Reaction of Isonitriles with Dibenzoylacetylene Leading to Furan and Difuropyran Derivatives

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A mixture of an isocyanide and dibenzoylacetylene in dry CH_2Cl_2 undergoes a smooth addition reaction at ambient temperature to furnish 3-[5-(alkylimino)-3,4-dibenzoyl-2-phenylfuran-2(2*H*)-yl]-1-phenylprop-2-yn-1-ones (1:2 adduct) and {2,5-bis(alkylimino)-4,7,8a-triphenyl-5*H*-difuro[2,3-*b*:3′,4′-*e*]-pyran-3(8a*H*)-yl}(phenyl)methanones (2:2 adduct). Single-crystal X-ray analyses conclusively confirmed the structures of the adducts.

Introduction. – It has been known from the studies of various groups that addition of nucleophiles devoid of acidic H-atoms such as Ph_3P, pyridine, amines, DMSO, phosphoranes, and isocyanides to electron-deficient acetylenic compounds leads to a 1:1 zwitterionic intermediate [1]. Isocyanides, however, differ in their reactivity profile due to the presence of the divalent C-atom and its pronounced reactivity towards electrophilic C(sp or sp^2) centers. In transformations involving these entities, this divalent C-atom is converted to a tetravalent state, which renders the addition irreversible. It may be recalled that this property of isocyanides has been crucial in the successful development of classical multicomponent reactions (MCRs) like *Passerini* 3CR and *Ugi* 4CR [2].

Winterfeldt et al. had observed that isocyanides readily add to dimethyl acetylenedicarboxylate (DMAD) [1g]. The initially formed 1:1 zwitterionic intermediate (*Fig. 1*) can undergo further reaction with DMAD and isocyanide in different molar proportions leading to various adducts. Subsequently, *George* and co-workers have established the structures of several adducts by single-crystal X-ray-analysis [3].

Furans and their reduced forms are common structural motifs in naturally occurring compounds such as pheromones, polyether antibiotics, and furano-terpenes [4–6]. Moreover, they are useful building blocks in the total synthesis of natural products and pharmaceuticals. Some furan derivatives have been shown to possess antitumor and cytotoxic, antimicrobial, antispasmodic, anti-inflammatory, cholesterol-acyltransferase

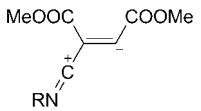
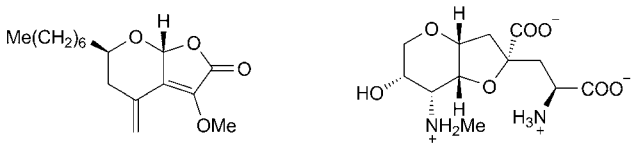


Fig. 1. *The* Winterfeldt *zwitterionic intermediate*

inhibitory, and several other biological activities [7]. Furthermore, furan heterocycles are found in synthetic materials, such as agrochemical bioregulators, essential oils, cosmetics, dyes, photosensitizers, flavoring and fragrance compounds, textiles and fibers, surface active agents and detergents, fire retardants, and polymer materials, and in fossil fuel industry [5b].

Furopyrans, 5-6 bicyclic systems with one O-atom in each ring, consist of a furan ring attached to a pyran ring. These fused heterocyclic compounds have attracted attention, because they constitute an important class of natural and non-natural products, many of which exhibit biological activities. Myxostiolide has been isolated from the fungus *Myxotrichum stipitatum* and serves as a plant-growth regulator, and dysiherbaine acts as a selective agonist of non-NMDA-type glutamate receptors in the central nervous system (*Fig. 2*) [8].

So far, the most common synthetic routes reported for the preparation of furo[2,3-*b*]pyran and furo[3,4-*b*]pyran ring systems involve syntheses of the rings *i*) from acyclic compounds, *ii*) by formation of the six-membered ring, and *iii*) by formation of the five-membered ring [9].



