourless needles; Rf: 0.7 (chloroform: methanol, 100:1, V/V); mp 131-132 ºC; $[\alpha]_D^{20}$ -47.72; IR (KBr)/cm^{-1}: 1655 ($COCH_3$), 1565 cm^{-1} (C=C); ^{1}H NMR ($CDCl_3$); δ : 2.39 (s,3H, $COCH_3$), 2.53 (s, 3H, $CH_{3(furan)}$, 3.49 (s, 6H, $2OCH_3$), 3.52 (s, 3H, NCH_3), 4.04 ((m, 4H, H-2', H-3', H-4'b, H-4'a), 4.87 (d, 1H, $J_{1',2'}$ 7.65 Hz), 6.45 (s, 1H, $CH_{(furan)}$; MS: m/z(%), 267 (17.40, M^+), 252 (2.17, M^+-CH_3), 236 (0.45, M^+-OCH_3), 206 (2.94), 180 (3.66), 178 (3.89), 166 (7.36), 150 (21.45),

136 (2.94), 122 (3.11), 95 (1.24), 71 (100), 59 (11.52), 56 (7.82), 43 (26.32), $COCH_3$, 41 (13.96); Anal. Calcd for $C_{12}H_{19}N_3O_6$: C, 62.91; H, 7.90; N, 5.23%; found: C, 62.89; H, 7.92; N, 5.26%.

2.2. Pharmacological screening

2.2.1. *MAO-B activity*

2.2.1.1. *Enzyme preparation:* rabbit brain was homogenized in 9 volumes of ice-cold 0.1 M sodium phosphate buffer (pH 7.4) with an Ultra-Turrax 18/2 homogenizer. The separated mitochondrial fraction was suspended in phosphate buffer to give a final volume of 1 mL g^{-1} weight of tissue (Green and El Hait 1980).

2.2.1.2. *Enzyme assay:* monoamine oxidase-B activity was assayed in presence and absence of the examined compounds each separately using benzylamine as substrate by continuous recording on a Pye Unicam SP8-100 double Beam Spectrophotometer of the increase in extinction at 250 nm produced at 38 ºC.

2.2.1.3. *Determination of V_{max} and K_m:* the V_{max} and K_m of MAO-B catalyzed reaction in presence or absence of each examined compound was carried out by plotting the velocity of the reaction (v) against substrate concentration [S], each separately.

2.2.2. Antimicrobial and Antifungal screening

The biological activity of the synthesized compounds **6a,b,c, 7a, 8b, 10a,c, 11c,d,** and **16** have been studied for their antimicrobial and antifungal activities by using Nutrient Agar (NA) and Sabouraud Dextrose Agar (SDA) diffution method, respectively in DMSO solvent against four bacterial species (*Escherichia coli, Bacillus sp., Staphylococcus sp., and Sarcina sp.*) and six fungal species (*Aspergillus niger, Aspergillus fmigatus, Alternaria sp., Fusarium sp., Chaetomium sp., and Penicillium sp.*).The bacteria were subcultured on Nutrient Agar medium (NA), whereas, fungi were subcultured on Sabouraud Dextrose Agar (SDA). The composition is given in g L^{-1} unless otherwise stated. The pH value of the media was adjusted to 7 ± 0.1 prior to sterilization with 0.1 M sodium hydroxide or hydrochloric acid. All media were prepared with distilled water and sterilized by autoclaving at 121 ºC for 20 min. *Nutrient Agar (NA):* Peptone, 5; beef extract, 3; NaCl, 5; agar, 20. *Sabouraud Dextrose Agar (SDA):* Peptone, 10; glucose, 40; agar, 20.

2.2.2.1. Antimicrobial and Antifungal assay:

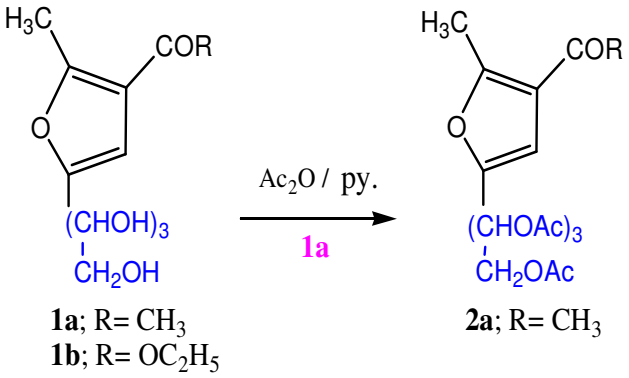
The stock solution (1 mg ml^{-1}) of the test chemicals were prepared by dissolving 10 mg of the test compound in 10 mL dimethylsulphoxide (DMSO) solvent. Petri plates (150 mm × 15mm) were prepared by pouring 60 mL of SDA and allowed to solidify. Plates were dried and 1 mL of each standardized inoculums suspension was poured and uniformly spread. The excess inoculums was drained and the inoculums was allowed to dry for 15 min. Eight equidistant wells were made in the medium using a sterile cork borer (6 mm in diameter and 50 µL of the test chemicals (1 mg mL^{-1}) diluted in DMSO 2% were placed into the wells. The Petri-dishes containing bacteria and fungi species were incubated at 37 ºC for 24h, and 48h, respectively. The tests were carried in triplicate. The antimicrobial activity was measured as the diameter (mm) of clear zone of growth inhibition.

