Reactions of methyl 1-acetylenyl-9,10-anthraquinone-2-carboxylates with hydrazine

*I. D. Ivanchikova,[a,b] R. N. Myasnikova,[a] and M. S. Shvartsberg[a,b]**

[a]Institute of Chemical Kinetics and Combustion, Siberian Branch of the Russian Academy of Sciences, 3 ul. Institutskaya, 630090 Novosibirsk, Russian Federation. Fax: +7 (383 2) 34 2350. E-mail: shvarts@ns.kinetics.nsc.ru
[b]Novosibirsk State University, 2 ul. Pirogova, 630090 Novosibirsk, Russian Federation. Fax: +7 (383 2) 35 5237

Methyl 1-acetylenyl-9,10-anthraquinone-2-carboxylates react with NH_2NH_2 in ethanol at 80 °C to give commensurable amounts of substituted 7*H*-dibenzo[*de*,*h*]quinolin-7-ones and 3,4-dihydro-3-aminonaphtho[2,3-*f*]isoquinoline-4,7,12-triones. The main route of the reaction apparently includes nucleophilic addition of the reagent to the triple bond of the ester followed by intramolecular cyclization of the adduct with either the carbonyl or the methoxycarbonyl groups involved.

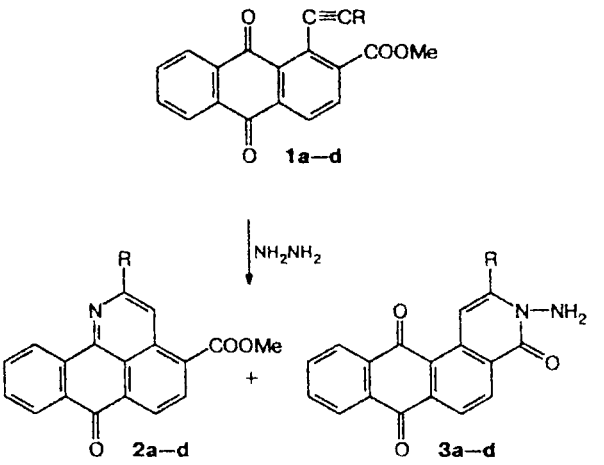
Key words: methyl 1-acetylenyl-9,10-anthraquinone-2-carboxylates, hydrazine, cyclocondensation, 7*H*-dibenzo[*de*,*h*]quinolin-7-ones, 3,4-dihydro-3-aminonaphtho[2,3-*f*]isoquinoline-4,7,12-triones.

Esters of *ortho*-acetylenylsubstituted carbo- and heteroaromatic carboxylic acids react with NH_2NH_2 to give hydrazides, which further are easily cyclized, depending on the structure and reaction conditions, into γ- or δ-*N*-aminolactam or pyridazine ring.[1,2] In continuation of the studies on the synthesis of fused heterocyclic quinoid compounds based on acetylenic derivatives of simple quinones,[3—7] we studied the reactions of NH_2NH_2 with methyl 1-acetylenyl-9,10-anthraquinone-2-carboxylates (**1**).

Since compounds **1** have several electrophilic reaction centers, NH_2NH_2 may first attack not only the alkoxycarbonyl group but also the activated triple bond and the carbonyl groups of the quinoid ring. Direct reactions of NH_2NH_2 with acetylenylquinones have not been studied, but it is known that *N*-nucleophiles such as secondary amines can add to them at the triple bond.[6,8—10] Under quite drastic conditions, *N*-nucleophiles are capable of reacting with the carbonyl groups of quinones as well.[11]

Esters **1a—d** were introduced into the reaction with an excess of NH_2NH_2 in EtOH at 80 °C. Depending on the structure of an acetylenic substituent in the initial compound **1**, the reaction duration varies from 1—2 min for **1d** to 1 h and more for **1a—c**. In all cases, two main products were obtained that could be easily separated by chromatographing. One product was found to belong to the series of substituted 7*H*-dibenzo[*de*,*h*]quinolin-7-ones (**2**), and its formation is surprisingly accompanied by the disruption of the N—N bond and loss of the nitrogen atom of the reagent (Scheme 1).

