1,3-Dipolar cycloadditions of heterocycles XXI.* Cycloaddition of 2,5-dimethyl-3-furonitrile oxide with cyclic and heterocyclic compounds

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Received 24 November 1989

Dedicated to Professor P. Hrnčiar, DrSc., in honour of his 60th birthday

Cycloadditions of 2,5-dimethyl-3-furonitrile oxide to 1,3-cyclohexadiene, 1,3-cyclooctadiene, indene, acenaphthylene, 2,5-dihydrofuran, 2*H*,4*H*,7*H*-1,3-dioxepine, 1,4-epoxy-1,4-dihydronaphthalene, dimethyl 7-oxabicyclo[2,2,1]hept-2-ene-5,6-dicarboxylate, dimethyl 7-(diphenylmethylene)bicyclo[2,2,1]hept-2-ene-5,6-dicarboxylate, and dimethyl 7-oxabicyclo[2,2,1]hepta-2,3-diene-2,3-dicarboxylate, are described. Regio- as well as *endo*/*exo* selectivity of the reactions is discussed. The formation of 1,3-addition products in cycloadditions to conjugated dienes was not observed.

In our previous papers we have assessed the reactivity of 2-furonitrile oxide towards some oxygen-containing heterocyclic compounds [1]. Now β-substituted furan derivatives constitute a group of not readily accessible compounds, interesting for their potential biological activity [2]. Some β-substituted furans have already been commercialized as fungicides, for example Furcarbanil (*Ic*) and Furmecyclox (*Id*) [3]. This has motivated our further investigation into 1,3-cycloaddition reactions with heterocyclic dipolarophiles, with the aim to prepare condensed, 2,5-dimethyl-3-furyl-substituted, isoxazolines. In the course of these investigations we described [4] the preparation and reactions of 2,5-dimethyl-3-furonitrile oxide (*Ia*) with alkenes and alkynes. Now we deal with the cycloadditions of *Ia* to some cyclic and heterocyclic dipolarophiles (Scheme 1).

2,5-Dimethyl-3-furonitrile oxide (*Ia*) was generated *in situ* from the corresponding oxime *Ib* by the action of sodium hypochlorite in the presence of catalytic amount of triethylamine [5], and in the presence of dipolarophile. Nitrile oxide *Ia* reacted smoothly and with good to very good yields with both cyclic and heterocyclic dipolarophiles (1,3-cyclohexadiene, 1,3-cyclooctadiene, indene, acenaphthylene, 2,5-dihydrofuran, 2*H*,4*H*,7*H*-1,3-dioxepine (*II*), 1,4-epoxy-1,4-dihydronaphthalene, dimethyl 7-oxabicyclo[2,2,1]hept-2-ene-5,6-

* For Part *XX* see *Collect. Czechoslov. Chem. Commun. 53*, 1753 (1988).

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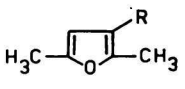

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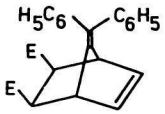

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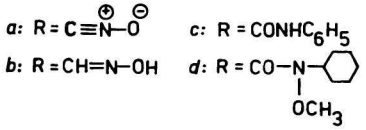

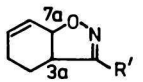

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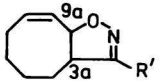

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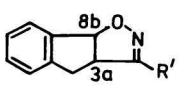

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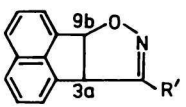

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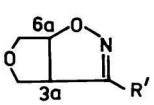

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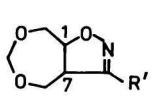

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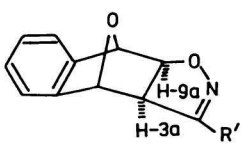

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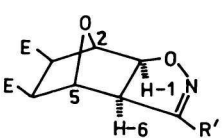

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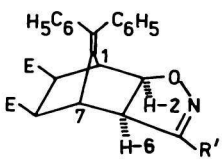