Myxostiolide Dysiherbaine

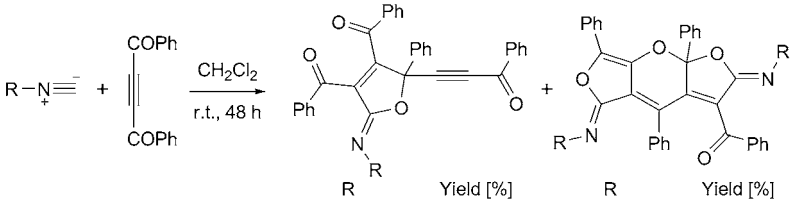
Fig. 2. *Examples of biologically active furopyrans*

Results and Discussion. – To the best of our knowledge, there is no report in the literature on the synthesis of the difuro[2,3-*b*:*3′,4′-e*]pyran ring system. As part of our current studies on the development of new efficient strategies for the preparation of interesting bioactive molecules and drug cores [10], herein, we report a reaction leading to dihydrofurans and difuro[2,3-*b*:*3′,4′-e*]pyrans. Thus, a mixture of an isocyanide **1** and dibenzoylacetylene (**2**) underwent a smooth addition reaction in dry CH_2Cl_2. The reaction proceeded at ambient temperature and went to completion within 48 h to afford 3-[5-(alkylimino)-3,4-dibenzoyl-2-phenylfuran-2(2*H*)-yl]-1-phenylprop-2-yn-1-ones (**3**; 1:2 adduct) and {2,5-bis(alkylimino)-4,7,8a-triphenyl-5*H*-difuro[2,3-*b*:*3′,4′-e*]pyran-3(8a*H*)-yl}(phenyl)methanones (**4**; 2:2 adduct) (*Scheme 1*). TLC and ^{1}H-NMR analysis of the reaction mixtures clearly indicated the formation of only **3** and **4** (see *Exper. Part*)[1]).

The structures of the isolated products **3** and **4** were deduced from ^{1}H- and ^{13}C-NMR spectroscopy. The ^{1}H-NMR spectrum of **3b** showed a *singlet* (δ(H) 1.41) readily recognized as arising from the ^{t}Bu group, as well as characteristic signals with appropriate chemical shifts and coupling constants for the 20 aromatic H-atoms. The ^{1}H-decoupled ^{13}C-NMR spectrum of **3b** exhibited characteristic signals at 29.8 and 55.4 (^{t}Bu), 60.1 (PhC–O), 86.4 and 87.2 (2 sp C-atoms), 188.7 and 189.8 (2 benzoyl C=O),

[1]) The best results were obtained when the two reactants were reacted in a 1:1.4 ratio.

Scheme 1. *Reaction between Isocyanides and Dibenzoylacetylene*



	R	Yield [%]
3a	Cyclohexyl	36
3b	^{t}Bu	43

	R	Yield [%]
4a	Cyclohexyl	44
4b	^{t}Bu	47

1 **2**

and 177.0 arising from the acetylenic conjugated C=O group. The signals of other twelve CH and seven quaternary C-atoms were observed with appropriate chemical shifts in the downfield region of the spectrum, in agreement with the proposed structure (see *Exper. Part*).

The ^{1}H-NMR spectrum of **4b** showed two single sharp lines (δ(H) 1.26 and 1.29), attributed to two ^{t}Bu groups along with characteristic signals with appropriate chemical shifts and coupling constants for the 20 aromatic H-atoms. The ^{1}H-decoupled ^{13}C-NMR spectrum of **4b** exhibited characteristic signals at δ(C) 30.06, 30.12, 55.1, and 55.7 (2 ^{t}Bu), 108.1 (O–C–O), and 190.4 (C=O). Signals of twelve CH and twelve quaternary C-atoms with appropriate chemical shifts were observed in the downfield region of the spectrum, in agreement with the proposed structure (see *Exper. Part*).

Single-crystal X-ray analyses of **3a** and **4a** confirmed conclusively the structures of the isolated products. ORTEP Diagrams of **3a** and **4a** are shown in *Figs. 3* and *4*, respectively[2])[3]).