Scheme 1



R = Bu (**a**), Ph (**b**), CH_2OPh (**c**), H (**d**)

The yields of compounds **2a—d** were 24—64%. The structures of compounds **2a—c** were confirmed by data from elemental analysis and ^1H NMR spectroscopy (Table 1). We failed to isolate quinolinone **2d** in the analytically pure state because of the presence of a minor amount of an intensely colored admixture that was difficult to separate. However, there is no doubt that this compound belongs to the same pyridinanthrone group as **2a—c** does. This is confirmed by the formation of product **3d**, an analog of **3a—c**, along with **2d**; close R_f values in chromatographing of **2a—d**; and characteristic luminescence under UV light.

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Table 1. 7*H*-Dibenzo[*de*,*h*]quinolin-7-ones (**2**)

Compound*	Yield (%)	M.p./°C (solvent)	Found / Calculated (%) C	H	N	Molecular formula	^{1}H NMR, δ (*J*/Hz)
2a	36.7	143—144.5 (benzene—hexane)	76.55 76.50	5.67 5.54	4.18 4.06	$C_{22}H_{19}NO_3$	1.00 (t, 3 H, Me, *J* = 7.0); 1.20—1.60 (m, 2 H, γ-CH_2); 1.70—2.10 (m, 2 H, β-CH_2); 3.07 (t, 2 H, α-CH_2, *J* = 7.0); 4.05 (s, 3 H, COOMe); 7.50—7.95 (m, 2 H, H(9,10)); 8.36 (d, 1 H, H(8), *J* = 7.8); 8.45—8.55 (m, 2 H, H(5,6)); 8.57 (s, 1 H, H(3)); 8.97 (d, 1 H, H(11), *J* = 7.8)
2b	63.8	267—268 (toluene—hexane)	78.88 78.89	4.30 4.14	3.88 3.83	$C_{24}H_{15}NO_3$	4.09 (s, 3 H, COOMe); 7.45—7.90 (m, 5 H, H(9,10), Ph); 8.25—8.50 (m, 3 H, H(8), Ph); 8.60 (m, 2 H, H(5,6)); 9.13 (d, 1 H, H(11), *J* = 8.0); 9.31 (s, 1 H, H(3))
2c	41.7	192—193	75.96 75.94	4.43 4.33	3.66 3.54	$C_{25}H_{17}NO_4$	4.03 (s, 3 H, COOMe); 5.48 (s, 2 H, CH_2O); 7.05—7.45 (m, 5 H, Ph); 7.50—7.90 (m, 2 H, H(9,10)); 8.40 (d, 1 H, H(8), *J* = 7.0); 8.45—8.70 (m, 2 H, H(5,6)); 8.95 (s, 1 H, H(3)); 8.98 (d, 1 H, H(11), *J* = 7.0)

* Compound **2d** was not obtained in the analytically pure state; the yield of crude product was 23.6%.

The other product that is formed simultaneously with compound **2** can be attributed, according to data from elemental analysis, to *N*-aminolactams or, unlikely, to diazinones or diazepinones.[1,2] The structures of these compounds were difficult to determine because of their extremely low solubility. Nevertheless, the data obtained indicate quite strongly that they are δ-lactams, 3,4-dihydro-3-aminonaphtho[2,3-*f*]isoquinoline-4,7,12-triones **3** (the yields of **3a—d** were 24—58%; Table 2). Indeed, the δ-lactam structure of the most soluble compound **3a** unambiguously follows from its ^{1}H NMR spectrum, which exhibits singlets at δ 7.35 (CH group of the heterocycle), δ 5.04 (NH_2 group), and a triplet at δ 2.92 (α-CH_2 of the butyl substituent), as well as from the IR spectrum containing an absorption band at 3330 cm^{-1}, which corresponds to the (N)—NH_2 group.[1,2] Taking into account that all esters **1a—d** react with NH_2NH_2 under the same conditions (*cf.* Ref. 1 and 2) and compounds **3a—d** obtained behave in the same manner when chromatographed, it is

