1,3-Dipolar cycloadditions of heterocycles XIV.* Preparation and photochemistry of 3-aryl-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazoles

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1,3-Dipolar cycloaddition of substituted benzonitrile oxides, where substituents were H, 4-CH_3, 4-OCH_3, 2-OCH_3, 4-F, 2-F, 4-Cl, 4-NO_2, 3-NO_2, and 4-Br, to 5*H*-furan-2-one takes place totally regioselectively to give 3-aryl-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazoles. Irradiation of the prepared isoxazoles furnished only polymeric material.

Установлено, что 1,3-диполярное циклоприсоединение замещенных бензонитрилоксидов, где заместителями являются H, 4-CH_3, 4-OCH_3, 2-OCH_3, 4-F, 2-F, 4-Cl, 4-NO_2, 3-NO_2 и 4-Br, к 5*H*-фуран-2-ону проходит полностью региоселективно и приводит к образованию 3-арил-4-оксо-3a,4,6,6a-тетрагидрофуро[3,4-*d*]изоксазолов. В результате облучения полученных изоксазолов образовывались исключительно полимерные продукты.

Our previous papers on the photochemistry of condensed isoxazolines described their unusually straightforward photorearrangement [1—6]. 3-Aryl-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazoles (*II*) formed by 1,3-dipolar cycloaddition of substituted benzonitrile oxides (*I*, Scheme 1) to 2,5-dihydrofuran rearranged upon irradiation to 4-aryl-5-formyl-2,3-dihydro-6*H*-1,3-oxazines (*III*) [1, 2, 6]. Evidence gathered sofar indicated that the course and selectivity of the photorearrangement were strongly structure dependent [7—12]. It was therefore of interest to learn the behaviour of the tetrahydrofuran ring containing the carbonyl group as in *IV*.

Despite numerous references to cycloadditions of nitrile oxides [13] there is only one short communication describing the cycloaddition of the parent benzonitrile oxide to 5*H*-furan-2-one [14]. Metelli and Betinetti obtained only one head to tail

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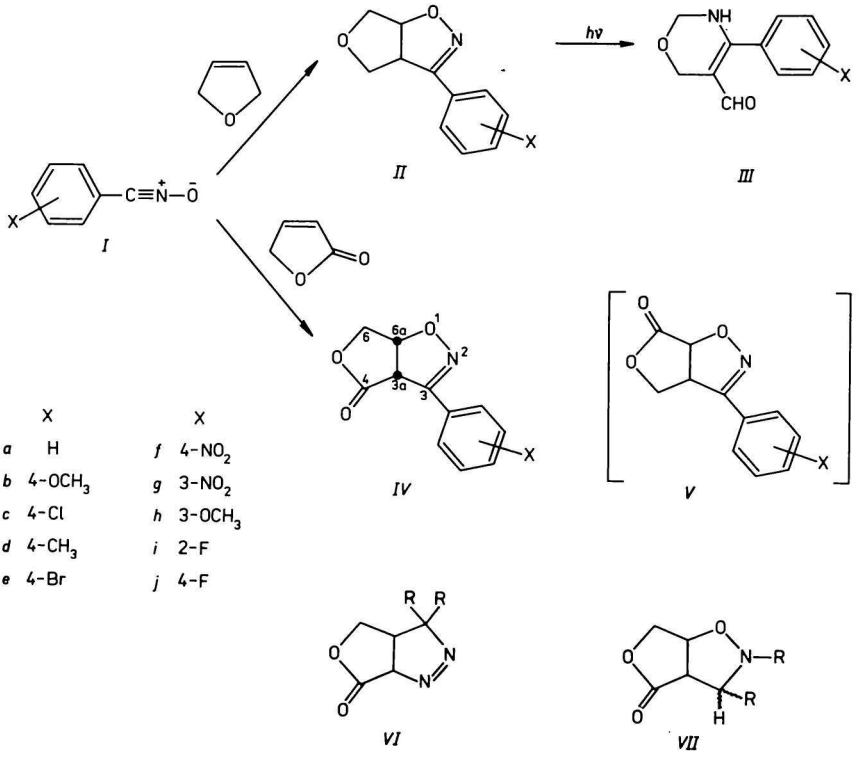
regioisomer 3-phenyl-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVa*) in 54 % yield. Only scarce information on the dipolarophilic reactivity of 5*H*-furan-2-one [15, 16] prompted us to study its cycloadditions with substituted benzonitrile oxides in detail.