3. RESULTS AND DISCUSSION

3.1. Chemistry

Study of the ^{1}H NMR spectrum ($CDCl_3$, 500 MHz) of the *O*-acetylated furan derivative **2a (González and Sánchez 1965)** for the sugar region showed, the H-1' proton signal as a doublet at δ 5.94 ($J_{1',2'}$ 4.60 Hz), followed by a doublet of doublet at 5.51 ($J_{1',2'}$ 4.60 Hz and $J_{2',3'}$ 7.65 Hz) for H-2', a multiplet at 5.08 for H-3', and two doublet of doublets at 4.16 and 4.05 ppm corresponding to the geminal protons; H-4'b and H-4'a, respectively having coupling constants $J_{3',4'b}$, $J_{3',4'a}$, and $J_{4'b,4'a}$ 3.05, 5.35, and 12.25 Hz, respectively. The observed value of the coupling constant $J_{1',2'}$ (4.60 Hz), is, however, intermediate between that expected for antiparallel and gauche arrangements of H-1' and H-2'. This value indicates a major contribution from the conformer having H-1' and H-2' in gauche arrangement in the planar zigzag conformation **I**. The rotamer having antiparallel arrangement between H-1' and H-2' would be destabilized by 1,3-inreraction between C-3' and the aryl residue. The coupling constant $J_{2',3'}$ 7.65 Hz, indicates a high population of the rotamer having an antiparallel arrangement between H-2' and H-3'. The fact that $J_{3',4'b}$ and $J_{3',4'a}$ values are different (3.05 Hz and 5.35 Hz, respectively), indicating that one proton at C-4' is gauche to H-3' and that the other one although not exclusively antiparallel, it is the predominant conformer. This is compatible with the conformation **I** or with the rotamer **II** having antiparallel acetoxyl groups at C-3' and C-4'. The latter, would however, have parallel interaction between the acetoxyl groups at C-2' and C-4' and is presumably less favored, see scheme 1 and figure 1.

Acetylation of **3** (El Sadek and Zagzoug, 1991), afforded the di-N-acetyl-tetra-*O*-acetyl derivative **4**, which upon deacetylation with methanolic ammonia, afforded **5** in 98 % yield, see experimental part and Scheme 2.



1a; R= CH_3
1b; R= OC_2H_5

2a; R= CH_3

Scheme 1

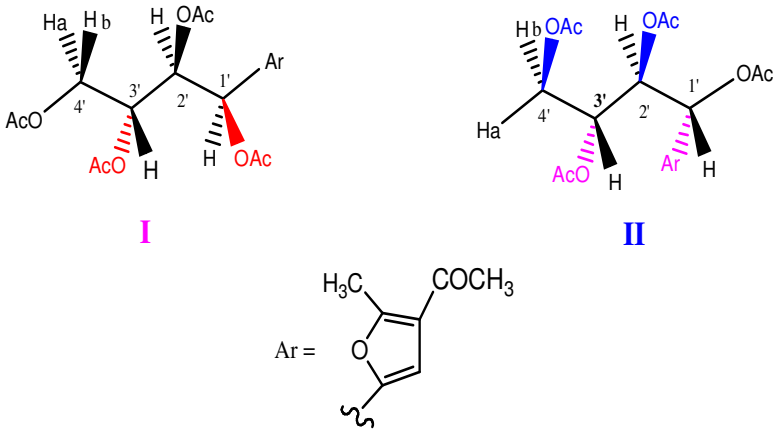
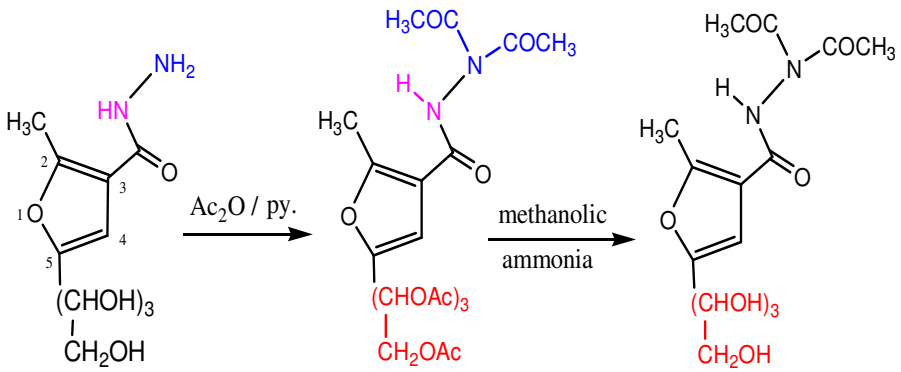