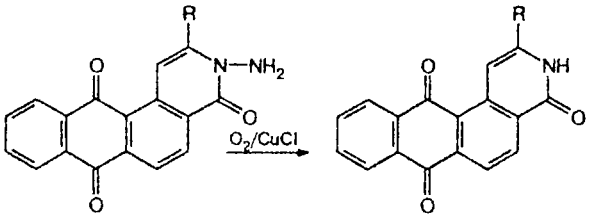
Table 2. Substituted 3,4-dihydronaphtho[2,3-*f*]isoquinoline-4,7,12-triones (**3** and **4**)

Compound	Yield (%)	M.p./°C (solvent)	Found / Calculated (%) C	H	N	Molecular formula
3a[a]	35.0	189—190 (benzene—hexane)	72.76 72.82	5.09 5.24	8.09 8.09	$C_{21}H_{18}N_2O_3$
3b	23.4	313—315 (toluene—hexane)	75.34 75.40	4.00 3.85	7.59 7.65	$C_{23}H_{16}N_2O_3$
3c	36.2	292—294 (toluene)	72.68 72.72	4.12 4.07	7.22 7.07	$C_{24}H_{16}N_2O_4$
3d	58.2	325—327 (toluene)	70.44 70.34	3.51 3.47	9.52 9.65	$C_{17}H_{10}N_2O_3$
4a[b]	92.3	354—355.5 (toluene)	76.00 76.12	5.30 5.17	4.27 4.23	$C_{21}H_{17}NO_3$
4c[b]	53.7	303—305 (toluene)	75.00 75.58	3.77 3.96	3.48 3.67	$C_{24}H_{15}NO_4$

[a] ^{1}H NMR, δ: 1.01 (t, 3 H, Me, *J* = 6.2 Hz); 1.20—1.85 (m, 4 H, β- and γ-CH_2); 2.92 (t, 2 H, α-CH_2, *J* = 7.0 Hz); 5.04 (s, 2 H, NH_2); 7.35 (s, 1 H, H(1)); 7.70—7.85 (m, 2 H, H(8,9)); 8.15—8.35 (m, 3 H, H(5(6),7,10)); 8.75 (d, 1 H, H(6(5)), *J* = 8.4 Hz).
[b] Obtained by deamination of **3a** and **3c**, respectively.

safe to say that compounds **3b—d**, like **3a**, are *N*-aminolactams. To confirm this, compounds **3a** and **3c** were deaminated by the action of O_2 in the presence of CuCl in pyridine[2] to give lactams **4a,c**, which is typical of *N*-aminolactams (Scheme 2).

Scheme 2



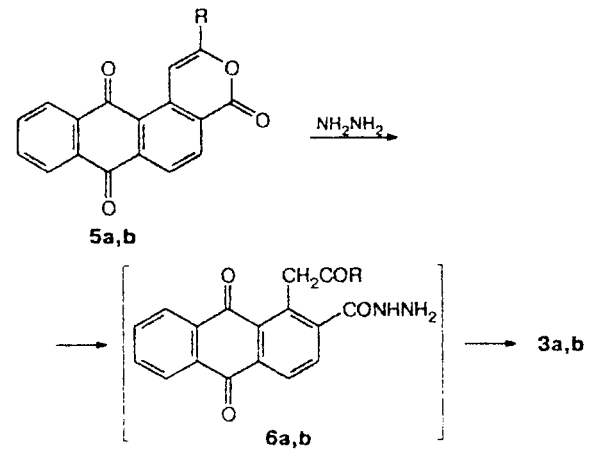
3a,c **4a,c**

R = Bu (**a**), CH_2OPh (**c**)

In spite of the fact that compounds **3a—d** generally belong to *N*-aminolactams, both δ- and γ-lactams may be formed. Other factors being equal, such a change in regiodirection of cyclization depends on the structure of the acetylenic substituent in the initial compound and most often occurs, although usually in part, in passing from arylethynyl to alkynyl derivatives.[12,13] That is why the size of the heterocycle in lactam **3b** should, at the least, be determined directly in order to ascertain that the direction of lactamization of esters **1a—d** remains constant.