The requisite nitrile oxides were generated *in situ* by a novel method, starting from the corresponding oximes, sodium hypochlorite, and a catalytic amount of triethylamine in two-phase (H_2O/CH_2Cl_2) system [17]. In principle two regioisomeric cycloadducts can arise, the respective head to head (*V*) and head to tail (*IV*) orientations being judged according to the relative position of the isoxazoline oxygen and the carbonyl group. In all instances only the type — head to tail adducts *IV* — has been isolated. The structure assignment was performed based on the comparison of spectral data with those of the model compound 3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*II*). Thus signals of the bridgehead protons of *IIb* were found at $\delta = 5.29$ ppm (H-6a) and at 3.62—4.32 ppm (H-3a), the corresponding carbons showed signal at $\delta = 85.95$ ppm (C-6a) and 54.12 ppm (C-3a) in ^1H and ^{13}C NMR spectra, respectively [6]. Compound isolated from the reaction of *Ib* with 5*H*-furan-2-one displayed signals of the bridgehead protons in the same region, $\delta = 5.60$ ppm (H-6a) and 4.58 ppm (H-3a); 82.25 ppm (C-6a) and 54.18 ppm (C-3a). Moreover signal of bridgehead proton with the highest δ values, ascribed to the proton in the vicinity of isoxazoline oxygen, showed a d, d, d multiplicity, indicating for 3-(4-methoxyphenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVb*) a head to tail structural arrangement. Optional head to head regioisomer would have to have for the corresponding proton a doublet at higher δ value, the proton being deshielded by two acceptor groups. However, regioisomer *IV* was formed exclusively with no discoverable quantity of *V*. Coupling constant for the protons at ring junction $J_{3a,6a} = 6$ Hz indicated *cis* orientation, documenting the *cis* stereospecificity of 1,3-dipolar cycloaddition of nitrile oxides to 5*H*-furan-2-one as well. Isoxazoline and lactone moiety asserted themselves by the typical spectral patterns in UV and IR spectra ($\nu(C=O) = 1788\ cm^{-1}$), respectively.

In the same manner were prepared adducts of the substituted benzonitrile oxides and 5*H*-furan-2-one (*IVa*, *IVd*—*IVj*) (Scheme 1). Like in the case of *Ib*, only one head to tail isomer *IV* was formed, in agreement with the already published data. Both diazoalkanes [15] and nitrones [16] attach to the β-position of the α,β-unsaturated system with the atom possessing the greatest atomic orbital coefficient (adducts *VI* and *VII*). Compound *IVc* (4-Cl) had to be prepared by classical procedure using 4-chlorobenzhydroximoyl chloride and triethylamine. Attempted synthesis directly from oxime by the described procedure produced always an inseparable mixture of *IVc* and unreacted 4-chlorobenzaldehyde oxime. Yields were in the range 21—56 %, calculated from isolated product. Reaction mixtures contained in all cases beside cycloadduct the starting oxime and dimers of nitrile



X		X	
a	H	*f*	4-NO_2
b	4-OCH_3	*g*	3-NO_2
c	4-Cl	*h*	3-OCH_3
d	4-CH_3	*i*	2-F
e	4-Br	*j*	4-F

Scheme 1

oxide as well. Structure of *IVc—IVj* was established in the same manner as above. Presence of substituent did not influence markedly signal positioning in ^1H and ^{13}C NMR spectra. Measured in deuteriochloroform signals of H-3a and H_2-6 protons almost coincided with the residual signal of the solvent, in deuterated dimethyl sulfoxide signals of H-3a experienced a marked shift, *e.g.* for *IVd* from $\delta = 4.59$ ppm to 5.02 ppm. In contrast to mass spectra of *II*, where cycloreversion was the dominant pattern, adducts *IV* failed to display cycloreversion fragments.

Irradiation of *IVa* in acetonitrile ($c = 5 \times 10^{-5}$ mol dm^{-3}) by the light with $\lambda_{max} = 253.7$ nm caused dwindling of its concentration, easily monitored by repeated scans of the UV spectrum. The intensity of absorption maximum at $\lambda = 264$ nm decreased steadily, forming at first two isosbestic points at $\lambda = 292$ nm and 238 nm (Fig. 1). Further irradiation decomposed the primary photoproduct which despite great effort and utilizing all available separation methods escaped isolation as pure substance. Spectra of the reaction mixture only showed signals of aromatic protons. Irradiation of *IV* in methanol followed a different course. Thus

irradiation of *IVd* produced a new, prominent maximum at $\lambda = 230$ nm in its UV spectrum (Fig. 2). Further irradiation and preparative experiments failed to yield an isolable product as well.