A plausible mechanism for the formation of the 1:2 adduct is proposed in *Scheme 2*. The reactive 1:1 zwitterionic intermediate **5**, generated *in situ* from the reaction between **1** and dibenzoylacetylene (**2**), may attack the C=O group of another molecule **2** to give the isonitrilium alkoxide intermediate **6**. The latter may undergo an intramolecular nucleophilic addition of the alkoxide to furnish the isolated imino γ-lactone **3**.

Winterfeldt et al. reported a similar structure for the 1:2 adduct of an isocyanide **1** (R = cyclohexyl and R = ^{t}Bu) and DMAD (**7**). In the proposed mechanism, the reactive 1:1 zwitterionic intermediate **8** is trapped by another molecule of **7** to directly

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[2]) *Selected X-ray crystallographic data for compound* **3a**: $C_{39}H_{31}NO_4$, triclinic, space group = $P\bar{1}$, a = 10.130(3) Å, b = 10.864(4) Å, c = 14.938(5) Å, α = 93.368(6), β = 95.270(5), γ = 109.139(5), V = 1539.6(9) Å^3, T = 295(2) K, Z = 2, D_{calc} = 1.246 g·cm^{-3}, μ = 0.080 mm^{-1}, 1945 observed reflections, final R_1 = 0.086, wR_2 = 0.189 and for all data R_1 = 0.239, wR_2 = 0.270. CCDC-852555 contains the corresponding supplementary crystallographic data. These data can be obtained free of charge from the *Cambridge Crystallographic Data Centre via* www.ccdc.cam.ac.uk/data_request/cif.

[3]) *Selected X-ray crystallographic data for compound* **4a**: $C_{46}H_{42}N_2O_4$, monoclinic, space group = $P2_1/c$, a = 9.6730(17) Å, b = 10.8199(19) Å, c = 39.7244(70) Å, β = 93.601(3)°, V = 4149.38(15) Å^3, T = 295(2) K, Z = 4, D_{calc} = 1.23 g·cm^{-3}, μ = 0.202 mm^{-1}, 3080 observed reflections, final R_1 = 0.134, wR_2 = 0.361 and for all data R_1 = 0.254, wR_2 = 0.435. CCDC-852556 contains the corresponding supplementary crystallographic data. These data can be obtained free of charge from *The Cambridge Crystallographic Data Centre via* www.ccdc.cam.ac.uk/data_request/cif.