It has been shown previously with 4*H*-2-butylanthra[2,1-*c*]pyran-4,7,12-trione (**5a**)[4] as an example that δ-lactones of this type are recyclized into *N*-aminolactams with the size of the heterocycle retained during hydrazinolysis (Scheme 3).

Scheme 3



5a,b **6a,b**

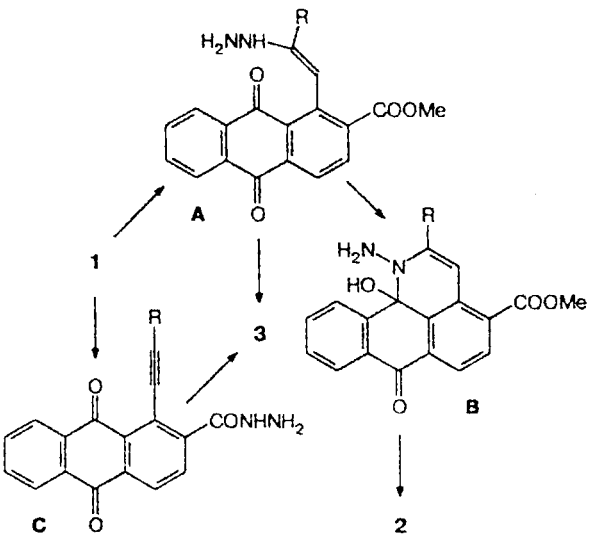
R = Bu (**a**), Ph (**b**)

Lactone **5a** reacts with NH_2NH_2 under mild conditions (propanol, 20 °C, 10 min) to give 1-(2-oxohexyl)-9,10-anthraquinone-2-carbohydrazide (**6a**). Compound **6a** is very labile and cyclized into lactam **3a** even in solution at 20 °C and during isolation. The transformations of **5a** into **6a** and further into **3a** can be followed easily by TLC. The structure of **6a** was confirmed by data from the ^1H NMR spectrum, which contains a singlet at δ 4.00 (1-CH_2 group of the 2-oxohexyl substituent) and a broadened signal at δ 4.35 (NH_2 group of hydrazide).

To determine the size of the lactam ring in **3b**, phenylsubstituted δ-lactone **5b**, whose structure is strictly proved,[4] was introduced into the reaction with NH_2NH_2. Because intermediate **6b** was more stable than analogous butyl ketone **6a**, it was isolated and analyzed. In boiling propanol, compound **6b** is completely recyclized into *N*-aminolactam **3b**, which is identical to that obtained from ester **1b** and NH_2NH_2. Therefore, the replacement of the alkynyl substituent by the phenylethynyl one in ester **1** does not affect the regiodirection of cyclization. It is also significant that only one isomer of *N*-aminolactam is formed from any ester **1a—d**. Apparently, these facts suggest that the cyclization of esters **1a—d** into lactams **3a—d** is characterized by a uniform structural direction and *N*-aminolactams **3c,d** can be assigned the δ-lactam structure, which was unambiguously determined for **3a,b**.

Thus, 7*H*-dibenzo[*de,h*]quinolin-7-ones **2a—d** and 3,4-dihydro-3-aminonaphtho[2,3-*f*]isoquinoline-4,7,12-triones (**3a—d**) were obtained by the reaction of esters **1a—d** with NH_2NH_2. There is no doubt that the initial stage of formation of compound **2** is the addition of NH_2NH_2 at the triple bond of ester **1**. We assume that adduct **A** further undergoes intramolecular cyclization into intermediate **B**, which is reduced to compound **2** by an excess NH_2NH_2 (Scheme 4).

