Reactions of Enaminones with Diazocarbonyl Compounds

by **Füsun Şeyma Güngör***, **Neslihan Hancıoğlu**, and **Olcay Anaç***

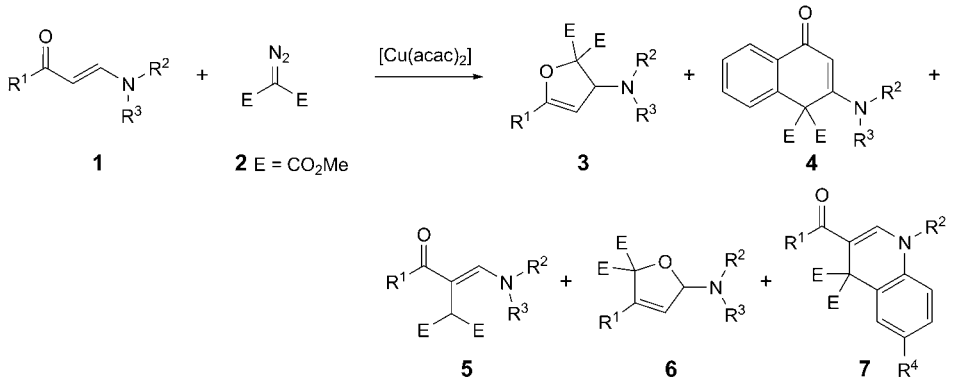
Istanbul Technical University, Faculty of Science and Letters, Department of Chemistry, 34469, Maslak, Istanbul, Turkey (phone: +90-212-2856825; fax: +90-212-2856386; gungorfus@itu.edu.tr)

The [Cu(acac)$_2$]-catalyzed reactions of several tertiary enaminones with three diazocarbonyl compounds, *i.e.*, dimethyl diazomalonate, ethyl diazoacetoacetate, and ethyl diazoacetate, yielded amino- and additionally carbonyl-substituted dihydrofurans, together with further furan derivatives. Due to the conjugation of α-carbonyl/α-Ph groups, reactions proceeded only *via* 1,5-electrocyclization of corresponding keto-ylides. On the other hand, in the absence of any α-substituent, tertiary enaminone and ethyl diazoacetate, reacted *via* an accompanying mechanism by a push-pull cyclopropane intermediate, to yield a 2,4-dicarbonyl-substituted furan in one step with moderate yield.

Introduction. – We have already reported our initial findings on the reactions of tertiary enaminones **1** with dimethyl diazomalonate **2** in the presence of [Cu(acac)$_2$] [1]. In contrast to the present literature [2], we determined the formation of significant amounts of dihydrofuran derivatives **3** along with naphthalenone **4**, and formal C_α–H insertion products **5**. Moreover, we recently performed [Cu(acac)$_2$]-catalyzed reactions of dimethyl diazomalonate with several more elaborate enaminone systems to extend this chemistry and to investigate the critical parameters (*Scheme 1*) [1][3].

The chemoselectivities of the reactions, including those of the new tertiary enaminones, [1][3] indicated that significant amounts of 2,3-dihydrofuran **3**, 2,5-

Scheme 1



dihydrofuran **6**, naphthalenone **4**, and quinoline derivatives **7** could be obtained mostly as main products. When dialkylamino groups were incorporated in the starting substrates, formal C_α–H insertion products **5** were also found as minor products. Obviously, *via* various possible pathways, this method can be applied in the synthesis of poly-functional derivatives as valuable building blocks, when the starting enaminones are well chosen.