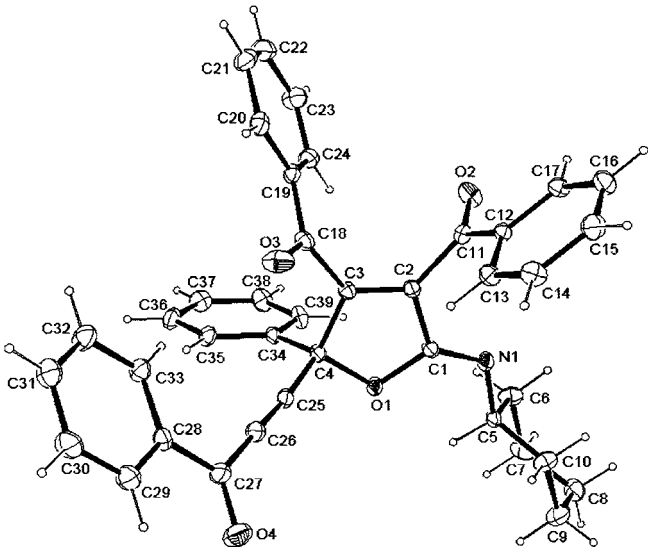


Fig. 3. *ORTEP Plot of the molecular structure of* **3a**

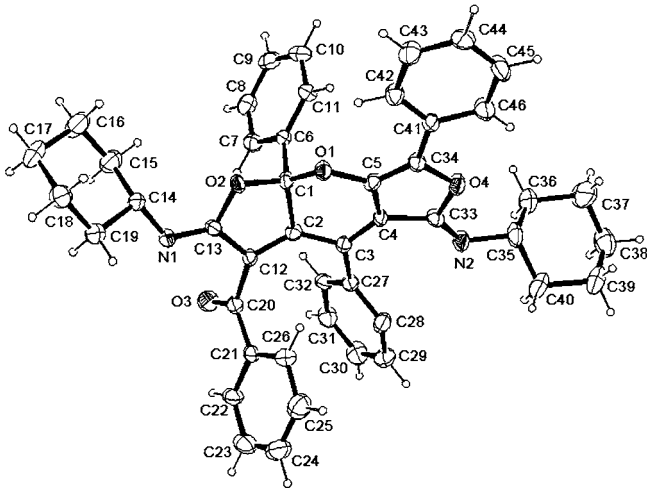
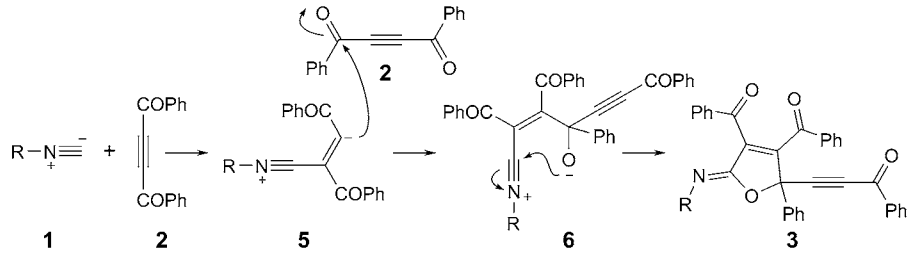


Fig. 4. *ORTEP Plot of the molecular structure of* **4a**

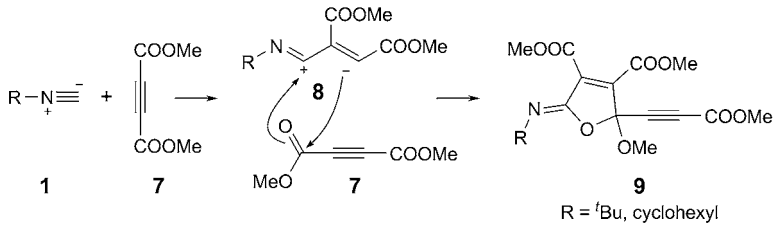
give dimethyl 5-(alkylimino)-2-methoxy-2-(3-methoxy-3-oxoprop-1-yn-1-yl)-2*H*-furan-3,4-dicarboxylates **9** (*Scheme 3*) [1g]. The structure of **9** was established by X-ray crystallography neither by *Winterfeldt et al.* nor later by *George* and co-workers [3].

A mechanistic rationalization for the formation of the 2 : 2 adduct is depicted in *Scheme 4*. Nucleophilic addition of the zwitterionic intermediate **5** on the C=O group of another zwitterion **5** would form the primary 2 : 2 adduct **10**. Nucleophilic attack of the alkoxide group on the adjacent C=O group, followed by the addition of the latter on the adjacent isonitrilium moiety, may produce the 2,7-dioxabicyclo[3.2.0]heptene

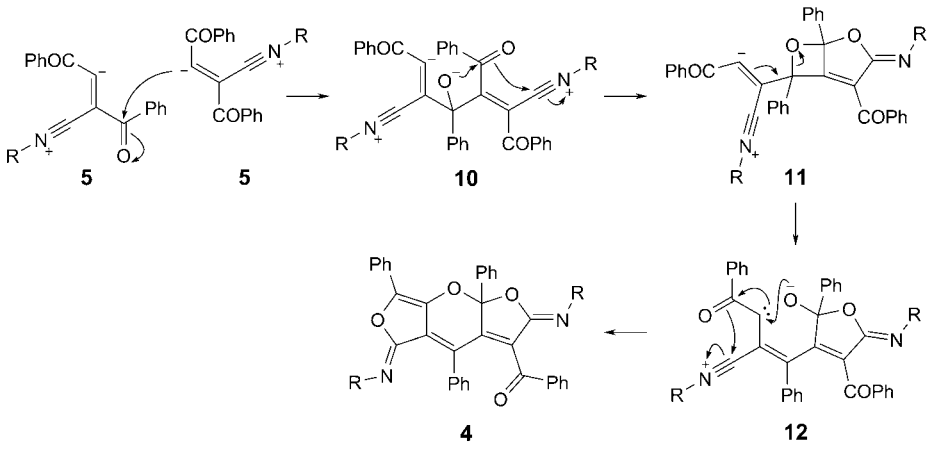
Scheme 2. *Proposed Mechanism for the Formation of* **3**



Scheme 3. *Mechanism for the Formation of* **9** *Proposed by* Winterfeldt et al.