Figure 1. Steriochemistry of compound 2a



Scheme 2

Condensation of **3** with a number of aldehydes afforded the corresponding carbohydrazone derivatives **6a-c**, in yields of 84, 92, and 90 %, respectively. Acetylation of the carbohydrazone derivatives **6a-c** afforded the corresponding *O*-acetyl derivatives **7a-c**, respectively. Oxidative cyclization of the compounds **7a,b**, afforded the corresponding 1,3,4-oxadiazole derivatives **8a,b** in yields 51 and 84 %, respectively. The assignment of the signals for the sugar protons in ^{1}H NMR spectra of these compounds **8a,b** was based on the 2D ^{1}H NMR spectrum of compound **8a**. In addition, deacetylation of compounds **8a,b** afforded the corresponding de-*O*-acetylated derivatives **9a,b** in 97 % yield, see experimental part, scheme 3 and figure 2.

Furthermore, periodate oxidation of compouns **6a-c** and **6d** (El Sadek and Zagzoug 1991), afforded the corresponding formyl derivatives **10a-c** and **10d** (El Sadek and Zagzoug 1991), respectively. Reduction of compounds **10a-d** with sodium borohydride, afforded the corresponding hydroxymethyl derivatives **11a-d**. Acetylation of compounds **11a**,**c** afforded the corresponding *O*-acetyl derivatives; **12a,c** respectively. Oxidative cyclization of **12a** afforded the corresponding 1,3,4-oxadiazole derivative **13**. On the other hand, the prolonged acetylation of compound **11b**, resulted in the N-acetyl-*O*-acetyl derivative, that may has the open structure **12b$_1$** or N-acetyl-2,3-dihydro-1,3,4-oxadiazol **12b$_2$**. However the spectral data confirmed the cyclized structure **12b$_2$** where, the proton H_b was shown at upper field δ 7.254 ppm than that of H_a (8-8.2) ppm as appeared in the previous prepared carbohydrazones, see experimental part and scheme 4.

Efforts to synthesize acyclic *C*-glycosyl selenadiazole derivative **15** by treatment the semicarbazone derivative **14** in glacial acetic acid with selenium dioxide under reflux have been unsuccessful. Instead, 5-(7,8-dihydroxytetrahydrofuran-2-yl)-2,4,5-trioxo-3-(1,2,3-selenadiazol-5-yl)nonanal **16** has been obtained. The structure of **16** was deduced from the respective spectral data as well as the action of acetic acid and SeO_2. IR spectrum of compound **16** showed the disappearance of characteristic absorption bands of NH and NH_2 groups, while hydroxyl groups of the sugar moiety appeared a at 3310 cm^{-1}. The carbonyl groups appeared at 1717 cm^{-1} instead of 1751 cm^{-1} in compound **14**; these shift of carbonyl absorption band ensured the disappearance of amide group and formation of new carbonyl groups. In addition ^{1}H NMR spectrum of this product, showed the disappearance of the signals at δ 6.5 and 2.5 ppm due to the furyl proton and the methyl group protons at position-4 and -2, respectively in the furan ring of compound **14**. Instead it showed signals at δ 9.763 for the aldehyde group proton, 7.483 for the selenadiazole ring proton, and 1.223 ppm corresponding to the methine proton. The sugar moiety was appeared as a multiplet at δ 4.369 ppm. In the light of these arguments, it can be deduced that the reaction of the acyclo *C*-glycosyl semicarbazone derivative **14** in glacial acetic acid with selenium dioxide might be proceed as a result of action of acidic medium in both furan ring opening and anhydro formation (Sánchez and Roldán 1972). As well as action of SeO_2 in both oxidation of methyl or methylene group adjacent to the carbonyl group (Finar, 1973), and cyclization of semicarbazone part into the selenadiazole ring (Jalilian, 2003), see scheme 5 below.