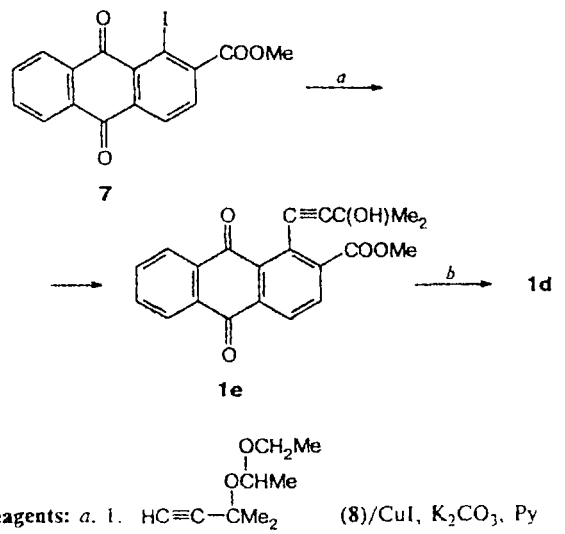
Scheme 4



The precursors of naphthoisoquinolinetriones **3** can be both adducts **A** and hydrazides **C**.

Since no intermediate product is detected in the reaction of compound **1** with NH_2NH_2, its initial stage seems to be the rate-limiting one. The reaction duration strongly depends on the structure of the acetylenic substituent in **1**. For example, it is much shorter for ethynyl-substituted ester **1d** than for **1a—c**. On the other hand, it is unlikely that the structure of the acetylenic substituent in esters **1** affects substantially the rate of reaction of the methoxycarbonyl group with NH_2NH_2. If the latter is true, the formation of both quinolinone **2d** and aminolactam **3d** from fast-reacting ester **1d** mostly occurs through adduct **A**. In principle, lactams **3a—c**, which are formed tens times more slowly than lactam **3d**, can be obtained in two ways (through intermediates **A** and **C**). However, in all cases, quinolinones **2a—c** are dominant in the reaction products, and the molar ratio **2b** : **3b** even reaches 2.5. This makes it certain that esters **1a—d**, independently of the structure, react with NH_2NH_2 in the initial stage mainly at the triple bond and the reaction essentially proceeds *via* adduct **A**.

The synthesis of the initial compounds **1a—c** has been described earlier.[4] In the presence of a bulky substituent in position 2, the substitution of the acetylenic group for a halogen atom in position 1 of anthraquinone, catalyzed by Pd complexes, occurs poorly, which is apparently due to steric hindrances.[4] That is why compound **1d** was synthesized by Cu-catalyzed cross-linking[14] of methyl 1-iodo-9,10-anthraquinone-2-carboxylate **7** with acetalized 2-methylbut-3-yn-2-ol (**8**) and subsequent decomposition of the acetylenic alcohol **1e** by alkali according to the general scheme proposed by us.[15,16]



Reagents: *a*. 1. (**8**)/CuI, K_2CO_3, Py 2. H_3O^+; *b*. KOH, C_6H_6.

The decomposition of **1e** in boiling benzene in the presence of KOH powder occurred slowly (2.5 h) and was accompanied by considerable resinification to give **1d** in 34% yield. We attempted to obtain compound **1d** by decomposition, under similar conditions, of methyl 1-benzoylethynyl-9,10-anthraquinone-2-carboxylate (**1f**) synthesized by the condensation of **7** with copper benzoylacetylide (DMF, 60 °C, 2 h), but failed.

Experimental

^1H NMR spectra were recorded on Bruker AM-250 and Jeol FX-90 spectrometers in $CDCl_3$ at 25 °C. IR spectra were recorded on a UR-20 spectrophotometer in $CHCl_3$.