Scheme 4. *Proposed Mechanism for the Formation of* **4**



imino-lactone intermediate **11**. The latter may undergo bond migration and ring opening to form carbene **12**. The carbene moiety may be intramolecularly attacked by the adjacent alkoxide group, followed by the addition of the resulting enolate on the adjacent isonitrilium moiety, to afford the isolated difuro[2,3-*b*:3′,4′-*e*]pyran **4**.

In summary, we have developed a new reaction between isocyanides and dibenzoylacetylene leading to 3-[5-(alkylimino)-3,4-dibenzoyl-2-phenylfuran-2(2*H*)-yl]-1-phenylprop-2-yn-1-ones (**3**; 1 : 2 adduct) and {2,5-bis(alkylimino)-4,7,8a-triphenyl-5*H*-difuro[2,3-*b*:3′,4′-*e*]pyran-3(8a*H*)-yl}(phenyl)methanones (**4**; 2 : 2 adduct) under

mild reaction conditions, *i.e.*, under neutral conditions, and the substances were mixed without any activation or modification. To the best of our knowledge, this is the first report on the synthesis of difuro[2,3-*b*:3′,4′-*e*]pyrans. Moreover, compounds **3** and **4** prepared in the present study may find useful applications as intermediates and/or target compounds in organic synthesis, and also in bioorganic and medicinal chemistry.

This research was supported by the Research Council of the University of Tehran as research project (6102036/1/03).

Experimental Part

General. All the chemicals were obtained from *Merck* (Germany), and were used without further purification. *Dibenzoylacetylene* (**2**) was prepared as described in [11]. Column chromatography (CC): silica gel *60* (*Merck*). M.p.: *Electrothermal 9100* apparatus; uncorrected. IR Spectra: *Shimadzu IR-460* spectrometer; $\tilde{\nu}$ in cm^{-1}. ^1H- and ^{13}C-NMR spectra: *Bruker DRX-500 Avance* (at 500.1 and 125.8 MHz, resp.) instrument; in $CDCl_3$ soln.; δ in ppm rel. to Me_4Si (= 0 ppm), J in Hz. EI-MS (20 eV): *Agilent Technologies (HP) 5973* mass spectrometer; in *m*/*z* (rel. %). Elemental analyses: *Heraeus CHN-O-Rapid* analyzer. X-Ray crystallography: *Bruker SMART* diffractometer; CCD area detector; graphite monochromatized MoK_α radiation.

General Procedure for the Preparation of Compounds **3** *and* **4**. A soln. of the appropriate isocyanide **1** (1 mmol) and **2** (1.4 mmol) in dry CH_2Cl_2 (3 ml) was stirred at r.t. for 48 h. After completion of the reaction (TLC monitoring), the solvent was removed, and the residue was purified by CC (SiO_2; hexane/AcOEt 4 : 1). The products were further purified by recrystallization from hexane/AcOEt 1 : 1.