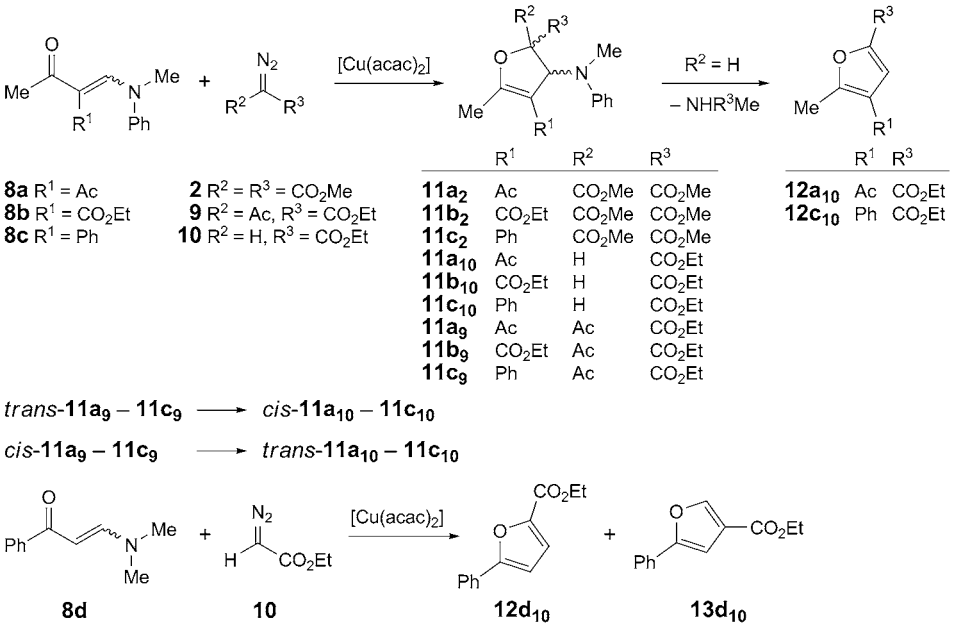
Herein, we report our results from the reactions of (*E*)-enaminones with different substitution patterns. In addition, the the diazo compound was also varied. Thereby, we aimed at clarifying the steric and electronic effects of the reactants on the reaction path.

Results and Discussion. – The [Cu(acac)$_2$]-catalyzed (acac = acetylacetonate = pentane-2,4-dionate) reactions of enaminones **8a** and **8b**, with dimethyl diazomalonate (**2**), ethyl diazoacetoacetate (= ethyl 2-diazo-3-oxobutanoate; **9**), and ethyl 2-diazoacetate (**10**), respectively, were studied first (*Scheme 2*). The reasons for starting with **8a** and **8b** can be summarized as follows: Compounds **8a** and **8b** were regarded to be capable of forming some novel products by electrocyclization of the carbonyl-ylide intermediates with the Ph ring to yield benzo[*c*][1,5]oxazocine derivatives. This pathway is, however, not possible for enaminones of type **8d** (*Scheme 2*).

Rotation around formal C=C bonds in this kind of conjugated enaminones such as (*E*/*Z*)-**8b** might be possible [4], and a preference between these two different conjugated ylides might also be conceivable.

Besides (*E*/*Z*)-**8b**, α-phenyl (*E*/*Z*)-enaminone **8c** was also used as starting compound to determine the effect of the α-Ph group on the possible rotation of C=C bond of ylide intermediates.

Scheme 2



Finally, benzoyl enamine **8d**, which had been reacted previously with diazo compound **2** to yield **3**–**5** (*Scheme 1*) [1][3], was studied once more with respect to its reaction with ethyl 2-diazoacetate (**10**) to clarify the effect of diazomethyl moiety in these enaminone-ylide reactions. Our experimental results are compiled in the *Table*.

Table. *Reactions of Enaminones* **8a**–**8d** *with Diazo Compounds* **2**, **9**, *and* **10**[a])

Enaminone	Diazo compound	**11** (Yield [%])[b])	**12** (Yield [%])[b])	**13** (Yield [%])[b])
8a	**2**	1 **11a$_2$** (60%)	–	–
8b	**2**	1 **11b$_2$** (55%)	–	–
8b	**2**	1 **11a$_2$** (65%)	–	–
8a	**10**	1 *cis*-**11a$_{10}$** (18%)/ *trans*-**11a$_{10}$** (27%) 1 : 1.73[c])	0.2 **12a$_{10}$** (50%)	–
8b	**10**	1 *cis*-**11b$_{10}$** (45%)/ *trans*-**11b$_{10}$**[d]) 1 : 1[c])	–	–
8c	**10**	1 *cis*-**11c$_{10}$**/*trans*-**11c$_{10}$** 1 : 1[c])	–	–
8d	**10**	–	8.7 **12d$_{10}$** (50%)	1 **13d$_{10}$** (40%)
8a	**9**	1 *cis*-**11a$_9$**/*trans*-**11a$_9$** 1 : 3.5[c])	–	–
8b	**9**	1 *cis*-**11b$_9$**/*trans*-**11b$_9$** 1 : 4.53[c])	–	–
8c	**9**	1 *cis*-**11c$_9$**/*trans*-**11c$_9$** 1 : 1[c])	–	–

[a]) Normalized by GC values. [b]) Yield of isolated product. [c]) The ratio *cis*-**11**/*trans*-**11** was determined by ^{1}H-NMR of the reaction mixture. [d]) Obtained with ethyl 2-(diazoaceto)acetate dimers.