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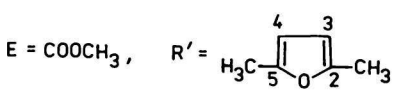
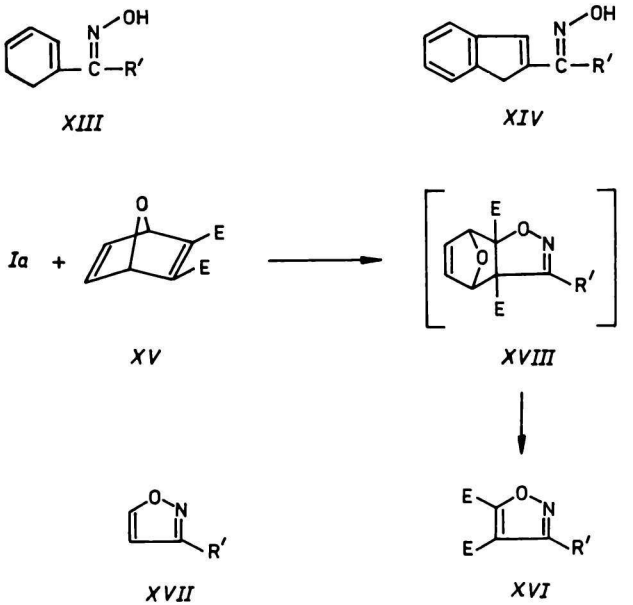


Scheme 1

-dicarboxylate, dimethyl 7-(diphenylmethylene)bicyclo[2,2,1]hept-2-ene-5,6--dicarboxylate (*III*), and dimethyl 7-oxabicyclo[2,2,1]hepta-2,3-diene-2,3-dicarboxylate). The structure of thus formed fused heterocyclic compounds *IV*—*XII* was inferred from structures of starting material and ^{1}H NMR and ^{13}C NMR spectral data, correlated with the corresponding chemical shifts of analogous 2-furyl [1], phenyl [6—10], and trifluoroacetyl derivatives [11]. The reaction of nitrile oxide *Ia* with a conjugated alkene, such as indene, or cyclic dienes



Scheme 1 (Continued)

(1,3-cyclohexadiene, 1,3-cyclooctadiene) gave only cycloadducts *IV*—*VI*, *i.e.* regioisomeric cycloadducts did not arise in substantial amounts (based on the ^{1}H NMR spectra of crude reaction mixtures). This tallies well with the behaviour of nitrile oxides in cycloadditions [12]. Contrary to the observed outcome of the reaction of benzonitrile oxides [6] and trifluoroacetonitrile oxide [11], *Ia* gave with conjugated dienes no linear oximes *XIII*, *XIV* These would have arisen in a 1,3-addition, initiated by an electrophilic attack of nitrile oxides at the conjugated double bond. If this assumption were true, the proportion of 1,3-addition products would be expected to rise with the increasing electrophilicity of the dipole [12]. MNDO calculations, presented in [4], indicated an increased nucleophilicity of *Ia*, due to the presence of two methyl groups, over that of 3-furonitrile oxide or benzonitrile oxide. This could account for the missing linear 1,3-adducts in the reactions of *Ia*.

The reaction of *Ia* with indene thus produced only 3-(2,5-dimethyl-3-furyl)--3a,8b-dihydroindeno[2,3-*d*]isoxazole (*VI*). The structure of the product was inferred from the comparison of its ^{1}H NMR spectrum with that of the 3--phenyl-substituted analogue [3], and by analysis of the signal multiplicity pattern (H-8b in *VI*). In case of the other regioisomer a different pattern, namely a doublet of doublet of doublet, would be expected for the hydrogen adjacent to oxygen of isoxazoline. In a similar manner the structures of adducts with

1,3-cyclohexadiene (*IV*) and 1,3-cyclooctadiene (*V*) were assigned. In the case of cycloaddition of *Ia* to acenaphthylene only one adduct can possibly arise, namely 3-(2,5-dimethyl-3-furyl)-3a,9b-dihydroacenaphthyleno[1,2-*d*]isoxazole (*VII*).

In addition to hydrocarbon-type dipolarophiles we have also investigated the reactivity of *Ia* towards selected heterocyclic compounds, such as 2,5-dihydrofuran (*VIII*), 2*H*,4*H*,7*H*-1,3-dioxepine (*IX*), 1,4-epoxy-1,4-dihydronaphthalene (*X*), dimethyl 7-oxabicyclo[2,2,1]hept-2-ene-5,6-dicarboxylate (*XI*), dimethyl 7-(diphenylmethylene)bicyclo[2,2,1]hept-2-ene-5,6-dicarboxylate (*XII*). Products have been assigned structure with the help of spectral data of analogous 2-furyl-substituted derivatives [4, 10]. In accord with our previously published results these reactions too produced exclusively *exo* derivatives *X*—*XII*.