3-[3,4-Dibenzoyl-5-(cyclohexylimino)-2,5-dihydro-2-phenylfuran-2-yl]-1-phenylprop-2-yn-1-one (**3a**). Colorless crystals. Yield: 0.069 g (36%; relative to cyclohexyl isocyanide). M.p. 122–123°. IR (KBr): 1690 (C=O), 1646, 1591, 1489, 1443, 1328, 1262, 1220, 1177, 1081, 1020, 965, 900, 844, 771, 733, 681, 628. ^1H-NMR (500.1 MHz, $CDCl_3$): 1.17–1.93 (*m*, 10 H); 3.82–3.93 (*m*, 1 H); 7.14 (*dd*, *J* = 7.4, 7.3, 2 H); 7.32 (*dd*, *J* = 7.4, 7.3, 2 H); 7.37 (*t*, *J* = 7.5, 1 H); 7.39–7.46 (*m*, 5 H); 7.50 (*t*, *J* = 7.4, 1 H); 7.52 (*dd*, *J* = 7.3, 7.1, 2 H); 7.62–7.72 (*d*, *J* = 8.2, 2 H; *d*, *J* = 8.1, 2 H; *m*, 1 H); 8.25 (*d*, *J* = 8.2, 2 H). ^{13}C-NMR (125.8 MHz, $CDCl_3$): 24.6; 24.7; 25.7; 33.3; 33.5; 57.0; 60.2; 86.6; 87.1; 125.8; 128.4; 128.5; 128.8; 129.1; 129.1; 129.4; 129.8; 129.9; 133.9; 134.1; 134.6; 135.9; 136.1; 136.2; 136.5; 136.6; 151.0; 154.6; 177.0; 188.4; 189.7. EI-MS: 578 (6, M^+), 552 (5), 524 (6), 500 (16), 480 (16), 468 (7), 440 (6), 423 (7), 412 (7), 390 (12), 375 (20), 369 (20), 362 (10), 347 (9), 313 (16), 291 (11), 264 (13), 239 (19), 211 (13), 183 (8), 149 (16), 137 (9), 105 (100), 77 (86), 66 (28), 57 (37). Anal. calc. for $C_{39}H_{31}NO_4$ (577.68): C 81.09, H 5.41, N 2.42; found: C 80.92, H 5.49, N 2.34.

*[2,5-Bis(cyclohexylimino)-2,8a-dihydro-4,7,8a-triphenyl-5*H*-difuro[2,3-*b*:3′,4′-*e*]pyran-3-yl](phenyl)methanone* (**4a**). Red crystals. Yield: 0.199 g (44%; relative to cyclohexyl isocyanide). M.p. 221–222°. IR (KBr): 1676 (C=O), 1599, 1491, 1450, 1408, 1359, 1284, 1234, 1129, 1068, 1013, 962, 894, 843, 773, 738, 686. ^1H-NMR (500.1 MHz, $CDCl_3$): 1.07–1.74 (*m*, 20 H); 3.73–3.80 (*m*, 1 H); 3.85–3.88 (*m*, 1 H); 6.94 (*t*, *J* = 7.7, 2 H); 7.08 (*t*, *J* = 7.4, 1 H); 7.21 (*d*, *J* = 7.4, 2 H); 7.28 (*t*, *J* = 7.7, 2 H); 7.39–7.46 (*m*, 5 H); 7.51 (*d*, *J* = 7.4, 2 H); 7.55 (*dd*, *J* = 7.9, 7.7, 2 H); 7.64 (*dd*, *J* = 7.5, 2.2, 2 H); 8.05 (*d*, *J* = 7.6, 2 H). ^{13}C-NMR (125.8 MHz, $CDCl_3$): 23.6; 24.0; 24.6; 24.7; 25.7; 25.8; 33.1; 33.4; 33.6; 33.6; 55.8; 56.6; 107.9; 125.5; 125.9; 126.0; 126.9; 127.1; 128.0; 128.1; 128.9; 128.9; 128.9; 129.0; 129.4; 129.8; 130.1; 131.0; 132.5; 133.3; 135.5; 136.4; 137.2; 138.5; 145.9; 146.6; 154.2; 190.1. EI-MS: 687 (3, M^+), 607 (10), 589 (20), 564 (6), 507 (23), 481 (23), 464 (11), 433 (10), 418 (6), 401 (14), 374 (46), 349 (27), 330 (9), 289 (9), 270 (8), 240 (9), 203 (10), 186 (8), 122 (19), 105 (100), 91 (8), 77 (74), 68 (20), 55 (31). Anal. calc. for $C_{46}H_{42}N_2O_4$ (686.85): C 80.44, H 6.16, N 4.08; found: C 80.43, H 6.22, N 3.97.

