The Reaction of (*N*-Isocyanimino)triphenylphosphorane with Biacetyl in the Presence of Aromatic Carboxylic Acids: Efficient One-Pot Three-Component Reaction for the Synthesis of 3-(5-Aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-one Derivatives

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Reactions of biacetyl (= butane-2,3-dione) with (*N*-isocyanimino)triphenylphosphorane in the presence of aromatic carboxylic acids proceed smoothly at room temperature and under neutral conditions to afford 3-(5-aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-one derivatives in high yields.

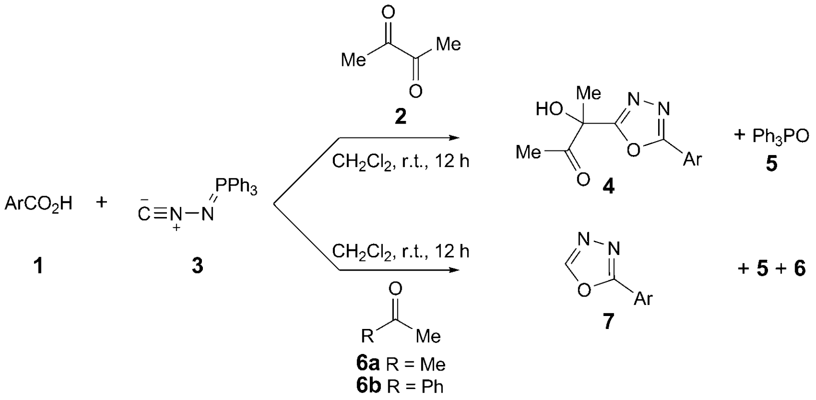
Introduction. – Due to the atom economy, convergent character, and simplicity of one-pot procedures, multi-component condensation reactions (MCRs) have an advantageous position among other reactions. The development of novel MCRs is receiving growing interest from industrial-chemistry research groups and represents a challenge for organic chemists [1] [2]. The drive toward the ideal synthesis embracing step count, ideally just one, and yield, ideally 100%, has been pursued intensively since scientists began to construct molecules. Of course, there are many other factors that affect these two aspects of synthesis, including cost, starting material availability, safety, environmental concerns, and overall ease of the process, including workup and purification [3]. The nature of the synthesis project also plays a role. Complex-molecule total synthesis is often driven by step count, while showcasing innovative chemistry. Traditional structure–activity relationship (SAR) evaluations in medicinal chemistry typically involve the preparation of an advanced intermediate that can be analogued readily to introduce the molecular diversity necessary to prepare a collection, or library, of structurally related compounds. One strategy that potentially meets the goals of total synthesis and library production is multicomponent reaction (MCR) chemistry, in which three or more starting materials are brought together in a highly convergent approach to rapidly build up molecular structure and complexity [4].

1,3,4-Oxadiazoles have attracted interest in medicinal chemistry as surrogates of carboxylic acids, esters, and carboxamides. They are an important class of heterocyclic compounds that have a wide range of pharmaceutical and biological activities including antimicrobial, antifungal, anti-inflammatory, and antihypertensive properties [5–9].

Several methods have been reported in the literature for the synthesis of 1,3,4-oxadiazoles. These protocols are multistep in nature [10–16]. The most general method involves the cyclization of diacylhydrazides with a variety of reagents, such as $SOCl_2$, $POCl_3$, or H_2SO_4, usually under harsh reaction conditions. Few reliable and operationally simple examples have been reported for the one-pot synthesis of 1,3,4-oxadiazoles, especially from readily available carboxylic acids and acid hydrazides [17–21].

In recent years, several synthetic methods have been reported for the preparation of (*N*-isocyanimino)triphenylphosphorane (CNNPPh$_3$; **3**) [22][23]. There are several reports on the use of **3** in the synthesis of metal complexes [22][23]. However, application of **3** in the synthesis of organic compounds is rather rare [24–32]. As part of our ongoing program to develop efficient and robust methods for the preparation of heterocyclic compounds [25–35], we sought to develop a convenient preparation of 3-(5-aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-ones **4a**–**4l**. Here, we report a one-pot three-component reaction, which, starting from readily available biacetyl (=butane-2,3-dione; **2**), affords **4a**–**4l** (*Scheme 1*). In the reaction, **2** is more reactive than aldehyde derivatives (see [24]), and, therefore, the final products **4** (*Scheme 1*) were formed under fairly mild and inexpensive reaction conditions (see [24] and *Exper. Part*).