The three possible tautomers of the compound **16** were optimized to obtain the most stable conformers of each using molecular dynamics Conformational search and Amber force field (Gabedit graphical software version 2.4.0) (Allouche, 2011). Then the resulted conformers were geometry optimized using DFT/B3LYP with cc-pvdz basis set. (Orca software version 2.8-) (An Ab Initio, DFT and Semiempirical electronic structure package An Ab Initio, DFT and Semiempirical electronic structure package), where it showed that there are three tautomers of which the first one is the most stable one (energy= -3462.08624 a.u.), see figure 3 below.

Methylation the acyclic *C*-nucleoside **17** in acetone with dimethyl-sulfate in presence of sodium hydroxide resulted in the 1',4'-anhydro-*N*-methyl-di-*O*-methyl derivative **18** in 86% yield with retention of configuration due to the initial formation of the kinetically formed 4'-monomethyl derivative which undergoes S_N2 attack from the back by favorably disposed 2'-hydroxyl group (El Sallam and El Shemany 1994). Its infrared spectrum, showed the acetyl carbonyl absorption band at 1655 cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$), showed the furyl proton at position-4 as the most downfield signal at δ 6.450. At the sugar region, the H-1' was shown as a doublet at δ 4.870 ($J_{1',2'}$ 7.65 Hz), followed by the rest of the sugar protons (H-2', H-3', H-4'a, H-4'b) as a multiplet at δ 4.035 ppm. The *N*-methyl protons were shown as a singlet at δ 3.519 followed by three singlets at δ 3.488, 2.528, and 2.388 ppm, corresponding to the protons of the di-*O*-methyl groups, methyl at position-2 in the furan ring, and the acetyl protons, respectively. The mass spectrum showed the molecular ion peak at m/z 267 in accord with the molecular formula $C_{14}H_{21}O_4N$. See experimental part and Scheme 6 below.

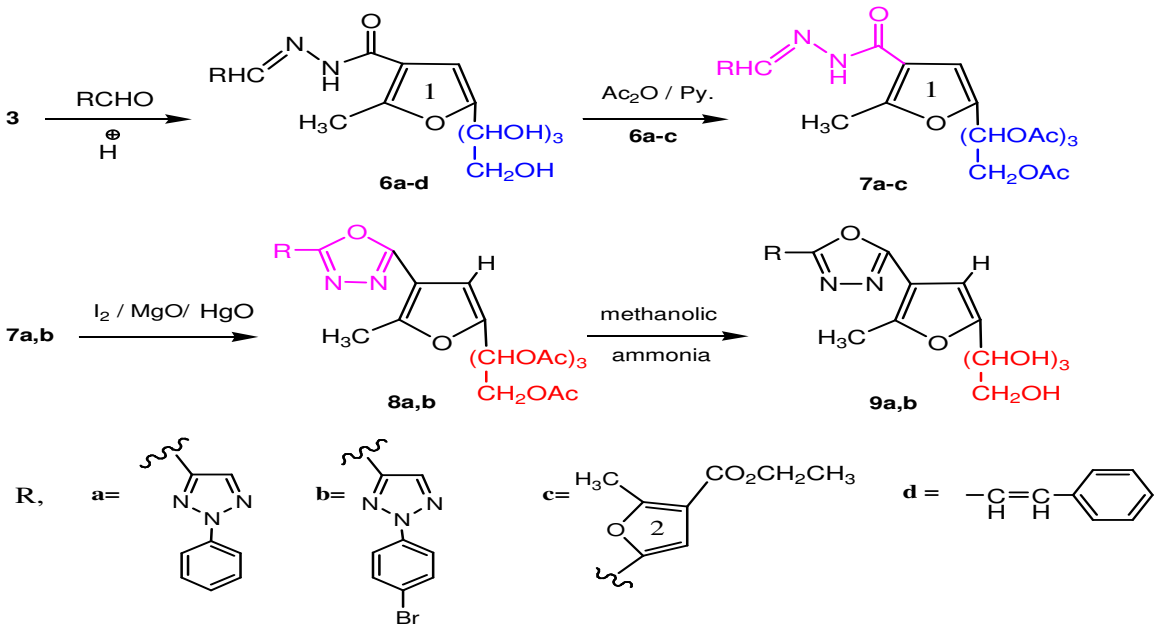
3.2. Pharmacological screening

3.2.1. MAO-B Activity

3.2.1.1. Effect of Tested Compounds on MAO-B

This study aimed to evaluate the effect of selected newly prepared compounds **6a** and **16** on MAO-B activity.