*3-{3,4-Dibenzoyl-5-[(*tert*-butyl)imino]-2,5-dihydro-2-phenylfuran-2-yl}-1-phenylprop-2-yn-1-one* (**3b**). Colorless crystals. Yield: 0.078 g (43%; relative to ^{t}BuNC). M.p. 110–111°. IR (KBr): 1685 (C=O), 1649, 1588, 1441, 1328, 1260, 1223, 1175, 970, 896, 850, 769, 735, 681. ^1H-NMR (500.1 MHz, $CDCl_3$): 1.41 (*s*, 9 H); 7.14 (*dd*, *J* = 7.9, 7.7, 2 H); 7.32 (*dd*, *J* = 7.8, 7.7, 2 H); 7.37 (*t*, *J* = 7.7, 1 H); 7.37–7.52 (*m*, 8 H); 7.63–7.71 (*d*, *J* = 8.0, 2 H; *d*, *J* = 7.4, 2 H; *m*, 1 H); 8.25 (*d*, *J* = 7.4, 2 H). ^{13}C-NMR (125.8 MHz, $CDCl_3$):

29.8; 55.4; 60.1; 86.4; 87.2; 125.8; 128.4; 128.5; 128.8; 129.1; 129.1; 129.4; 129.7; 129.8; 133.9; 134.1; 134.6; 136.0; 136.2; 136.5; 136.6; 137.0; 150.3; 152.5; 177.0; 188.7; 189.8. EI-MS: 551 (7, M^+), 537 (2), 496 (4), 480 (14), 454 (9), 423 (17), 391 (8), 377 (10), 366 (31), 349 (33), 289 (12.5), 272 (6), 215 (7), 202 (8), 189 (6), 177 (13), 162 (11), 122 (30), 105 (100), 77 (93), 57 (18). Anal. calc. for $C_{37}H_{29}NO_4$ (551.64): C 80.56, H 5.30, N 2.54; found: C 80.70, H 5.56, N 2.33.

*{2,5-Bis[(*tert*-butyl)imino]-2,8a-dihydro-4,7,8a-triphenyl-5*H*-difuro[2,3*-b*:3′,4′*-e*]pyran-3-yl}(phenyl)methanone* (**4b**). Red crystals. Yield: 0.197 g (47%; relative to *tert*-butyl isocyanide). M.p. 193–194°. IR (KBr): 1673 (C=O), 1592, 1490, 1449, 1405, 1355, 1279, 1208, 1164, 1091, 1009, 920, 882, 841, 771, 735, 681. ^{1}H-NMR (500.1 MHz, $CDCl_3$): 1.26, 1.29 (2 *s*, 18 H); 6.94 (*t*, *J* = 7.7, 2 H); 7.08 (*t*, *J* = 7.4, 1 H); 7.21 (*d*, *J* = 7.3, 2 H); 7.29 (*t*, *J* = 7.7, 2 H); 7.38–7.48 (*m*, 5 H); 7.51 (*d*, *J* = 7.3, 2 H); 7.56 (*dd*, *J* = 7.9, 7.6, 2 H); 7.65 (*dd*, *J* = 7.6, 2.0, 2 H); 8.03 (*d*, *J* = 7.5, 2 H). ^{13}C-NMR (125.8 MHz, $CDCl_3$): 30.06; 30.12; 55.1; 55.7; 108.1; 125.5; 125.9; 126.7; 127.0; 127.2; 128.1; 128.1; 128.8; 128.9; 128.9; 129.0; 129.3; 129.7; 130.2; 132.2; 132.3; 133.2; 135.4; 136.5; 137.5; 138.3; 144.9; 145.3; 152.1; 190.4. EI-MS: 635 (4, M^+), 579 (76), 522 (17), 495 (9), 473 (6), 445 (10), 417 (78), 400 (31), 390 (21), 374 (56), 350 (20), 313(5), 239 (6), 105 (100), 77 (78), 57 (86). Anal. calc. for $C_{42}H_{38}N_2O_4$ (634.77): C 79.47, H 6.03, N 4.41; found: C 79.49, H 5.98, N 4.38.

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Received October 24, 2013