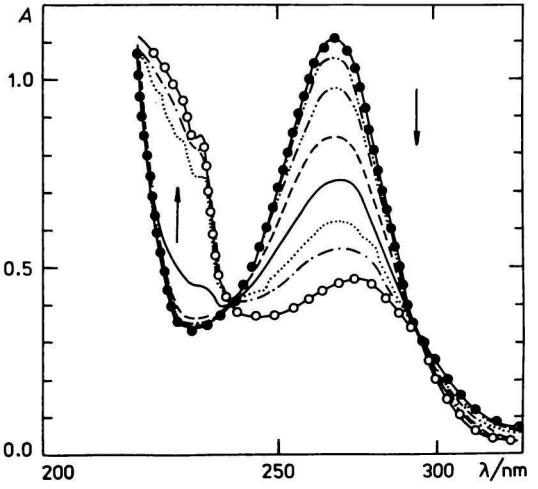


Fig. 1. Photoreaction of *IVa* in acetonitrile; irradiated with monochromatic light, $\lambda = 253.7$ nm, $c = 5 \times 10^{-5}$ mol dm^{-3}, in a 0.02 m thick cuvette, volume 34 cm^3 for t/min: 0, 2.5, 6, 12, 20, 30, 40, and 85.

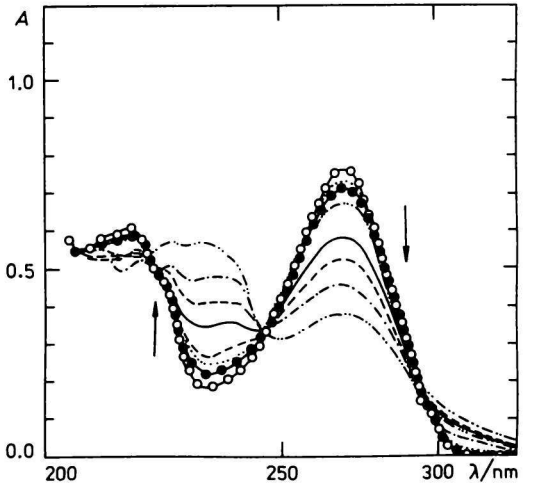


Fig. 2. Photoreaction of *IVd* in methanol; irradiated with monochromatic light, $\lambda = 253.7$ nm, $c = 5 \times 10^{-4}$ mol dm^{-3}, in a 0.001 m thick cuvette, volume 10 cm^3 for t/min: 0, 10, 20, 37, 80, 120, 180, and 260.

Quantitative measurements were done from the concentration decrease of the starting compounds. In acetonitrile the following quantum yields (Φ) values were measured

Derivative	*IVa* (H)	*IVd* (4-CH_3)	*IVj* (4-F)
Φ	0.014	0.008	0.010

All sofar measured quantum yields were higher in acetonitrile than in methanol [2—6]. In case of adducts *IV* the situation was reversed, indicating a different reaction type at the beginning. In methanol the following values were measured

Derivative	*IVb* (4-OCH_3)	*IVd* (4-CH_3)	*IVj* (4-F)
Φ	0.005	0.016	0.035

Based on similar spectral changes observed during the irradiation of *IV* and during the conversion of $II \xrightarrow{h\nu} III$, we have assumed that similar processes were at work, producing in case of *IV* labile photoproducts. Irradiation with longer wavelength light ($\lambda_{max} = 364$ nm), absorbed by the carbonyl chromophore surprisingly failed to initiate any photoreaction.

In conclusion we may say that the introduction of the carbonyl group in the tetrahydrofuran skeleton (derivatives *IV*) lowers the quantum yields as compared with photoconversion $II \xrightarrow{h\nu} III$, *e.g.* for *IIa* $\Phi = 0.04$, for *IVa* $\Phi = 0.014$ and destabilizes the primary photoproducts.