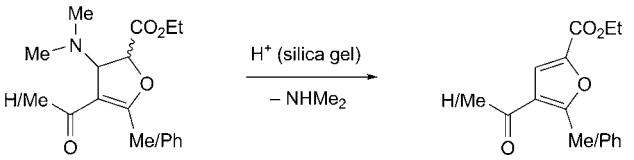
From all reactions of **8a**–**8d** with diazo compounds **2**, **9**, and **10**, dihydrofurans **11** and furans **12** were obtained by cyclization of the corresponding 1,5-carbonyl-ylide intermediates.

In the reactions with **9**, we could easily detect the dihydrofuran derivatives **11a$_9$**–**11c$_9$** by GC/MS and ^{1}H-NMR of the reaction mixture as the sole formal [1 + 1] products (*Table*). Attempts of purification of **11a$_9$** – **11c$_9$** failed due to decomposition, however, the decomposition products turned out to be identical with the dihydrofurans obtained from the reactions of the same enaminones and **10** (*cf.* the *Table*). Another remarkable observation was the formation of *cis*-**11a$_{10}$**–*cis*-**11c$_{10}$** from *trans*-**11a$_9$**–*trans*-**11c$_9$**, and the formation of *trans*-**11a$_{10}$**–*trans*-**11c$_{10}$** from *cis*-**11a$_9$**–*cis*-**11c$_9$** (*Scheme 2*). The mechanism of the observed decomposition reaction is not clear.

Moreover, neither analogous derivatives of **5**/**6** [4] nor novel benzo[*c*][1,5]oxazocine derivatives were detected in our reactions. Obviously, the presence of neither two C=O (*i.e.*, in **8a** and **8b**) nor C=O and Ph groups (*i.e.*, in **8c**) in the conjugated ene-carbonyls enhanced the dihydrofuran-producing carbonyl-ylide formation, suppressing the other pathways by donor-acceptor-substituted cyclopropane intermediates or by nitrogen-ylide intermediates [3].

The reaction of enaminones **8a** and **8d** with **10** yielded also **12a$_{10}$** and **12d$_{10}$**, respectively, which are formed by elimination of Me_2NH from the related parent compounds, **11a$_{10}$** and **11d$_{10}$** (*Scheme 3*). Compound **12d$_{10}$** was the main product (*Table*). This result is not surprising, considering of the relative stability of the related carbocations due to the presence of a Ph group for E_1 elimination of *cis*/*trans*-**11d$_{10}$**.

Scheme 3

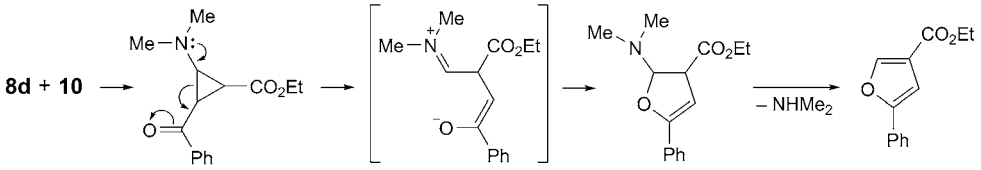


11a$_{10}$
11d$_{10}$

12a$_{10}$
12d$_{10}$

Additionally, an unexpected derivative, **13d$_{10}$** was also obtained (*Scheme 2*). This reaction probably proceeds by ring opening (due to the adjacent Ph group) of the donor-acceptor cyclopropane-carboxylate **14**. Recyclization of the formed intermediate **15** would lead to a dihydrofuran-carboxylate **16**, which, by subsequent Me_2NH elimination, furnishes the 2,4-disubstituted furan **13** (*Scheme 4*).