The outcome of the cycloaddition of dimethyl 7-oxabicyclo[2,2,1]hepta-2,3-diene-2,3-dicarboxylate (*XV*) was rather unexpected insofar as only 3-(2,5-dimethyl-3-furyl)isoxazole-4,5-dicarboxylate (*XVI*) was produced, while the other expected product — 3-(2,5-dimethyl-3-furyl)isoxazole (*XVII*) could not be detected (by ^{1}H NMR) in the crude reaction mixture. As it turned out, the primary cycloadduct *XVIII* was unstable under the reaction conditions, and underwent a *retro*-Diels—Alder reaction leading to *XVI*. The remarkable feature of the whole process is its total (100%) *site* selectivity, *i.e.* the reaction only took place at the deactivated double bond. Now 1,3-dipolar cycloadditions of oxanorbornadiene *XV* to the deactivated double bond are HOMO(dipole)—LUMO(dipolarophile) controlled [13—16]. Accordingly benzonitrile oxide gives 75 : 25 [13, 14], and benzoylnitrile oxide 55 : 45 *site* selectivity [16], in both cases preferring the attack at the deactivated double bond. Exclusive formation of *XVI* in the case of cycloaddition of *Ia* to *XV* can be interpreted in terms of a HOMO(*Ia*)—LUMO(*XV*) frontier orbital interaction. In other words, *Ia* has higher nucleophilicity than the above-mentioned nitrile oxides. High nucleophilicity of *Ia* was also predicted by MNDO calculations [4].

Experimental

Melting points are uncorrected. ^{1}H NMR and ^{13}C NMR spectra of $CDCl_3$ solutions were recorded on a Jeol JX-100 and a Varian VXR 300 spectrometer, respectively. Chemical shifts are in parts per million (δ) from internal tetramethylsilane. UV spectra were obtained with a Zeiss, Jena M-40 spectrometer (methanol, thermostated cuvettes, ε values in $m^2\,mol^{-1}$). The progress of reactions was monitored by TLC checks on silufol (silica gel/starch, fluorescence indicator); for detection UV light of the wavelength 254 nm was used. Chromatographic purification on silica gel columns used chloroform, or a mixture of cyclohexane—ethyl acetate ($\varphi_r = 4:1$) as eluant.

2,5-Dimethyl-3-furancarbaldehyde was prepared according to [17], and by the procedure described in [4] it subsequently converted to oxime (*Ib*). The synthesis of starting heterocyclic dipolarophiles has been described earlier [7—10, 15].

Cycloadducts IV—XII, XVI

To a stirred mixture, prepared from 0.01 mol (derivatives *VI*, *VII*, *X—XII*), or 0.05 mol (derivatives *IV*, *V*, *VIII*, *IX*) of dipolarophile dissolved in 25 cm^3 of dichloromethane, 15 cm^3 of 12% NaOCl, and 0.2 cm^3 of triethylamine, the 30 cm^3 dichloromethane solution of 0.01 mol of 2,5-dimethyl-3-furancarboxaldehyde oxime was at 0°C added during an hour. Stirring was continued for another hour at 0°C and for 18 h at laboratory temperature. The organic layer was separated and aqueous layer repeatedly extracted by dichloromethane. Combined extracts were dried by $MgSO_4$ and evaporated to dryness. The solid residue can be further purified by chromatography, crystallization or vacuum distillation, respectively.

3-(2,5-Dimethyl-3-furyl)-3a,4,5,7a-tetrahydrobenzoisoxazole (IV), yield = 51%, m.p. = 36—38°C. For $C_{13}H_{15}NO_2$ (M_r = 217.11) w_i(calc.): 71.92% C, 6.97% H, 6.45% N; w_i(found): 71.69% C, 6.81% H, 6.31% N. UV spectrum, λ_{max}/nm (log ε): 262 (2.73). ^{1}H NMR spectrum, δ: 5.90—6.17 (m, 3H, H-6, H-7, H-4′), 4.68 (d, d, 1H, H-7a) $J_{3a,7a}$ = 8.5 Hz, $J_{7,7a}$ = 2.0 Hz, 3.20—3.40 (m, 1H, H-3a), 2.45 (s, 3H, CH_3), 2.25 (s, 3H, CH_3), 1.25—2.32 (m, 4H, H_2-4, H_2-5). ^{13}C NMR spectrum, δ: 150.42, 156.36 (s, s, C-3, C-2′, C-5′), 122.34, 132.75 (d, d, C-7, C-6), 110.69 (s, C-3′), 105.43 (d, C-4′), 75.59 (d, C-7a), 45.64 (d, C-3a), 22.94 (t, C-5), 22.18 (t, C-4), 13.75 (q, CH_3), 13.17 (q, CH_3).