Experimental

Melting points are uncorrected. NMR spectra were recorded with a Tesla BS 487 C ^1H and Jeol XF-100 ^{13}C spectrophotometers in deuteriochloroform with hexamethyldisiloxane as internal standard, reported in δ/ppm. UV spectra were measured with a Perkin—Elmer model 323 with temperature control, in methanol. Absorption coefficients are reported in $m^2\ mol^{-1}$. Progress of irradiation was followed by TLC on Silufol. Irradiation experiments were done with a Toshiba GL-15 low-pressure Hg lamp in quartz sleeve, immersed in a 300 cm^3 reaction vessel [18] with forced circulation of solvent kept at 15 °C during the reaction. In preparative experiments the reaction mixtures were after the irradiation period concentrated on a rotary vacuum evaporator and separated on a silica gel column, with hexane—ethyl acetate mixture as eluant. Solvents were purified by conventional methods. Quantum yields at $\lambda = 253.7$ nm were determined with an apparatus described in [19]. Concentration decrease of *IV* was monitored by measuring the absorbance of the maximum at $\lambda \approx 265$ nm. 5*H*-Furan-2-one was prepared according to [20], distilled previous to the reaction. Isoxazolines were synthesized by the action of sodium hypochlorite, triethylamine, and the corresponding oxime on the 5*H*-furan-2-one [17].

1,3-Dipolar cycloadditions of substituted benzonitrile oxides to 5H-furan-2-one

To the mixture of 5*H*-furan-2-one (3.5 g; 42 mmol), triethylamine (0.2 g; 1.98 mmol), substituted benzaldehyde oxime (21 mmol), and dichloromethane (30 cm^3), stirred and kept at 0 °C was added a 11 % solution of sodium hypochlorite (20 cm^3; 2.5 g; 34 mmol). The mixture was stirred at 0 °C for an additional hour and left to stand overnight at ambient temperature. After the separation of layers the aqueous layer was extracted with dichloromethane (3 × 20 cm^3), combined organic portions were dried by $MgSO_4$, concentrated *in vacuo*, the product being isolated either by column chromatography or by crystallization.

3-(4-Methoxyphenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVb*) prepared from *Ib* in 52 % yield, m.p. = 135—137 °C (from chloroform—hexane). For $C_{12}H_{11}NO_4$ (M_r = 233.22) w_i(calc.): 61.80 % C, 4.75 % H, 6.01 % N; w_i(found): 62.03 % C, 5.00 % H, 5.93 % N. UV spectrum, λ_{max}/nm (log {ε}): 278 (3.11). IR spectrum, $\tilde{\nu}$/cm^{-1}: 1788 (C=O). ^{1}H NMR, δ/ppm: 7.79—7.93 and 6.89—7.01 (m, 4H, aromatic protons), 5.60 (d, d, d, 1H, H-6a), 4.62 (m, 2H, H_2-6), 4.58 (d, 1H, $J_{3a,6a}$ = 6.0 Hz, H-3a), 3.83 (s, 3H, OCH_3). ^{13}C NMR, δ/ppm: 171.01 (s, C=O), 161.65, 129.55, 114.22, 119.42 (aromatic carbons), 152.04 (s, C=N), 82.25 (d, C-6a), 73.42 (t, C-6), 54.18 (d, C-3a).

3-(4-Chlorophenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVc*) prepared from *Ic* by classical procedure *via* 4-chlorobenzhydroximoyl chloride according to [21], yield = 40 %, m.p. = 141—143 °C (from chloroform—hexane). For $C_{11}H_8ClNO_3$ (M_r = 237.64) w_i(calc.): 55.59 % C, 3.39 % H, 5.89 % N; w_i(found): 55.62 % C, 3.32 % H, 5.74 % N. IR spectrum, $\tilde{\nu}$/cm^{-1}: 1780 (C=O). UV spectrum, λ_{max}/nm (log {ε}): 269 (3.20). ^{1}H NMR, δ/ppm: 7.82—7.96 and 7.34—7.45 (m, 4H, aromatic protons), 5.62 (d, d, d, 1H, H-6a), 4.65 (m, 2H, H_2-6), 4.57 (d, 1H, $J_{3a,6a}$ = 7.0 Hz, H-3a). ^{13}C NMR spectrum: 170.61 (s, C=O), 151.65 (s, C=N), 137.09, 129.29, 128.38, 125.66 (aromatic carbons), 82.77 (d, C-6a), 73.55 (t, C-6), 53.92 (d, C-3a).