Scheme 4



14 **15** **16** **13**

According to literature [5], these kinds of 2-substituted furan-4-carboxylates were synthesized starting from the appropriate aldehyde in four steps in 30–40% yield. Our method led to the furan derivative **13d$_{10}$** with the same yield but in only one step. We performed the same reactions with additional catalysts ($[Rh_2(OAc)_4]$ and $[Cu(OTf)_2]$) to increase the yield 2,4-disubstituted furan **13**, however, without success.

Conclusions. – In summary, $[Cu(acac)_2]$-catalyzed reactions of tertiary enaminones **8a**–**8d** and diazocarbonyl compounds **2**, **9**, and **10** led to novel amino- and additionally-carbonyl-substituted 2,3-dihydrofurans **11** and/or furan derivatives **12**. Due to the additional conjugation of α-carbonyl (in **8a** and **8b**)/α-Ph (in **8c**) groups, the reactions proceeded only *via* 1,5-electrocyclization of corresponding keto-ylides. On the other hand, in the absence of any α-substituent, tertiary enaminone **8d** and ethyl 2-diazoacetate (**10**) gave a 2,4-substituted furan **13** by a push-pull cyclopropane intermediate in one step with moderate yield. We also observed that **11a$_9$**–**11c$_9$** easily decomposed to **11a$_{10}$**–**11c$_{10}$** during purification attempts. This degradation mechanism is not clear.

Experimental Part

General. All of the solvents and reactants are commercially available. The reactions of diazo compounds and enaminones were carried out under N_2. Dimethyl diazomalonate [6], ethyl 2-diazoacetate [7], and ethyl 2-(diazoaceto)acetate [8] were prepared according to literature procedures.

^{1}H- and ^{13}C-NMR spectra: in $CDCl_3$, with *Bruker AC* spectrometers; at 500 and 125, or 250 and 60 MHz, resp., at r.t.; δ in ppm rel. to Me_4Si as internal standard, J in Hz. GC/MS: *Hewlett-Packard* instrument equipped with a flame ionization detector; in m/z (rel. %). A cross-linked (phenylmethyl)siloxane cap. column (30 m × 0.32 mm × 0.25 μm) was used with He as the carrier gas (25 psi column head pressure); temp. program: starting with 100° then 5 min isothermal, ramp 20°/min; final 290°, and then 10 min isothermal, retention times (t_R) are given in min.

General Procedure for the Reaction of Enaminones with Diazo Compounds. To a soln. of **8** (2.1 mmol) in benzene (10 ml) was added [Cu(acac)$_2$] (9×10^{-3} mmol), and the mixture was heated at reflux. A soln. of diazo compound (1.4 mmol) in benzene (4 ml) was added to this soln. over 2.5 h under N_2. When the IR spectrum of the reaction mixture indicated total consumption of the diazo compound (absence of the characteristic diazo band), the mixture was filtered, evaporated, and purified by column chromatography (CC) or prep. TLC. The crude mixture contained varying amounts of unidentified compounds (max. 20% by GC).

*Dimethyl 4-Acetyl-5-methyl-3-[methyl(phenyl)amino]furan-2,2(3*H*)-dicarboxylate* (**11a$_2$**): Purification by CC (neutral Al_2O_3; hexane/AcOEt 9:1). Prep. TLC (Al_2O_3; hexane/AcOEt 90:10): **11a$_2$** Yield: 60% (t_R 13.86). Dark-yellow oil. ^{1}H-NMR (250 MHz): 7.29–7.22 (*m*, 2 arom. H); 6.95 (*d*, *J* = 8.0, 2 arom. H); 6.79 (distorted *t*, *J* = 7.2–7.1, 1 arom. H); 6.10 (*s*, CHN); 3.87 (*s*, COOMe); 3.48 (*s*, COOMe), 2.62 (*s*, MeN); 2.41 (*s*, C=CMe); 1.87 (*s*, MeCO). ^{13}C-NMR (60 MHz): 194.5; 168.7; 166.8; 164.8; 149.1; 129.2; 118.4; 113.3; 98.7; 91.6; 68.8; 53.8; 52.8; 32.2; 29.2; 14.8. EI-MS (70 eV): 347 (58, M^+), 241 (30), 197 (100), 167 (48), 151 (30), 107 (91), 77 (32), 59 (20).