3-(2,5-Dimethyl-3-furyl)-3a,4,5,6,7,9a-hexahydrocycloocta[d]isoxazole (V), yield = = 61%, b.p. = 100—110°C (p = 6.66 Pa). For $C_{15}H_{19}NO_2$ (M_r = 245.34) w_i(calc.): 73.43% C, 7.81% H, 5.70% N; w_i(found): 73.18% C, 7.58% H, 5.61% N. UV spectrum, λ_{max}/nm (log ε): 260 (2.75). ^{1}H NMR spectrum, δ: 5.98 (s, 1H, H-4′), 5.41—6.39 (m, 2H, H-8, H-9), 4.36 (m, 1H, H-9a), 3.50 (m, 1H, H-3a), 2.41 (s, 3H, CH_3), 2.25 (s, 3H, CH_3), 1.73—2.67 (m, 8H, H_2-4, H_2-5, H_2-6, H_2-7).

3-(2,5-Dimethyl-3-furyl)-3a,8b-dihydroindeno[2,3-d]isoxazole (VI), yield = 37%, m.p. 130—131°C. For $C_{16}H_{15}NO_2$ (M_r = 253.32) w_i(calc.): 75.86% C, 5.97% H, 5.52% N; w_i(found): 76.04% C, 5.80% H, 5.50% N. UV spectrum, λ_{max}/nm (log ε): 265 (2.47). ^{1}H NMR spectrum (deuterated DMSO), δ: 7.19—7.40 (m, 4H, H_{arom}), 6.12 (s, 1H, H-4′), 5.97 (d, 1H, H-8b) $J_{3a,8b}$ = 9.3 Hz, 4.36 (d, d, d, 1H, H-3a), 3.33 (d, d, 1H, H_A-4) J = 15.0 Hz, J = 9.0 Hz, 2.91 (d, 1H, H_B-4) J = 15.0 Hz, 2.29 (s, 3H, CH_3), 2.20 (s, 3H, CH_3). ^{13}C NMR spectrum (deuterated DMSO), δ:153.81 (s, C=N), 150.46, 149.28 (s, s, C-2′, C-5′), 141.05, 141.01 (s, s, C_{arom}), 129.40, 127.44, 125.79, 125.03 (doublets, C_{arom}), 110.89 (s, C-3′), 106.74 (d, C-4′), 87.96 (d, C-8b), 51.58 (d, C-3a), 36.42 (t, C-4), 13.87 (q, CH_3), 13.30 (q, CH_3).

3-(2,5-Dimethyl-3-furyl)-3a,9b-dihydroacenaphthyleno[1,2-d]isoxazole (VII), yield = 61%, m.p. = 136—138°C. For $C_{19}H_{15}NO_2$ (M_r = 289.31) w_i(calc.): 78.87% C, 5.22% H, 4.84% N; w_i(found): 78.70% C, 5.24% H, 4.91% N. UV spectrum, λ_{max}/nm (log ε): 286 (2.96). ^{1}H NMR spectrum (deuterated DMSO), δ: 7.25—7.81 (m, H_{arom}), 6.47 (s, 1H, H-4'), 6.41 (d, 1H, H-9b) $J_{3a,9b}$ = 8.7 Hz, 5.65 (d, 1H, H-3a), 2.28 (s, 3H, CH_3), 2.21 (s, 3H, CH_3). ^{13}C NMR spectrum (deuterated DMSO), δ: 151.35, 150.52, 150.11 (s, s, s, C-3, C-2', C-5'), 141.61, 140.54, 136.25, 131.32 (singlets, C_{arom}), 128.66, 128.47, 125.84, 124.36, 122.32, 120.73 (doublets, C_{arom}), 110.82 (s, C-3'), 106.99 (d, C-4'), 86.82 (d, C-9b), 59.41 (d, C-3a), 13.95 (q, CH_3), 13.44 (q, CH_3).