3-(4-Methylphenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVd*) prepared from *Id* in 56 % yield, m.p. = 155—156 °C (from chloroform—hexane). For $C_{12}H_{11}NO_3$ (M_r = 217.22) w_i(calc.): 66.35 % C, 5.10 % H, 6.45 % N; w_i(found): 66.29 % C, 4.99 % H, 6.69 % N. UV spectrum, λ_{max}/nm (log {ε}): 268 (3.08). IR spectrum, $\tilde{\nu}$/cm^{-1}: 1788 (C=O). ^{1}H NMR, δ/ppm: 7.78—7.87 and 7.20—7.27 (m, 4H, aromatic protons), 5.63 (d, d, d, 1H, H-6a), 4.67 (m, 2H, H_2-6), 4.59 (d, 1H, $J_{3a,6a}$ = 8 Hz, H-3a), 2.39 (s, 3H, CH_3); δ/ppm in deuterated dimethyl sulfoxide: 7.65—7.75 and 7.18—7.28 (m, 4H, aromatic protons), 5.57 (d, d, d, 1H, H-6a), 5.02 (d, 1H, $J_{3a,6a}$ = 8 Hz, H-3a), 4.56 (m, 2H, H_2-6), 2.31 (s, 3H, CH_3). ^{13}C NMR spectrum, δ/ppm: 170.75 (s, C=O), 152.42 (s, C=N), 141.25, 129.55, 127.86, 124.10 (aromatic carbons), 82.25 (d, C-6a), 73.35 (t, C-6), 54.05 (d, C-3a).

3-(4-Bromophenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVe*) prepared from *Ie* in 21 % yield, m.p. = 158—159 °C (from chloroform—hexane). For $C_{11}H_8BrNO_3$ (M_r = 282.09) w_i(calc.): 46.83 % C, 2.86 % H, 4.96 % N; w_i(found): 46.89 % C, 2.99 % H, 5.16 % N. UV spectrum, λ_{max}/nm (log {ε}): 270 (3.17). ^{1}H NMR, δ/ppm: 7.75—7.87 and 7.50—7.62 (m, 4H, aromatic protons), 5.65 (d, d, d, 1H, H-6a), 4.65 (m, 2H, H_2-6), 4.57 (d, $J_{3a,6a}$ = 8 Hz, H-3a). ^{13}C NMR, δ/ppm: 170.50 (s, C=O), 132.06,

129.34, 127.52, 125.44 (aromatic carbons), 82.72 (d, C-6a), 73.37 (t, C-6), 53.76 (d, C-3a).

3-(4-Nitrophenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVf*) prepared from *If* in 29 % yield, m.p. = 190—192 °C (from chloroform—hexane). For $C_{11}H_8N_2O_5$ (M_r = 248.19) w_i(calc.): 53.23 % C, 3.25 % H, 11.29 % N; w_i(found): 53.05 % C, 3.17 % H, 11.00 % N. UV spectrum, λ_{max}/nm (log {ε}): 222 (2.75), 305 (2.99). ^{1}H NMR, δ/ppm: 8.06—8.37 (m, 4H, aromatic protons), 5.75 (d, d, d, 1H, H-6a), 4.72 (m, 2H, H_2-6), 4.65 (d, 1H, $J_{3a,6a}$ = 7.5 Hz, H-3a). ^{13}C NMR, δ/ppm (deuterated dimethyl sulfoxide): 172.19 (s, C=O), 152.97 (s, C=N), 149.60, 134.66, 129.60, 125.96, 124.53 (aromatic carbons), 85.70 (d, C-6a), 74.28 (t, C-6), 54.15 (d, C-3a).

