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Synthesis and Structure of New 2-(2-Quinolyl)-1,3-tropolone Derivatives

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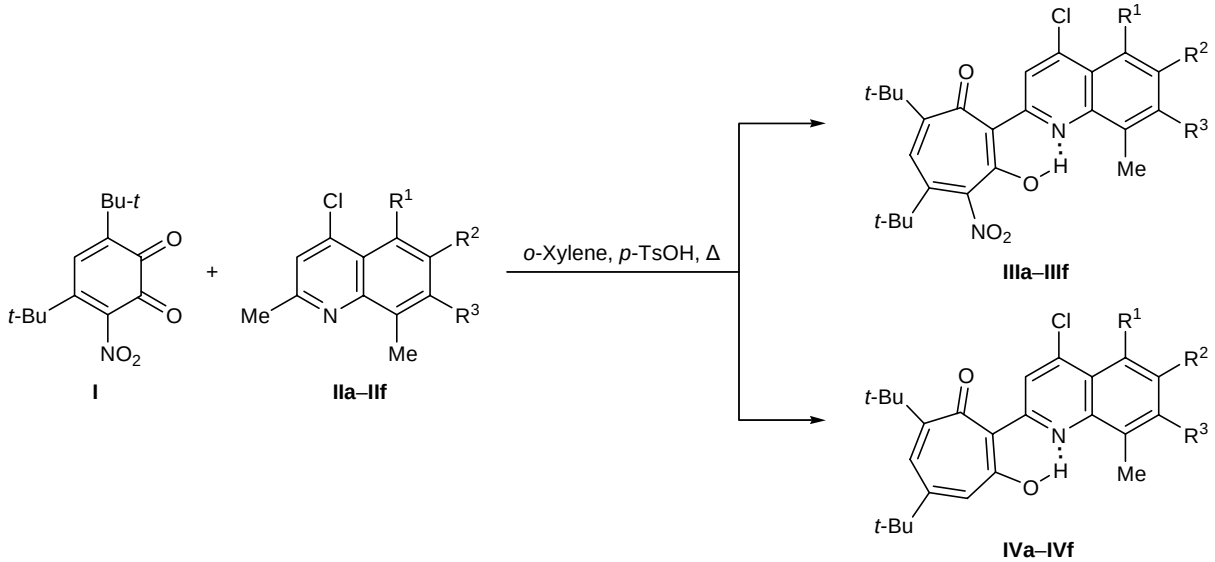
Abstract—4,6-Di-*tert*-butyl-3-nitro-1,2-benzoquinone reacts with substituted 2-methylquinolines to give the corresponding 2-(2-quinolyl)-4-nitro-1,3-tropolones and 2-(2-quinolyl)-1,3-tropolones.

We previously showed [1] that, contrary to expectations, the condensation of 3,5-di-*tert*-butyl-1,2-benzoquinone with 2-methylquinolines gives previously unknown 1,3-tropolone derivatives rather than 2-quinolyl-*o*-methylenequinones. The present communication extends the scope of application of this reaction to other sterically hindered *o*-quinones. The formation of C–C bond in reactions of carbonyl compounds with those containing an activated methyl or methylene group was extensively studied. However, the behavior of quinone derivatives in analogous processes was poorly explored. High reactivity and relatively high thermal stability of sterically hindered quinones makes them attractive from the viewpoint of synthesis of novel compounds which could possess practically important properties, including specific biological activity.

We have found that 4,6-di-*tert*-butyl-3-nitro-1,2-benzoquinone (**I**) reacts with substituted 2-methylquinolines **IIa**–**IIf** on heating in boiling *o*-xylene in the presence of *p*-toluenesulfonic acid (reaction time 1 h) to give previously unknown nitrotropolones **IIIa**–**IIIf**