3-(2,5-Dimethyl-3-furyl)-3a,4,6,6a-tetrahydrofuro[3,4-d]isoxazole (VIII), yield = = 70%, m.p. = 99—101°C. For $C_{11}H_{13}NO_3$ (M_r = 207.23) w_i(calc.): 63.75% C, 6.32% H, 6.75% N; w_i(found): 63.59% C, 6.45% H, 6.78% N. UV spectrum, λ_{max}/nm (log ε): 265 (2.51). ^{1}H NMR spectrum, δ: 5.96 (s, 1H, H-4'), 5.21 (d, d, 1H, H-6a) $J_{3a,6a}$ = = 8.0 Hz, $J_{6,6a}$ = 3.5 Hz, 3.57—4.32 (m, 5H, H-3a, H_2-4, H_2-6), 2.37 (s, 3H, CH_3), 2.21 (s, 3H, CH_3).

3-(2,5-Dimethyl-3-furyl)-3,5,10-trioxa-9-azabicyclo[5,3,0]decane (IX), yield = 83%, b.p. = 100—110°C (p = 6.66 Pa). For $C_{13}H_{15}NO_4$ (M_r = 249.28) w_i(calc.): 62.63% C, 6.07% H, 5.61% N; w_i(found): 62.90% C, 6.21% H, 5.74% N. ^{1}H NMR spectrum, δ: 6.02 (s, 1H, H-4'), 3.76—4.95 (m, 8H, H-1, H_2-2, H_2-4, H_2-6, H-7), 2.44 (s, 3H, CH_3), 2.24 (s, 3H, CH_3). ^{13}C NMR spectrum, δ: 152.06, 150.24, 149.89 (s, s, s, C-8, C-2', C-5'), 109.64 (s, C-3'), 104.90 (d, C-4'), 98.12 (t, C-4), 81.73 (d, C-1), 68.75 (t, C-2), 66.70 (t, C-6), 53.07 (d, C-7), 13.17 (q, CH_3), 12.70 (q, CH_3).

3-(2,5-Dimethyl-3-furyl)-3a,4,9,9a-tetrahydro-4,9-epoxynaphthaleno[3,2-d]isoxazole (X), yield = 78%, m.p. = 123—124°C. For $C_{17}H_{15}NO_3$ (M_r = 281.30) w_i(calc.): 72.58% C, 5.38% H, 4.97% N; w_i(found): 72.27% C, 5.15% H, 5.18% N. UV spectrum, λ_{max}/nm (log ε): 265 (2.81). ^{1}H NMR spectrum (deuterated DMSO), δ: 7.12—7.35 (m, 4H, H_{arom}), 6.29 (s, 1H, H-4'), 5.48 (s, 1H, H-9), 5.39 (s, 1H, H-4), 4.80 (d, 1H, H-9a) $J_{3a,9a}$ = 7.8 Hz, 3.88 (d, 1H, H-3a), 2.36 (s, 3H, CH_3), 2.17 (s, 3H, CH_3). ^{13}C NMR spectrum (deuterated DMSO), δ: 150.50, 149.94, 149.28 (s, s, s, C-3, C-2', C-5'), 145.49, 141.96 (s, s, C_{arom}), 127.88, 127.32, 121.38, 120.31 (doublets, C_{arom}), 110.62 (s, C-3'), 106.04 (d, C-4'), 86.28 (d, C-9), 85.16 (d, C-4), 80.68 (d, C-9a), 59.37 (s, C-3a), 13.50 (q, CH_3), 12.83 (q, CH_3).