Scheme 1. *Three-Component Synthesis of 3-(5-Aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-one Derivatives* **4** (see the *Table*). Electron-rich ketones such as acetone **6a** and acetophenone **6b** are not suitable starting materials in the three-component synthesis of **4**. We also used benzil ((PhCO)$_2$) instead of biacetyl (**2**) in this reaction, but no products corresponding to **4** were observed.



Results and Discussion. – The carboxylic acid derivative **1** with biacetyl (**2**) and (*N*-isocyanimino)triphenylphosphorane (**3**) in CH$_2$Cl$_2$ react in a 1:1:1 ratio at room temperature to produce 3-(5-aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-ones **4** and Ph$_3$PO (**5**; *Scheme 1* and *Table*). The reaction proceeds smoothly and cleanly under mild conditions, and no side reactions were observed. We also used acetone (**6a**) and acetophenone (**6b**) instead of biacetyl (**2**) in this reaction, but no corresponding products of type **4** were observed. In both cases, 2-aryl-1,3,4-oxadiazoles **7** and Ph$_3$P=O (**5**) were formed, as already reported in [26], and **6a** and **6b** were recovered unchanged (*Scheme 1*). As indicated in the *Table*, the reactions proceeded efficiently with the electron-poor ketone **2**, but electron-rich ketones such as **6a** and **6b** are not suitable starting materials in this reaction. We also used 1,2-diphenylethane-1,2-dione (benzil,

$(PhCO)_2$) instead of biacetyl (**2**) in this reaction, but no corresponding products **4** were observed. In this case, **7** and **5** were observed as the sole products (see [26]), and 1,2-diphenylethane-1,2-dione was recovered. The high steric hindrance in the case of benzil relative to biacetyl (**2**) may be responsible for the reduction of the reactivity.

Table. *Synthesis of 3-(5-Aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-one Derivatives* **4** (see *Scheme 1*)

4	Ar	Yield[a]) [%]	**4**	Ar	Yield[a]) [%]
a	4-Cl–C_6H_4	89	**g**	Ph	85
b	4-Br–C_6H_4	90	**h**	Thiophen-2-yl	82
c	3-Br–C_6H_4	92	**i**	4-Me–C_6H_4	85
d	2-Br–C_6H_4	80	**j**	3-Me–C_6H_4	88
e	4-F–C_6H_4	93	**k**	Naphthalen-2-yl	86
f	3-F–C_6H_4	90	**l**	Furan-2-yl	80

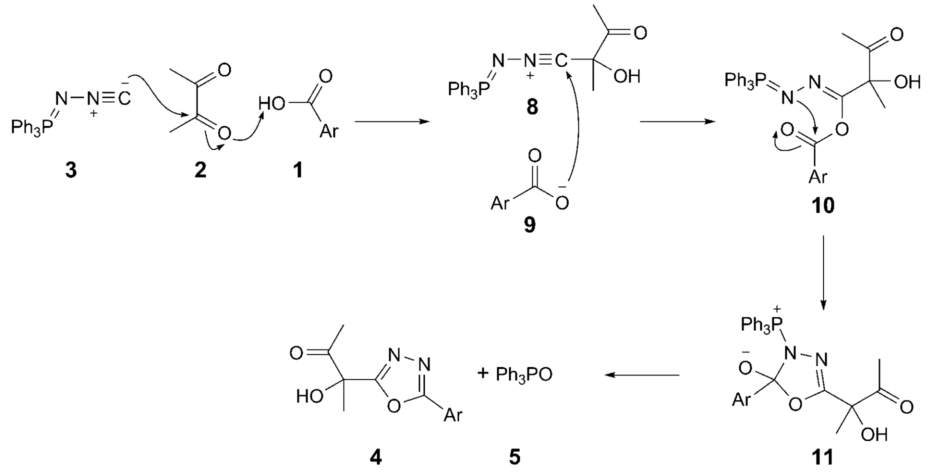
[a]) Yield of isolated products.