Methyl 1-(3-hydroxy-3-methylbutynyl)anthraquinone-2-carboxylate (1e). Ester **7** (3.80 g, 9.8 mmol), acetal **8** (3.04 g, 19.5 mmol), K_2CO_3 (5.32 g, 41.6 mmol), and CuI (2.86 g, 15.0 mmol) in 170 mL of pyridine were stirred in an argon atmosphere at 115 °C for 2 h (monitored by TLC: Silufol, dichloroethane), diluted with 400 mL of $CHCl_3$, and washed with water, a 15% NH_3 solution, and dilute HCl (1 : 2). The resulting solution was periodically shaken for 4 h with 400 mL of dilute HCl (1 : 1). Once the hydrolysis was completed (monitored by TLC: Silufol, dichloroethane—ether, 10 : 1), the organic layer was washed with water to neutral reaction and the solvent removed *in vacuo*. The residue was chromatographed on silica gel in dichloroethane and a dichloroethane—ether (10 : 1) mixture and recrystallized from a mixture of benzene with hexane to give compound **1e** (1.93 g, 57.3%), m.p. 134—135 °C. Found (%): C, 72.43; H, 4.62. $C_{21}H_{16}O_5$. Calculated (%): C, 72.40; H, 4.63. ^1H NMR, δ: 1.71 (s, 6 H, Me); 2.87 (s, 1 H, OH); 3.98 (s, 3 H, OMe); 7.70—7.90 (m, 2 H, H(6,7)); 8.00, 8.31 (both d, 2 H, H(3,4)); 8.15—8.30 (m, 2 H, H(5,8)). IR, ν/cm^{-1}: 2230 sh, 2235 (C≡C), 1680, 1740 (CO), 3350 br., 3550 (OH).

Methyl 1-benzoylethynylanthraquinone-2-carboxylate (1f). Ester **7** (1.18 g, 3.0 mmol) and copper benzoylacetylide (0.87 g, 4.5 mmol) in 90 mL of DMF were heated in an argon atmosphere at 60 °C for 1 h 50 min (monitored by TLC: Silufol, $CHCl_3$), diluted with 400 mL of $CHCl_3$, washed with water, and dried with $MgSO_4$. The yield of compound **1f** was 0.90 g (75.6%), m.p. 206—207 °C (from benzene—hexane). Found (%): C, 76.38; H, 3.54. $C_{25}H_{14}O_5$. Calculated (%): C, 76.14; H, 3.58. ^1H NMR, δ: 4.01 (s, 3 H, COOMe); 7.45—7.65 (m, 3 H, Ph); 7.75—7.90 (m, 2 H, H(6,7)); 8.16, 8.48 (both d, 2 H, H(3,4)); 8.25—8.50 (m, 4 H, H(5,8), Ph). IR, ν/cm^{-1}: 2215 (C≡C), 1655, 1690, 1745 (CO).

Methyl 1-ethynylanthraquinone-2-carboxylate (1d). Ester **1e** (1.05 g, 3.0 mmol) and calcined KOH powder (0.68 g, 12.0 mmol) in 160 mL of anhydrous benzene were refluxed with stirring for 2.5 h (monitored by TLC: Silufol, benzene—ether, 6 : 1) and filtered through a silica gel layer to give compound **1d** (0.30 g, 33.7%), m.p. 207—208 °C (decomp.; from benzene), the appearance changes at >168 °C. Found (%): C, 74.56; H, 3.47. $C_{18}H_{10}O_4$. Calculated (%): C, 74.89; H, 3.47. ^1H NMR, δ: 3.82 (s, 1 H, C≡CH); 3.98 (s, 3 H, COOMe); 7.75—7.90 (m, 2 H, H(6,7)); 8.00 (d, 1 H, H(3(4))); 8.15—8.45 (m, 3 H, H(4(3),5,8)). IR, ν/cm^{-1}: 1730, 1670, 1645 (CO).