Dimethyl 7-(2,5-dimethyl-3-furyl)-8-aza-9,10-dioxatricyclo[4,3,0,$1^{2,5}$]decane-3,4-dicarboxylate (XI), yield = 45%, m.p. = 129—130°C. For $C_{17}H_{19}NO_7$ (M_r = 349.31) w_i(calc.): 57.73% C, 5.48% H, 4.00% N; w_i(found): 57.62% C, 5.39% H, 3.92% N. UV spectrum, λ_{max}/nm (log ε): 268 (2.49). ^{1}H NMR spectrum, δ: 6.02 (s, 1H, H-4'), 5.05 (s, 1H, H-2), 4.90 (s, 1H, H-5), 4.79 (d, 1H, H-1), 3.76 (d, 1H, H-6) $J_{1,6}$ = 7.8 Hz, 3.67, 3.68 (s, s, 6H, 2 × $COOCH_3$), 3.15 (d, 1H, H-3) $J_{3,4}$ = 9.6 Hz, 3.01 (d, 1H, H-4), 2.41 (s, 3H, CH_3), 2.25 (s, 3H, CH_3). ^{13}C NMR spectrum, δ: 170.35, 170.30 (s, s, 2 × $COOCH_3$), 158.87, 158.20 (s, s, C-2', C-5'), 149.61 (s, C-7), 109.65 (s, C-3'), 105.19 (d, C-4'), 84.47 (d, C-2), 84.13 (d, C-5), 79.69 (d, C-1), 59.91 (q, OCH_3), 52.33 (d, C-3), 50.04 (d, C-4), 46.65 (d, C-6), 13.89 (q, CH_3), 13.21 (q, CH_3).

Dimethyl 5-(2,5-dimethyl-3-furyl)-10-(diphenylmethylene)-3-oxa-4-azatricyclo-[5,2,1,$0^{2,6}$]deca-4-ene-8,9-dicarboxylate (XII), yield = 91%, m.p. = 164—166°C. For $C_{31}H_{27}NO_6$ (M_r = 509.51) w_i(calc.): 73.07% C, 5.33% H, 2.74% N; w_i(found): 72.94% C, 5.54% H, 3.01% N. UV spectrum, λ_{max}/nm (log ε): 245 (3.04). ^{1}H NMR spectrum (deuterated DMSO), δ: 6.80—7.27 (m, 10H, H_{arom}), 5.95 (s, 1H, H-4′), 4.81 (d, 1H, H-2) $J_{2,6}$ = 8.7 Hz, 4.10 (d, 1H, H-6), 3.52 (s, 6H, 2 × $COOCH_3$), 3.36 (m, 2H, H-8, H-9), 3.08 (d, 1H, H-1) $J_{1,9}$ = 3.9 Hz, 2.75 (d, 1H, H-7), 2.22 (s, 3H, CH_3), 2.07 (s, 3H, CH_3). ^{13}C NMR spectrum (deuterated DMSO), δ: 151.82, 150.33, 149.88 (s, s, s, C-5, C-2′, C-5′), 149.91, 140.54, 139.05, 134.49 (singlets, C_{arom}, C-10, C-11), 129.20, 128.80, 128.28, 127.88, 127.41, 127.13 (doublets, C_{arom}), 110.72 (s, C-3′), 105.73 (d, C-4′), 81.14 (d, C-2), 53.42 (d, C-6), 52.09 (q, $COOCH_3$), 51.87 (q, $COOCH_3$), 47.22, 44.33, 43.53, 42.95 (doublets, C-9, C-8, C-7, C-1), 13.74 (q, CH_3), 13.19 (q, CH_3).

Dimethyl 3-(2,5-dimethyl-3-furyl)isoxazole-4,5-dicarboxylate (XVI), yield = 75%, m.p. = 74—76°C. For $C_{13}H_{13}NO_6$ (M_r = 279.22) w_i(calc.): 55.93% C, 4.68% H, 5.01% N; w_i(found): 55.92% C, 4.64% H, 5.05% N. UV spectrum, λ_{max}/nm (log ε): 218 (2.38). ^{1}H NMR spectrum (deuterated acetone), δ: 6.07 (s, 1H, H-4′), 3.85 (s, 3H, $COOCH_3$), 3.79 (s, 3H, $COOCH_3$), 2.30 (s, 3H, CH_3), 2.17 (s, 3H, CH_3). ^{13}C NMR spectrum, δ: 161.17, 158.73, 156.11, 155.45, 150.78 (singlets, C-3, C-4, C-5, 2 × $COOCH_3$), 151.09, 151.01 (s, s, C-2′, C-5′), 107.75 (s, C-3′), 106.04 (d, C-4′), 53.86 (q, OCH_3), 53.51 (q, OCH_3), 13.40 (q, CH_3), 13.25 (q, CH_3).

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Translated by P. Zálupský