*4-Ethyl 2,2-Dimethyl 5-Methyl-3-[methyl(phenyl)amino]furan-2,2,4(3*H*)-tricarboxylate* (**11b$_2$**). Purification by CC (neutral Al_2O_3; hexane/AcOEt 92:8). Prep. TLC (Al_2O_3; hexane/AcOEt 85:15). Yield: 55% (t_R 14.10). ^{1}H-NMR (250 MHz): 7.20 (*t*, *J* = 7.6, 2 H); 6.90 (*d*, *J* = 8.0, 2 H); 6.76 (*t*, *J* = 7.3, 1 H); 5.94 (br. *s*, 1 H); 4.00–3.94 (*m*, 2 H); 3.86 (*s*, 3 H); 3.56 (*s*, 3 H); 2.56 (*s*, 3 H); 2.38 (*s*, 3 H); 0.87 (*t*, *J* = 7.0, 3 H). ^{13}C-NMR (125 MHz): 168.7; 167.1; 165.3; 164.6; 150.7; 129.0; 118.5; 114.4; 103.9; 92.1; 70.1; 60.1; 54.0; 53.1; 32.7; 14.3; 14.1. EI-MS (70 eV): 377 (31.5, M^+), 332 (4), 271 (21), 240 (3), 227 (93), 211 (44), 199 (50), 183 (99), 167 (43), 123 (22), 107 (100), 77 (27), 59 (20).

*Dimethyl 5-Methyl-3-[methyl(phenyl)amino]-4-phenylfuran-2,2(3*H*)-dicarboxylate* (**11c$_2$**). Purification by CC (neutral Al_2O_3; hexane/AcOEt 90:10). Prep. TLC (Al_2O_3; hexane/AcOEt 95:5). Yield: 65% (t_R 15.26). ^{1}H-NMR (250 MHz): 7.33–7.09 (*m*, 2 H); 6.93 (*d*, *J* = 7.7, 2 H); 6.76 (distorted *t*, *J* = 7.0, 1 H); 6.24 (br. *s*, 1 H); 3.88 (*s*, 3 H); 3.46 (*s*, 3 H); 2.64 (*s*, 3 H); 2.25 (*s*, 3 H). ^{13}C-NMR (60 MHz): 168.1; 166.0; 152.4; 149.2; 133.1; 129.0; 128.6; 126.8; 126.4; 117.6; 113.3; 110.1; 89.9; 70.8; 53.6; 52.6; 30.1; 13.8. EI-MS (70 eV): 381 (29, M^+), 322 (4), 275 (80), 231 (100), 216 (65), 185 (52), 128 (25), 107 (72), 77 (21), 59 (12).

Ethyl trans-*4-Acetyl-5-methyl-3-[methyl(phenyl)amino]-2,3-dihydrofuran-2-carboxylate* (*trans*-**11a$_{10}$**). CC (silica gel; hexane/AcOEt 80:20). Yield: 60% (t_R 13.50). Yellow oil. ^{1}H-NMR (250 MHz): 7.21 (distorted *d* of distorted *t*, *J* = 7.2–8.2/2.0–1.5, 2 H); 6.96 (*d*, *J* = 8.8, 2 H); 6.76 (*t*, *J* = 7.3, 1 H); 5.41 (*d*, *J* = 1.5, 1 H); 4.75 (*d*, *J* = 2.9, 1 H); 4.26–4.15 (*m*, 2 H); 2.70 (*s*, 3 H); 2.40 (*s*, 3 H); 2.05 (*s*, 3 H); 1.24 (distorted *t*, *J* = 6.8–7.3, 3 H). ^{13}C-NMR (125 MHz): 195.0; 171.2; 169.7; 149.1; 129.6; 119.0; 114.9; 111.5; 82.5; 67.4; 62.3; 31.7; 29.4; 15.2; 14.3. EI-MS (70 eV): 303 (27, M^+), 230 (9), 196 (13), 181 (18), 153 (16), 125 (48), 107 (100), 77 (19).