The structures of the products were deduced from their IR, MS, and ^{1}H- and ^{13}C-NMR data. The mass spectra of these compounds displayed molecular-ion peaks at the appropriate *m/z* values. The ^{1}H-NMR spectrum of **4a** consisted of a *singlet* for Me (δ(H) 1.95), a *singlet* for MeCO (δ(H) 2.37), a *singlet* for OH (δ(H) 4.69), exchangeable by D_2O, and a *AA′BB′* for the aromatic H-atoms (δ(H) 7.49 and 7.99, $^3J = 8.5$). The ^{1}H-decoupled ^{13}C-NMR spectrum of **4a** showed ten distinct resonances, partial assignment of these resonances is given in the *Exper. Part*. The ^{1}H- and ^{13}C-NMR spectra of compounds **4b** – **4l** were similar to those of **4a**, except for the aromatic moiety, which exhibited characteristic signals with appropriate chemical shifts.

A mechanistic rationalization for this reaction is depicted in *Scheme 2*. On the basis of the chemistry of isocyanides [4], it is reasonable to assume that the first step involves nucleophilic addition of **3** to biacetyl (**2**), facilitated by its protonation with the acid **1**, leading to nitrilium intermediate **8**. This intermediate may be attacked by the conjugate base of the acid **9** to form the 1:1:1 adduct **10**. This adduct may undergo an intramolecular aza-*Wittig* [36] reaction of the iminophosphorane moiety with the ester C=O group to afford the isolated 3-(5-aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-ones **4** by removal of $Ph_3P{=}O$ (**5**) from intermediate **11**. In this reaction, the first two reaction steps are analogous to the well-known *Passerini* reaction [4], and the final step is analogous to the well-known intramolecular aza-*Wittig* reaction [36] (tandem *Passerini*/intramolecular aza-*Wittig* sequence).

Conclusions. – The reported method offers a mild, simple, and efficient route for the preparation of 3-(5-aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-one derivatives *via* a tandem *Passerini*/intramolecular aza-*Wittig* reaction. The ease of workup, high yields, and fairly mild reaction conditions render this reaction as a useful addition to modern synthetic methodologies [24]. In the reaction, biacetyl (**2**) is more reactive than aldehyde derivatives (see [24]), and, therefore, the final products **4** (*Scheme 1*) were formed under fairly mild and inexpensive reaction conditions (see [24] and *Exper. Part*). Other aspects of this process are under investigation.

Scheme 2. *Proposed Mechanism for the Formation of 3-(5-Aryl-1,3,4-oxadiazol-2-yl)-3-hydroxybutan-2-one Derivatives* **4** via *Tandem* Passerini/*Intramolecular Aza*-Wittig *Sequence*



Experimental Part

General. Starting materials and solvents were obtained from *Merck* (Germany) and *Fluka* (Switzerland) and were used without further purification. The methods used to follow the reactions were TLC and NMR, which indicated that there was no side product. Flash column chromatography (CC): *Merck* silica-gel powder (SiO_2). M.p.: *Electrothermal 9100* apparatus; uncorrected. IR Spectra: *Jasco 6300 FTIR* spectrometer. ^{1}H- and ^{13}C-NMR spectra: in $CDCl_3$; *Bruker DRX-250 AVANCE* spectrometer at 250.0 and 62.5 MHz, resp. EI-MS: *Finnigan-MAT 8430* mass spectrometer operating at an ionization potential of 20 eV. Elemental analyses: *Heraeus CHN-O-Rapid* analyzer.