3.2.1.2. Determination of V_{max} and K_m



Scheme 3

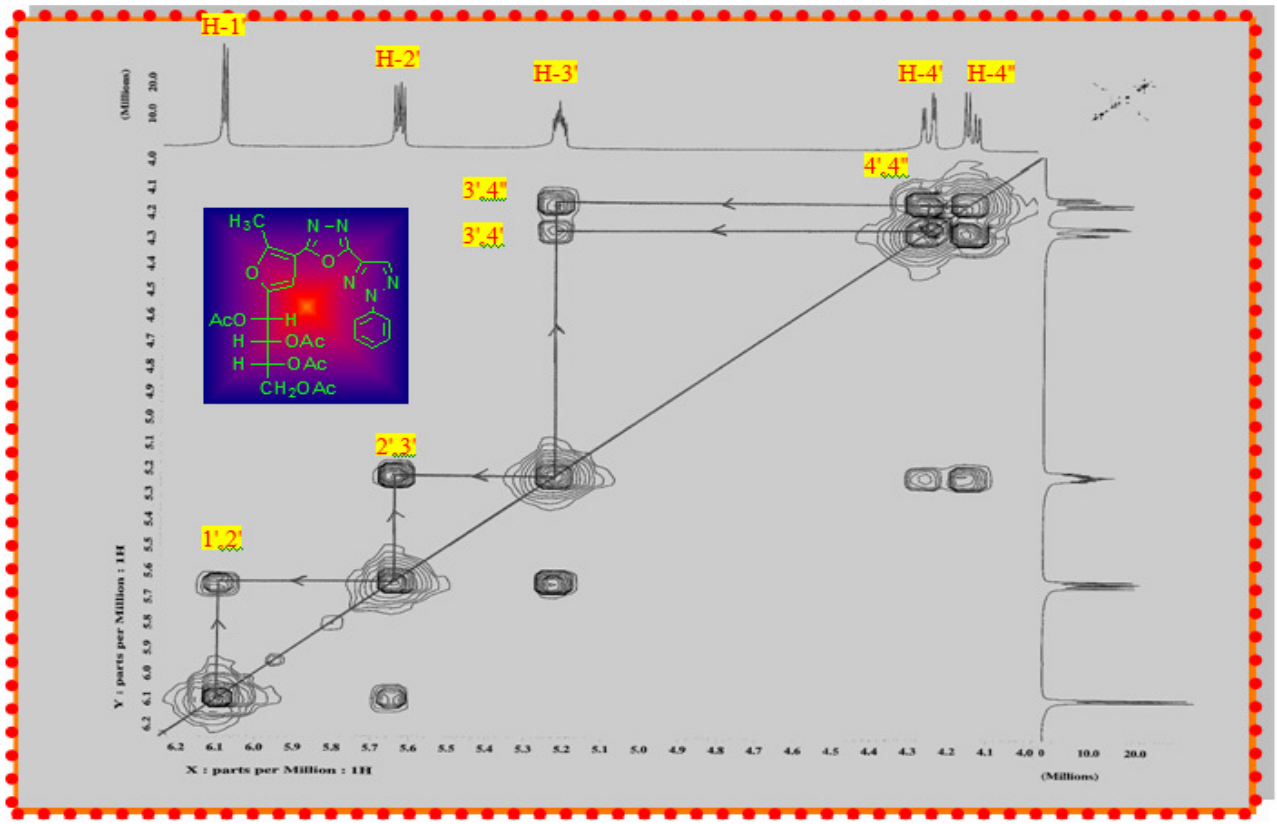