Ethyl cis-*4-Acetyl-5-methyl-3-[methyl(phenyl)amino]-2,3-dihydrofuran-2-carboxylate* (*cis*-**11a$_{10}$**). CC (neutral Al_2O_3; hexane/AcOEt 90:10). Yield: 45% (t_R 13.77). Yellow oil. ^{1}H-NMR (250 MHz): 7.23–7.20 (*m*, 2 H); 6.83 (*d*, *J* = 8.1, 2 H); 6.76 (*t*, *J* = 6.8, 1 H); 5.55 (*d*, *J* = 8.7, 1 H); 5.11 (*d*, *J* = 8.9, 1 H); 4.03–3.91 (*m*, 2 H); 2.64 (*s*, 3 H); 2.40 (*s*, 3 H); 1.89 (*s*, 3 H); 0.93 (*t*, *J* = 7.1, 3 H). ^{13}C-NMR (60 MHz): 194.7; 170.5; 167.3; 149.2; 129.1; 118.0; 113.4; 111.7; 83.3; 65.0; 61.6; 32.3; 29.1; 15.0; 13.6.

Diethyl trans-*5-Methyl-3-[methyl(phenyl)amino]-2,3-dihydrofuran-2,4-dicarboxylate* (*trans*-**11b$_{10}$**). CC (neutral Al_2O_3; hexane/AcOEt 95:5). Prep. TLC (Al_2O_3; hexane/AcOEt 90:10). Yield: 26% (t_R 13.67). Dark-yellow oil. ^{1}H-NMR (250 MHz): 7.21 (*t*, *J* = 7.7, 2 H); 6.90 (*d*, *J* = 8.1, 2 H); 6.76 (*t*, *J* = 7.0, 1 H); 5.42 (br. *s*, 1 H); 4.84 (*d*, *J* = 2.7, 1 H); 4.25–4.19 (*m*, 2 H); 4.02–3.97 (*m*, 2 H); 2.67 (*s*, 3 H); 2.37 (*s*, 3 H); 1.30 (distorted *t*, *J* = 6.8, 3 H); 0.97 (*t*, *J* = 7.1, 3 H). ^{13}C-NMR (125 MHz): 169.6; 168.7; 163.8; 148.6; 127.9; 117.1; 113.5; 102.2; 81.6; 66.9; 60.9; 58.7; 30.5; 13.2. EI-MS (70 eV): 333 (20, M^+), 288 (5), 226 (31), 197 (29), 181 (38), 170 (23), 153 (46), 127 (24), 107 (100), 77 (20), 51 (18).

Diethyl cis-*5-Methyl-3-[methyl(phenyl)amino]-2,3-dihydrofuran-2,4-dicarboxylate* (*cis*-**11b$_{10}$**). CC (neutral Al_2O_3; hexane/AcOEt 95:5). Prep. TLC (Al_2O_3; hexane/AcOEt 90:10). Yield: 45% (t_R 13.89). Yellow oil. ^{1}H-NMR (250 MHz): 7.18 (*t*, *J* = 7.8, 2 H); 6.80–6.70 (*m*, 3 H); 5.41 (*d*, *J* = 8.7, 1 H); 5.10 (*d*, *J* = 9.1, 1 H); 4.15–3.83 (*m*, 4 H); 2.59 (*s*, 3 H); 2.37 (*s*, 3 H); 1.29 (*t*, *J* = 7.1, 3 H); 1.00 (*t*, *J* = 7.2, 3 H). ^{13}C-NMR (125 MHz): 169.2; 166.5; 163.9; 149.4; 127.8; 116.9; 113.3; 102.5; 82.4; 65.0; 60.5; 60.2; 30.5; 13.1; 13.0.