General Procedure. To a magnetically stirred soln. of (N-*isocyanimino)triphenylphosphorane* (**3**; 1 mmol) and *biacetyl* (= *butane-2,3-dione*; **2**, 1 mmol) in CH_2Cl_2 (7 ml) was added dropwise of a soln. of *4-chlorobenzoic acid* (**1a**; 1 mmol) in CH_2Cl_2 (5 ml) at r.t. within 15 min. The mixture was stirred for 12 h. The solvent was removed under reduced pressure, and the viscous residue was purified by CC (SiO_2; petroleum ether/AcOEt 8 : 1). The solvent was removed under reduced pressure to yield the crystalline products.

3-[5-(4-Chlorophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxybutan-2-one (**4a**). White crystals. M.p. 111.8 – 113.3°. Yield: 237 mg (89%). IR (KBr): 3427 (OH), 2929, 2857, 2382, 2346, 1729, 1607, 1486, 1282, 1151, 838. ^{1}H-NMR: 1.95 (*s*, Me); 2.37 (*s*, MeCO); 4.69 (*s*, OH, exchanged by D_2O addition); 7.49, 7.99 (*AA'BB'*, *J* = 8.5, 8.5, 4 arom. H). ^{13}C-NMR: 23.30 (Me); 23.72 (MeCO); 75.75 (C–OH); 121.75 (arom. C); 128.38, 129.50 (4 arom. CH); 138.50 (C(4) of Ar); 164.98, 165.54 (2 C of oxadiazole); 204.44 (C=O). EI-MS: 267 (12, M^+), 226 (30), 223 (100), 180 (39), 167 (13), 149 (22), 137 (36), 75 (8), 57 (5). Anal. calc. for $C_{12}H_{11}ClN_2O_3$ (266.60): C 54.05, H 4.16, N 10.50; found: C 54.00, H 4.10, N 10.45.

3-[5-(4-Bromophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxybutan-2-one (**4b**). White crystals. M.p. 117.9 – 120.1°. Yield: 280 mg (90%). IR (KBr): 3433 (OH), 2926, 2857, 2361, 2343, 1727, 1603, 1405, 1274, 1152, 835. ^{1}H-NMR: 1.95 (*s*, Me); 2.37 (*s*, MeCO); 4.68 (*s*, OH, exchanged by D_2O addition); 7.65, 7.92 (*AA'BB'*, *J* = 8.5, 8.3, 4 arom. H). ^{13}C-NMR: 23.31 (Me); 23.70 (MeCO); 75.74 (C–OH); 122.20 (C(4) of Ar); 126.95 (arom. C); 128.49, 132.46 (4 arom. CH); 165.07, 165.60 (2 C of oxadiazole); 204.40 (C=O). EI-MS: 311 (5, M^+), 267 (100), 224 (30), 182 (67), 149 (15), 102 (9), 75 (10), 57 (5). Anal. calc. for $C_{12}H_{11}BrN_2O_3$ (311.11): C 46.32, H 3.56, N 9.00; found: C 46.25, H 3.50, N 8.93.

3-[5-(3-Bromophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxybutan-2-one (**4c**). White crystals. M.p. 102.3 – 104.6°. Yield: 286 mg (92%). IR (KBr): 3355 (OH), 2957, 2848, 2359, 2343, 1726, 1552, 1466, 1221, 1157, 805. ^{1}H-NMR: 1.95 (*s*, Me); 2.37 (*s*, MeCO); 4.71 (*s*, OH, exchanged by D_2O addition); 7.39 (*t*, *J* = 7.7, H–C(5) of Ar); 7.68, 7.99 (2*d*, *J* = 7.7, 8.0, H–C(4), H–C(6) of Ar); 8.19 (*s*, H–C(2) of Ar). ^{13}C-NMR: 23.30 (Me); 23.72 (MeCO); 75.76 (C–OH); 123.12 (C(3) of Ar); 125.10 (arom. C); 125.64, 129.93, 130.66, 135.12 (4 arom. CH); 164.46, 165.76 (2 C of oxadiazole); 204.37 (C=O). EI-MS: 311 (12, M^+), 268 (100), 227 (22), 183 (22), 155 (13), 90 (6), 75 (10). Anal. calc. for $C_{12}H_{11}BrN_2O_3$ (311.11): C 46.32, H 3.56, N 9.00; found: C 46.20, H 3.54, N 8.93.