The reaction of methyl 1-(hex-1-ynyl)anthraquinone-2-carboxylate (1a) with NH_2NH_2. A solution of **1a** (0.60 g, 1.7 mmol) and NH_2NH_2 (1.0 g, 31.2 mmol, 1.0 mL) in 25 mL of ethanol was refluxed for 1.5 h (monitored by TLC: Silufol, $CHCl_3$), diluted with 400 mL of $CHCl_3$, washed with water, and dried with $MgSO_4$. The resulting solution was chromatographed on silica gel with benzene and a benzene—ether

(10 : 1) mixture to give compound **2a** (0.22 g) (see Table 1) and compound **3a** (0.21 g) (see Table 2).

Compounds **2b—d** (see Table 1) and **3b—d** (see Table 2) were obtained similarly.

Hydrazinolysis of 4*H*-2-butyl- (5a) and 4*H*-2-phenylanthra[2,1-*c*]pyran-4,7,12-trione (5b). A solution of **5a** (0.31 g, 0.9 mmol) and и NH_2NH_2 (0.6 g, 18.7 mmol, 0.6 mL) in 15 mL of propanol were stirred at 95 °C for 30 min (monitored by TLC: Silufol, $CHCl_3$), diluted with 200 mL of $CHCl_3$, washed with water, and dried with $MgSO_4$. The resulting solution was chromatographed on silica gel with a benzene—ether (30 : 1) mixture to give compound **3a** (0.21 g, 65.6%).

Compound **3b** was obtained similarly from **5b** (45 min) in 84.3% yield.

When introduced into the reaction with NH_2NH_2 (0.20 g, 6.3 mmol, 0.2 mL) in 10 mL of propanol at 20 °C for 10 min, lactone **5a** (0.10 g, 0.3 mmol) was completely transformed into hydrazide **6a** (monitored by TLC: Silufol, $CHCl_3$). The reaction mixture was diluted with 100 mL of $CHCl_3$, washed with water, and the solvent was fast removed *in vacuo* in the cold. Benzene (10 mL) was added, and the precipitate that formed was filtered off and washed with ether to give compound **6a** (0.05 g, 45.5%), the isolation being accompanied by the cyclization of **6a** into **3a**. The crystals of hydrazide **6a** are yellowish orange. When heated and in solutions at 20 °C, **6a** is easily transformed into **3a**. ^{1}H NMR, δ: 0.90 (t, 3 H, Me); 1.15—1.55 (m, 4 H, β- and γ-CH_2); 1.90—2.10 (br.m, 2 H, $CO\underline{CH}_2Pr$); 4.00 (s, 2 H, $\underline{CH}_2COBu$); 4.35 (br.s, 2 H, NH_2); 7.70—7.85 (m, 2 H, H(6,7)); 8.10—8.45 (m, 3 H, H(3(4),5,8)); 8.55 (d, 1 H, H(4(3))).

In the same way, compound **5b** (0.20 g, 0.6 mmol) and NH_2NH_2 (0.45 g, 14.1 mmol, 0.45 mL) in 20 mL of propanol were transformed into **6b** for 2.5 h. Crude **6b** was suspended in 150 mL of benzene (the benzene was then partially (to ~20 mL volume) removed *in vacuo* at room temperature), filtered off, and washed with ether; this operation being repeated three times. The yield of **6b** was 0.18 g (81.8%). When heated, **6b** is cyclized into **3b**. Found (%): C, 71.65; H, 4.14; N, 7.05. $C_{23}H_{16}N_2O_4$. Calculated (%): C, 71.87; H, 4.20; N, 7.29.

3,4-Dihydro-2-butylnaphtho[2,3-*f*]isoquinoline-4,7,12-trione (4a). Oxygen was bubbled through a solution of compound **3a** (0.41 g, 1.1 mmol) and CuCl (0.06 g) in 40 mL of pyridine at 20 °C for 40 min (monitored by TLC: Silufol, $CHCl_3$). The reaction mixture was diluted with 1 L of $CHCl_3$, and washed with 10% HCl and water. The solvent was removed, and the residue was recrystallized from toluene to give **4a** (0.36 g) (see Table 2).

Compound **4b** was obtained in the same manner from **3b** (see Table 2).

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