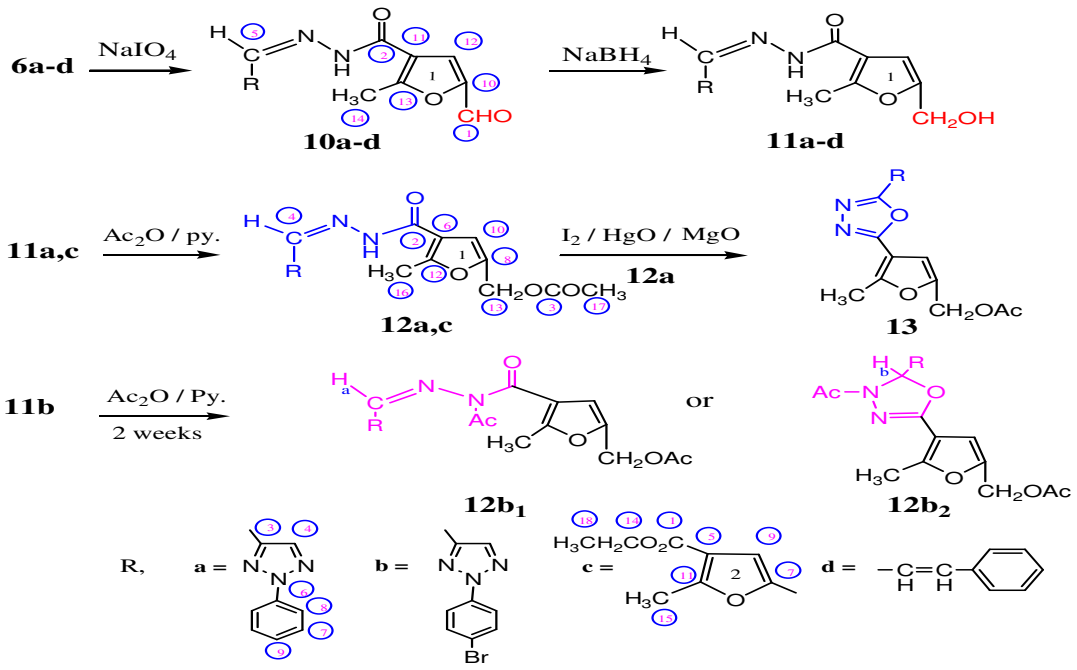
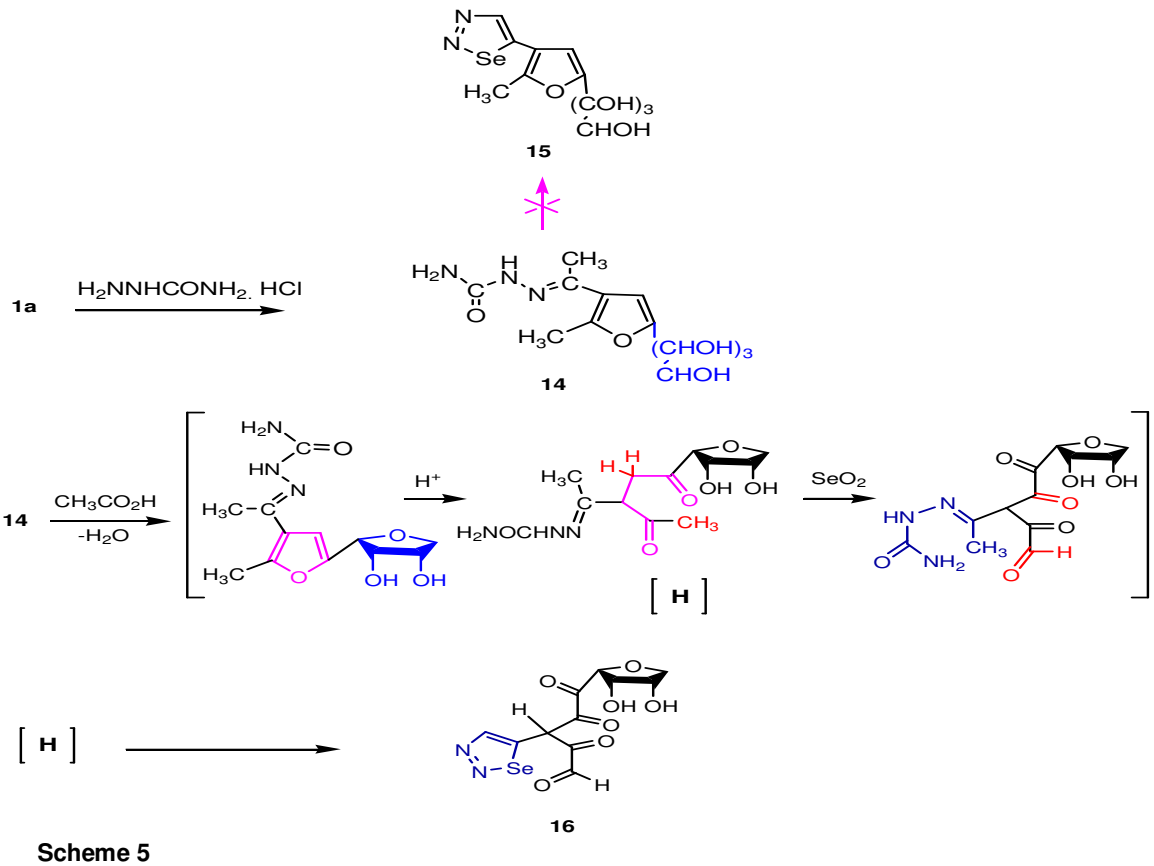