3-[5-(2-Bromophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxybutan-2-one (**4d**). Colorless oil. Yield: 249 mg (80%). IR (neat): 3419 (OH), 2937, 2855, 2346, 2344, 1729, 1569, 1457, 1270, 1140, 768. ^{1}H-NMR: 1.95 (*s*, Me); 2.37 (*s*, MeCO); 4.65 (*s*, OH, exchanged by D_2O addition); 7.25 – 7.93 (*m*, 4 arom. H). ^{13}C-NMR: 23.14 (Me); 23.78 (MeCO); 75.69 (C–OH); 121.78 (C(2) of Ar); 127.23 (arom. C); 127.63, 131.78, 132.84, 134.51 (4 arom. CH); 164.71, 165.94 (2 C of oxadiazole); 204.29 (C=O). EI-MS: 311 (3, M^+), 268 (33), 208 (67), 196 (50), 167 (30), 149 (60), 95 (20), 75 (15), 57 (23). Anal. calc. for $C_{12}H_{11}BrN_2O_3$ (311.11): C 46.32, H 3.56, N 9.00; found: C 46.27, H 3.47, N 8.95.

3-[5-(4-Fluorophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxybutan-2-one (**4e**). White crystals. M.p. 93.2 – 95.0°. Yield: 233 mg (93%). IR (KBr): 3327 (OH), 2962, 2862, 2371, 2345, 1729, 1611, 1501, 1465, 1290. 1148, 850. ^{1}H-NMR: 1.95 (*s*, Me); 2.37 (*s*, MeCO); 4.68 (*s*, OH, exchanged by D_2O addition); 7.16 – 7.26 (*m*, 2 arom. H); 8.03 – 8.09 (*m*, 2 arom. H). ^{13}C-NMR: 23.27 (Me); 23.71 (MeCO); 75.73 (C–OH); 116.47 (*d*, ^2J(C,F) = 22.0, arom. CH); 119.62 (*d*, ^4J(C,F) = 3.7, arom. CH); 129.45 (*d*, ^3J(C,F) = 9.4, arom. CH); 165.00 (*d*, ^1J(C,F) = 249.9, arom. C); 164.97, 165.47 (2 C of oxadiazole); 204.51 (C=O). EI-MS; 251 (3, M^+), 208 (7), 167 (33), 148 (100), 104 (9), 91 (6), 70 (17), 57 (40). Anal. calc. for $C_{12}H_{11}FN_2O_3$ (250.23): C 57.60, H 4.43, N 11.20; found: C 57.57, H 4.40, N 11.18.

3-[5-(3-Fluorophenyl)-1,3,4-oxadiazol-2-yl]-3-hydroxybutan-2-one (**4f**). White crystals. M.p. 81.2 – 83.1°. Yield: 225 mg (90%). IR (KBr): 3422 (OH), 2927, 2857, 2359, 2343, 1725, 1596, 1470, 1268, 1121, 869. ^{1}H-NMR: 1.95 (*s*, Me); 2.37 (*s*, MeCO); 4.71 (*s*, OH, exchanged by D_2O addition); 7.22 – 7.86 (*m*, 4 arom. H). ^{13}C-NMR: 23.29 (Me); 23.70 (MeCO); 75.75 (C–OH); 114.17 (*d*, ^2J(C,F) = 24.3, arom. CH); 119.27 (*d*, ^2J(C,F) = 21.3, arom. CH); 122.88 (*d*, ^4J(C,F) = 3.1, arom. CH); 125.14 (*d*, ^3J(C,F) = 8.2, arom. CH); 130.96 (*d*, ^3J(C,F) = 8.2, arom. CH); 162.78 (*d*, ^1J(C,F) = 248.1, arom. C); 164.79, 165.73 (2 C of oxadiazole); 204.37 (C=O). EI-MS: 251 (9, M^+), 207 (63), 167 (42), 149 (100), 121 (27), 95 (10), 70 (13), 57 (27). Anal. calc. for $C_{12}H_{11}FN_2O_3$ (250.23): C 57.60, H 4.43, N 11.20; found: C 57.55, H 4.41, N 11.15.