3-(3-Nitrophenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVg*) prepared from *Ig* in 27 % yield, m.p. = 170—172 °C (from chloroform—hexane). For $C_{11}H_8N_2O_5$ (M_r = 248.19) w_i(calc.): 53.23 % C, 3.25 % H, 11.29 % N; w_i(found): 52.96 % C, 3.12 % H, 11.25 % N. UV spectrum, λ_{max}/nm (log {ε}): 259 (3.19), 372 (sh, 2.56). IR spectrum, $\tilde{\nu}$/cm^{-1}: 1773 (C=O). Mass spectrum: *m/z* = 248 ($M^{+\cdot}$), 189 (base peak). ^{1}H NMR spectrum, δ/ppm (deuterated dimethyl sulfoxide): 7.06—8.67 (m, 4H, aromatic protons), 5.70 (d, d, d, 1H, H-6a), 5.22 (d, d, 1H, H-3a), 4.62 (m, 2H, H_2-6). ^{13}C NMR, δ/ppm (deuterated dimethyl sulfoxide): 171.93 (s, C=O), 152.06 (s, C=N), 148.04, 133.75, 130.64, 128.95, 125.05, 122.20 (aromatic carbons), 84.41 (d, C-6a), 73.50 (t, C-6), 53.50 (d, C-3a).

3-(3-Methoxyphenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVh*) prepared from *Ih* in 45 % yield, m.p. = 105—107 °C (from chloroform—hexane). For $C_{12}H_{11}NO_4$ (M_r = 233.22) w_i(calc.): 61.80 % C, 4.75 % H, 6.01 % N; w_i(found): 61.86 % C, 4.77 % H, 5.93 % N. UV spectrum, λ_{max}/nm (log {ε}): 267 (2.95), 305 (2.43). IR spectrum, $\tilde{\nu}$/cm^{-1}: 1788 (C=O). ^{1}H NMR, δ/ppm: 6.92—7.60 (m, 4H, aromatic protons), 5.65 (d, d, d, 1H, H-6a), 4.64 (m, 2H, H_2-6), 4.57 (d, 1H, $J_{3a,6a}$ = 7.5 Hz, H-3a), 3.84 (s, 3H, OCH_3). ^{13}C NMR, δ/ppm: 170.61 (s, C=O), 159.70, 129.82, 128.12, 120.72, 117.47, 112.27 (aromatic carbons), 152.43 (s, C=N), 82.51 (d, C-6a), 73.29 (t, C-6), 53.92 (d, C-3a), 29.62 (q, OCH_3).

3-(2-Fluorophenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVi*) prepared from *Ii* in 32 % yield, m.p. = 109—111 °C (from chloroform—hexane). For $C_{11}H_8FNO_3$ (M_r = 221.18) w_i(calc.): 59.73 % C, 3.64 % H, 6.33 % N; w_i(found): 59.89 % C, 3.71 % H, 6.50 % N. UV spectrum, λ_{max}/nm (log {ε}): 262 (2.98). ^{1}H NMR, δ/ppm: 7.02—7.95 (m, 4H, aromatic protons), 5.63 (d, d, d, 1H, H-6a), 4.85 (d, 1H, $J_{3a,6a}$ = 9.0 Hz, H-3a), 4.64 (m, 2H, H_2-6). ^{13}C NMR, δ/ppm: 170.69 (s, C=O), 169.27, 133.20, 130.46, 125.0, 117.57, 116.14, (aromatic carbons), 152.34 (s, C=N), 82.55 (d, C-6a), 73.43 (t, C-6), 54.42 (d, C-3a).

3-(4-Fluorophenyl)-4-oxo-3a,4,6,6a-tetrahydrofuro[3,4-*d*]isoxazole (*IVj*) prepared from *Ij*, in 55 % yield, m.p. = 121—122 °C (from chloroform—hexane). For $C_{11}H_8FNO_3$ (M_r = 221.18) w_i(calc.): 59.73 % C, 3.64 % H, 6.33 % N; w_i(found): 59.84 % C, 3.72 % H, 6.41 % N. UV spectrum, λ_{max}/nm (log {ε}): 264 (3.02). IR spectrum, $\tilde{\nu}$/cm^{-1}: 1788 (C=O). Mass spectrum: *m/z* = 221 ($M^{+\cdot}$), (base peak). ^{1}H NMR, δ/ppm: 7.87—8.01 and 7.03—7.27 (m, 4H, aromatic protons), 5.65 (d, d, d, 1H, H-6a), 4.67 (m, 2H, H_2-6), 4.60 (d, 1H, $J_{3a,6a}$ = 8 Hz, H-3a). ^{13}C NMR, δ/ppm: 170.75 (s, C=O), 172.57 (s, C—F), 155.80, 130.34, 129.81, 116.82, 115.39 (aromatic carbons), 151.51 (s, C=N), 82.65 (d, C-6a), 73.42 (t, C-6), 54.06 (d, C-3a).

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