Diethyl trans-*2-Acetyl-5-methyl-3-[methyl(phenyl)amino]-2,3-dihydrofuran-2,4-dicarboxylate* (*trans*-**11b$_9$**). CC: t_R 13.67. ^{1}H-NMR (500 MHz): 7.19 (*t*, *J* = 8.1, 2 H); 6.90 (*d*, *J* = 7.8, 2 H); 6.73 (*t*, *J* = 7.1, 1 H); 5.95 (br. *s*, 1 H); 4.30–4.22 (*m*, 2 H); 3.93–3.88 (*m*, 1 H); 3.83–3.79 (*m*, 1 H); 2.60 (*s*, 3 H); 2.35 (*s*, 3 H); 2.15 (*s*, 3 H); 1.31–1.26 (*m*, 3 H); 0.77 (*t*, *J* = 6.8, 3 H). ^{13}C-NMR (125 MHz): 196.4; 167.8; 166.6; 163.4; 128.0; 117.5; 112.8; 102.5; 95.2; 68.7; 62.2; 59.7; 31.3; 25.8; 24.8; 13.0; 12.7. EI-MS (70 eV): 375 (33, M^+), 288 (8), 227 (99), 199 (88), 181 (20), 153 (13), 107 (100), 77 (13).

Ethyl 2-Acetyl-5-methyl-3-[methyl(phenyl)amino]-4-phenyl-2,3-dihydrofuran-2-carboxylate (**11c$_9$**). Prep. TLC (neutral Al_2O_3; hexane/AcOEt 80:20): **11c$_9$** (3:1 isomer mixture (^{1}H-NMR). t_R 15.26. Light-yellow oil. ^{1}H-NMR (250 MHz): 7.29–7.05 (*m*, 7 H); 6.95 (*d*, *J* = 7.9, 2 H); 6.78 (*t*, *J* = 7.1, 1 H); 6.21 (br. *s*, 1 H); 4.36–4.31 (*m*, 2 H); 2.58 (*s*, 3 H); 2.26 (*s*, 3 H); 2.23 (*s*, 3 H); 1.33 (*t*, *J* = 7.1, 3 H). ^{13}C-NMR (125 MHz): 199.8; 168.9; 152.7; 149.5; 133.4; 129.5; 128.8; 126.6; 126.5; 118.2; 113.4; 110.5; 94.7; 71.4; 63.0; 32.4; 27.3; 14.3; 14.1. EI-MS (70 eV): 379 (10, M^+), 231 (100), 203 (63), 186 (56), 158 (23), 128 (30), 107 (74), 77 (21).

Ethyl 4-Acetyl-5-methylfuran-2-carboxylate (**12a$_{10}$**). Yield: 55%. t_R 10.04. White solid. M.p. 98–100°. ^{1}H-NMR (250 MHz): 8.01 (*s*, C=CH); 4.37–4.29 (*m*, COOC*H*$_2$Me); 2.52 (*s*, Me); 2.41 (*s*, COMe); 1.39–1.32 (*m*, COOCH$_2$*Me*). ^{13}C-NMR (60 MHz): 192.8; 159.2; 149.9; 142.2; 130.1; 128.0; 61.0; 28.7; 14.3; 10.4. EI-MS (70 eV): 196 (88, M^+), 162 (90), 153 (100), 137 (8), 109 (20).

Ethyl 5-Phenylfuran-2-carboxylate (**12d$_{10}$**): Yield 50%. t_R 10.8. Yellow oil. ^{1}H-NMR (250 MHz): 7.78 (*d*, *J* = 7.0, 2 H); 7.41 (distorted *t*, *J* = 7.2, 2 H); 7.35–7.33 (*m*, 1 H); 6.72 (*d*, *J* = 3.5, 1 H); 4.37 (*q*, *J* = 7.1, 2 H); 1.38 (*t*, *J* = 7.1, 3 H). EI-MS (70 eV): 217 (15, $[M+1]^+$), 216 (100), 188 (72), 171 (60), 144 (70), 115 (95).

Ethyl 5-Phenylfuran-3-carboxylate (**13d$_{10}$**): Yield: 40%. t_R 10.69. ^{1}H-NMR (250 MHz): 8.01 (*s*, C=CH); 7.66 (*d*, *J* = 7.3, 2 arom. H); 7.39 (*t*, *J* = 7.4, 2 arom. H); 7.30 (*d*, *J* = 7.1, 1 arom. H) 6.95 (*s*, C=CH), 4.31 (*q*, *J* = 7.1, COOC*H*$_2$Me); 1.35 (*t*, *J* = 7.1, COOCH$_2$*Me*). ^{13}C-NMR (125 MHz): 162.1; 154.1; 127.8; 127.1; 123.0; 120.3; 103.5; 59.5; 13.3.

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