3-Hydroxy-3-(5-phenyl-1,3,4-oxadiazol-2-yl)butan-2-one (**4g**). White crystals. M.p. 104.1 – 106.0°. Yield: 274 mg (85%). IR (KBr): 3230 (OH), 2986, 2848, 2360, 2344, 1725, 1608, 1482, 1293, 1155, 782. ^{1}H-NMR: 1.95 (*s*, Me); 2.36 (*s*, MeCO); 4.69 (*s*, OH, exchanged by D_2O addition); 7.50 – 7.56 (*m*, 3 arom. H); 8.05 (*d*, *J* = 6.5, 3 arom. H). ^{13}C-NMR: 23.25 (Me); 23.71 (MeCO); 75.73 (C–OH); 123.30 (arom. C); 127.11, 129.08, 132.13 (5 arom. CH); 152.81, 152.84 (2 C of oxadiazole); 204.56 (C=O). EI-MS: 233 (30, M^+), 215 (4), 189 (100), 147 (32), 119 (4), 104 (35), 77 (17), 43 (24). Anal. calc. for $C_{12}H_{12}N_2O_3$ (232.24): C 62.06, H 5.21, N 12.06; found: C 62.00, H 5.18, N 12.01.

3-Hydroxy-3-[5-(thiophen-2-yl)-1,3,4-oxadiazol-2-yl]butan-2-one (**4h**). White crystals. M.p. 70.0 – 71.5°. Yield: 195 (82%). IR (KBr): 3246 (OH), 2924, 1724, 1602, 1418, 1353, 727. ^{1}H-NMR: 1.92 (*s*, Me); 2.35 (*s*, MeCO); 4.80 (*s*, OH, exchanged by D_2O addition); 7.11 – 7.19 (*m*, 1 H of thiophene); 7.56 (*d*, *J* = 4.7, 1 H of thiophene); 7.75 (*d*, *J* = 3.2, 1 H of thiophene). ^{13}C-NMR: 23.56, 24.08 (2 Me); 76.05 (C–OH); 124.68 (C of thiophene); 128.54, 130.85, 131.12 (3 CH of thiophene); 162.30, 165.20 (2 C=N); 204.90 (C=O). EI-MS: 238 (3, M^+), 196 (72), 194 (38), 168 (4), 153 (62), 127 (23), 111 (55), 97 (5), 83 (13), 69 (14). Anal. calc. for $C_{10}H_{10}N_2O_3S$ (238.26): C 50.41, H 4.23, N 11.76; found: C 50.29, H 4.19, N 11.70.

3-Hydroxy-3-[5-(4-methylphenyl)-1,3,4-oxadiazol-2-yl]butan-2-one (**4i**). White crystals. M.p. 97.5 – 98.5°. Yield: 209 mg (85%). IR (KBr): 3422 (OH), 2921, 1728, 1612, 1498, 1362, 826. ^{1}H-NMR: 1.92, 2.34, 2.39 (3*s*, 3 Me); 4.91 (*s*, OH; exchanged by D_2O addition); 7.27 (*d*, *J* = 8.0, 2 arom. H); 7.89 (*d*, *J* = 8.0, 2 arom. H). ^{13}C-NMR: 21.93, 23.52, 24.10 (3 Me); 76.10 (C–OH); 120.71 (arom. C); 127.33, 130.05 (4 arom. CH); 143.07 (arom. C); 165.56, 166.17 (2 C=N); 205.13 (C=O). EI-MS: 246 (3, M^+), 223 (5), 204 (50), 161 (45), 118 (43), 105 (5), 91 (50), 77 (9), 65 (18), 43 (100). Anal. calc. for $C_{13}H_{14}N_2O_3$ (246.26): C 63.40, H 5.73, N 11.38; found: C 63.37, H 5.69, N 11.33.