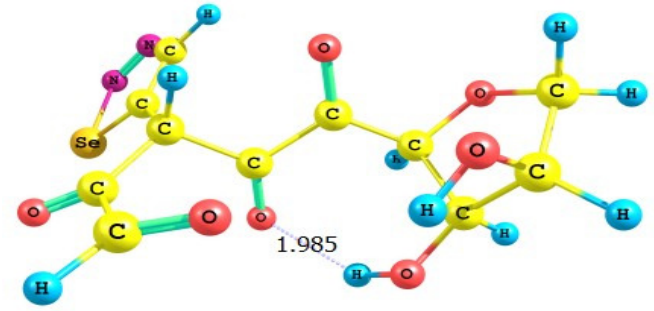
Figure 2. 2D H^1NMR of compound 8



Scheme 4



Scheme 5



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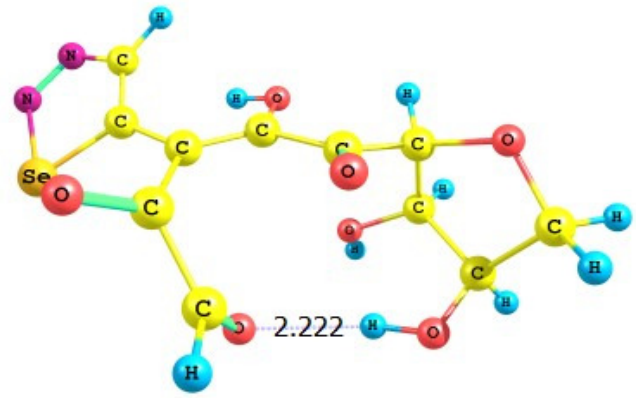
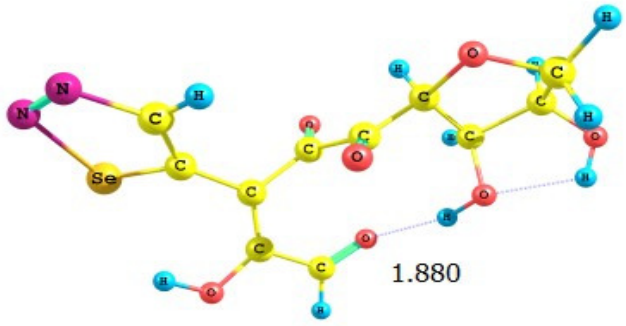
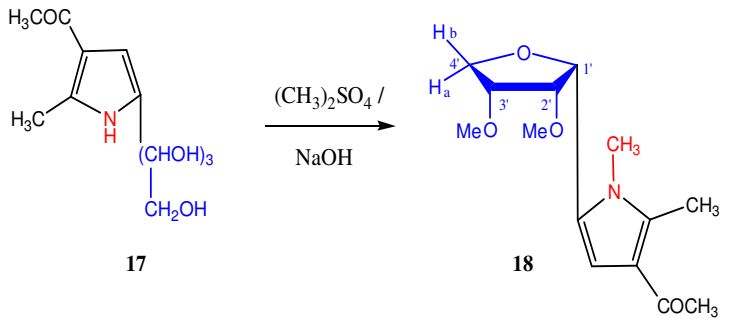


E= -3462.07466 a.u.



E= -3462.082034 a.u.