3-Hydroxy-3-[5-(3-methylphenyl)-1,3,4-oxadiazol-2-yl]butan-2-one (**4j**). White crystals. M.p. 75.0 – 76.5°. Yield: 217 mg (88%). IR (KBr): 3421 (OH), 2926, 1728 (C=O), 1609, 1458, 1377, 806.

^{1}H-NMR: 1.92, 2.35, 2.39 (3*s*, 3 Me); 4.92 (*s*, OH; exchanged by D_2O addition); 7.26 – 7.38 (*m*, 2 arom. H); 7.75 – 7.89 (*m*, 2 arom. H). ^{13}C-NMR: 21.53, 23.51, 24.10 (3 Me); 76.12 (C–OH); 123.34 (arom. C); 124.52, 127.83, 129.24, 133.23 (4 arom. CH); 139.26 (arom. C); 165.74, 166.16 (2 C=N); 205.10 (C=O). Anal. calc. for $C_{13}H_{14}N_2O_3$ (246.26): C 63.40, H 5.73, N 11.38; found: C 63.37, H 5.66, N 11.30. EI-MS: 247 (2, M^+), 204 (100), 161 (53), 133 (4), 118 (46), 91 (39), 77 (5), 65 (14), 43 (76), 41 (4).

3-Hydroxy-3-[5-(naphthalen-2-yl)-1,3,4-oxadiazol-2-yl]butan-2-one (**4k**). White crystals. M.p. 120.0 – 121.6°. Yield: 243 mg (86%). IR (KBr): 3448 (OH), 2923, 1725 (C=O), 1577, 1458, 1362, 755. ^{1}H-NMR: 1.99, 2.40 (2*s*, 2 Me); 4.79 (*s*, OH; exchanged by D_2O addition); 7.54 – 7.61 (*m*, 2 arom. H); 7.87 – 7.91 (*m*, 2 arom. H); 7.95 (*d*, *J* = 8.6, 1 arom. H); 8.11 (*d*, *J* = 8.6, 1 arom. H); 8.54 (*s*, 1 arom. H). ^{13}C-NMR: 23.67, 24.13 (2 Me); 76.18 (C–OH); 120.82 (arom. C); 123.47, 127.55, 128.13, 128.31, 128.56, 129.24, 129.46 (7 arom. CH); 133.07, 135.19 (2 arom. C); 165.83, 166.30 (2 C=N); 205.01 (C=O). EI-MS: 282 (2, M^+), 240 (100), 197 (67), 153 (72), 127 (47). Anal. calc. for $C_{16}H_{14}N_2O_3$ (282.29): C 68.07, H 5.00, N 9.92; found: C 68.00, H 4.94, N 9.89.

3-[5-(Furan-2-yl)-1,3,4-oxadiazol-2-yl]-3-hydroxybutan-2-one (**4l**). White crystals. M.p. 100.6° (dec.). Yield: 178 mg (80%). IR (KBr): 3423 (OH), 2924, 1722 (C=O), 1636, 1458, 1370, 752. ^{1}H-NMR: 1.94, 2.35 (2*s*, 2 Me); 4.66 (*s*, OH, exchanged by D_2O addition); 6.56 – 6.63 (*m*, 1 H of furan); 7.19 (*d*, *J* = 3.2, 1 H of furan); 7.65 (*s*, 1 H of furan). ^{13}C NMR: 23.64, 24.02 (2 Me); 76.02 (C–OH); 112.61, 115.28, 146.46 (3 CH of furan); 139.20 (C of furan); 163.01, 165.08 (2 C=N); 204.62 (C=O). EI-MS: 222 (2, M^+), 196 (29), 180 (10), 153 (27), 127 (11), 111 (24), 95 (10), 83 (6), 43 (100), 41 (5). Anal. calc. for $C_{10}H_{10}N_2O_4$ (222.20): C 54.05, H 4.54, N 12.61; found: C 54.00, H 4.49, N 12.58.

This work was supported by the 'Zanjan University' and the '*Iran National Science Foundation: INSF*'.

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Received June 3, 2010