Figure 3. Possible tautomers of the compound 16



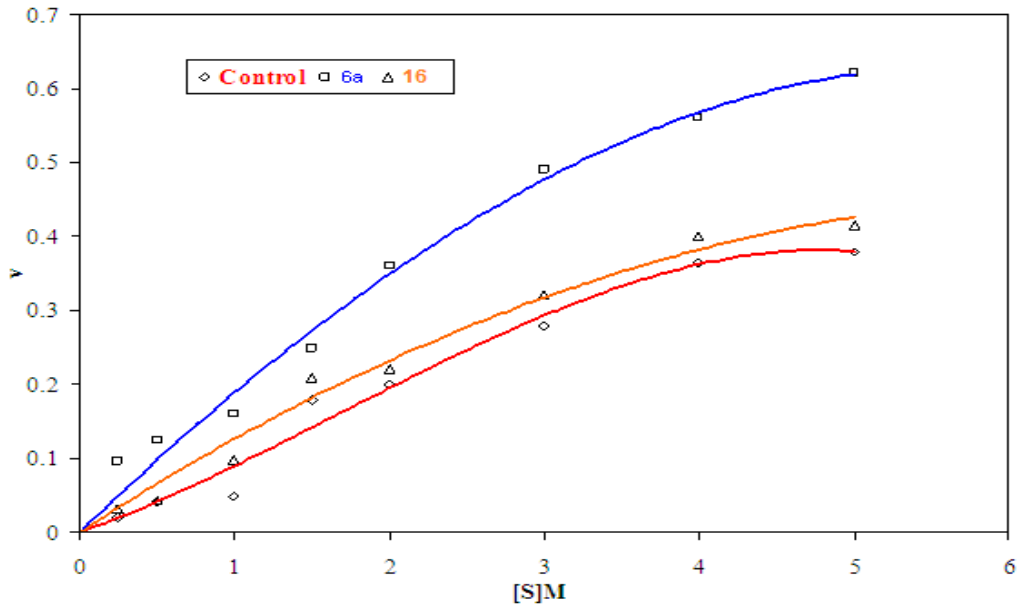
17 **18**

Scheme 6

Table 1. Effect of substrate concentration on the velocity of MAO-catalyzed reaction in presence and absence of two examined compounds **6a** and **16** compared to control.

Substrate conc.	velocity		
	Control	6a	16
0.25×10^{-3}	0.020	0.096	0.033
0.50×10^{-3}	0.042	0.124	0.044
1.00×10^{-3}	0.050	0.160	0.099
1.50×10^{-3}	0.180	0.250	0.210
2.00×10^{-3}	0.200	0.360	0.220
3.00×10^{-3}	0.280	0.490	0.320
4.00×10^{-3}	0.365	0.560	0.400
5.00×10^{-3}	0.380	0.620	0.415

Figure 4. Effect of substrate concentration on the velocity of MAO-B catalyzed reaction in presence and absence of the examined compounds **6a** and **18** compared to control.



The maximum velociy (V_{max}) and K_m of MAO-B catalyzed reaction were determined in presence and absence of each examined compound by plotting v against [S], each separately. Whereby, the V_{max} values equal to 0.61, and 0.41 for compounds **6a** and **16**, respectively. Meanwhile their K_m values were 1.51 and 1.81 respectively. The obtained results revealed that MAO-B was activated in presence of compounds **6a** and **16**, each separately by 6.93, and 2.93 fold, respectively. Our obtained data showed that compound **6a** was an effective MAO-B activator, which increases the affinity of substrate to bind with the active site of MAO-B enzyme. That may be attributed to the different substituents at the furan ring of the prepared compounds, Table 1, Figure 4.

Table 2. Inhibition zone of tested compounds against selected microorganisms (mm)

Compound No.	Bacteria				Fungi					
	Gram positive bacteria			Gram negative bacteria						
	Bacillus sp.	Sarcina sp.	Staphylococcus sp.	E. coli	Aspergillus niger	Aspergillus fumigatus	Alternaria sp.	Fusarium sp.	Chaetomium sp.	Penicillium Sp.
6a	….	1	1	….	1	….	1	2	….	1
6b	….	….	….	….	1	….	….	….	….	0.5
6c	….	….	1	….	0.5	….	0.5	1	….	1
7a	….	….	….	….	1	….	….	….	….	2
8b	….	….	1	….	….	….	….	….	….	2
10a	….	….	….	….	….	1	….	….	….	….
10c	….	….	1	….	….	….	….	1	….	1
11c	….	….	0.5	….	….	….	….	1	….	1
11d	….	….	0.5	….	….	….	….	….	….	….
16	….	4	2	….	1	1	….	1	2	….

…. No effect

3.2.2. Antimicrobial and Antifungal activities:

The compounds **6a,b,c, 7a, 8b, 10a,c, 11c,d, and 16** have been studied for their antimicrobial and antifungal activities by using Nutrient Agar (NA) and Sabouraud Dextrose Agar (SDA) diffution method, respectively in DMSO solvent against four bacterial species (Escherichia coli, Bacillus sp., Staphylococcus sp., and Sarcina sp.) and six fungal species (Aspergillus niger, Aspergillus fmigatus, Alternaria sp., Fusarium sp., Chaetomium sp., and Penicillium sp.), Table 2.

It was found that the selenadiazole derivative **16**, the most effective against Gram positive bacteria (Sarcina sp.) and (Staphylococcus sp.). Whereas, for antifungal activity, the compound **6a** achieved higher antifungal activity against (Aspergillus niger, Alternaria sp., and Fusarium sp.) as compared with compound **6c**. This may be attributed to the strong electron withdrawing effect and higher stability of the triazole ring in compound **6a**. In contrast to the steric hinderance caused by furan ring in compound **6c**.

4. CONCLUSION

In summary, Some new *C*-glycoside derivatives and 1,2,3-selenadiazole derivative have been prepared as well as their physical and biological properties on bacteria, fungi, and MAO-B were studied. Their activities depend on their chemical structures.

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