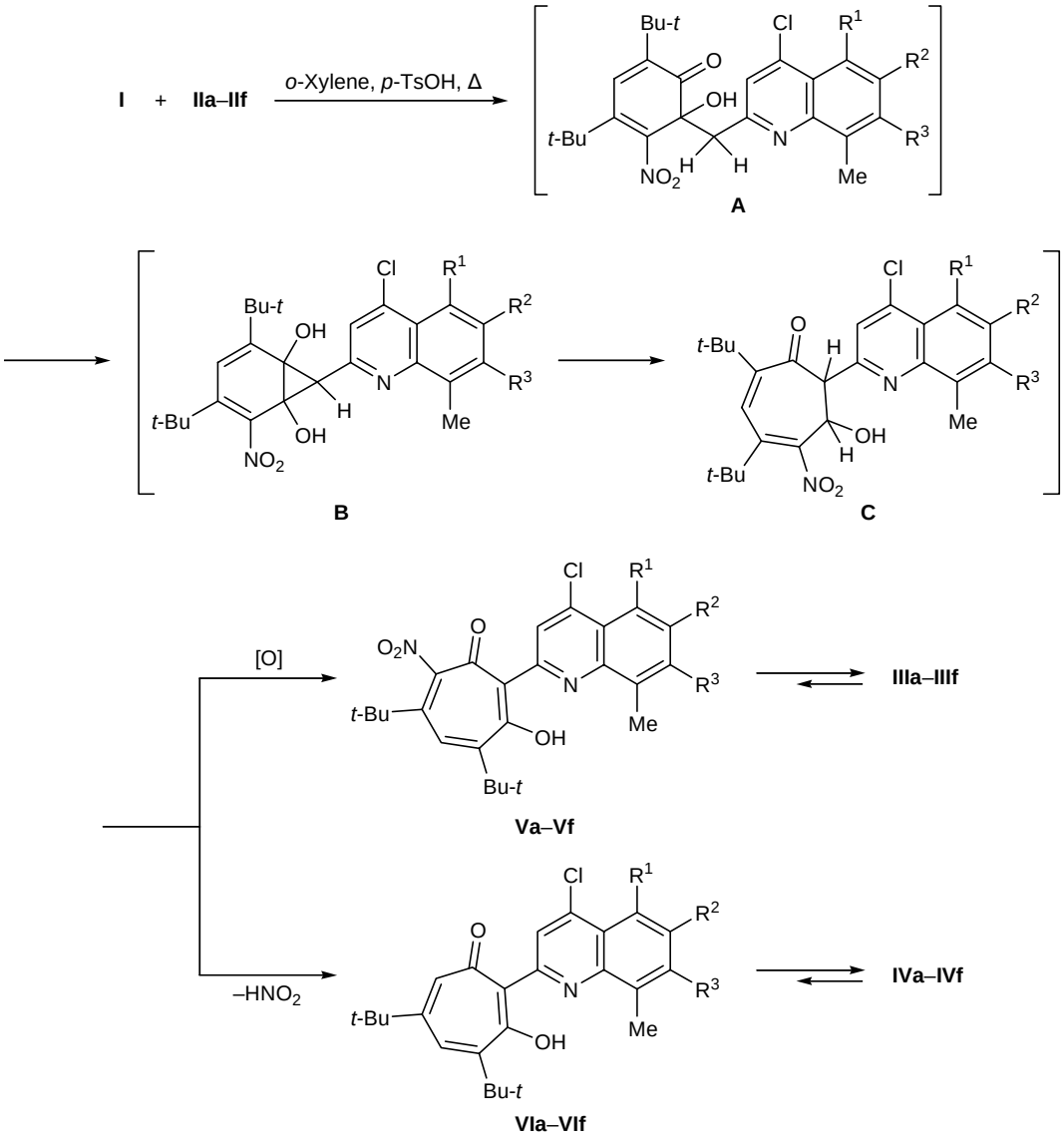
Scheme 1.



I **IIa–IIf** **IIIa–IIIf** **IVa–IVf**

$R^1 = R^2 = R^3 = H$ (**a**); $R^1 = R^3 = H$, $R^2 = Me$ (**b**); $R^1 = R^2 = H$, $R^3 = Me$ (**c**); $R^1 = NO_2$, $R^2 = R^3 = H$ (**d**); $R^1 = NO_2$, $R^2 = Me$, $R^3 = H$ (**e**); $R^1 = NO_2$, $R^2 = H$, $R^3 = Me$ (**f**).

Scheme 2.

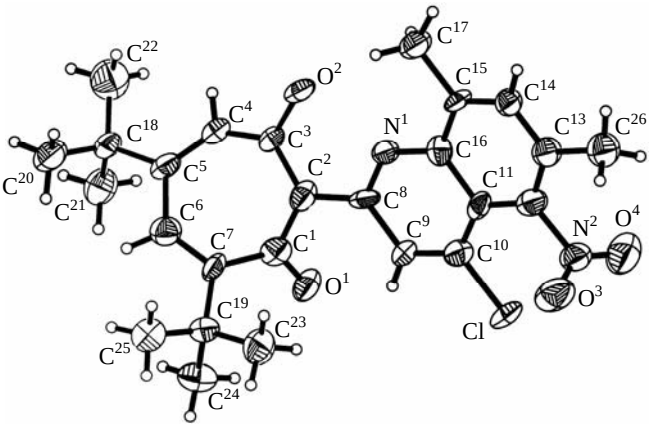


(Scheme 1). The reactions were also accompanied by formation of a small amount of (3–4%) of previously described [1] tropolones **IVa**–**IVf** having no nitro group in the seven-membered ring. Presumably, the observed transformation follows Scheme 2. Intermediate norcaradiene derivative **B** formed in the first stage via 1,3-sigmatropic shift of hydrogen in the primary condensation product (**A**) undergoes thermal isomerization into 2,3-dihydrotropolone **C**. The subsequent oxidation of intermediate **C** with quinone **I** could give rise to two isomeric products **V** and **III**. More thermodynamically stable tropolones **III** are formed as a result of prototropic isomerization and rotation of the seven-membered carbocycle about the ordinary C–C bond. Concurrent elimination of nitrous acid molecule, which is accompanied by 1,3-sigmatropic hydrogen shift leads to compounds **IV**.

Tropolones **IIIa**–**IIIf** and **IVa**–**IVf** were characterized by ^{1}H NMR, IR, and mass spectra. The ^{1}H NMR spectra of **IVa**–**IVf** contained a sharp singlet at δ 18–19 ppm due to proton of the hydroxy group in the tropolone ring, which is involved in intramolecular hydrogen bond with the quinoline nitrogen atom (six-membered H-chelate ring). Signals from the hydroxy protons of **IIIa**–**IIIf** are appreciably broadened and are displaced upfield by 0.5–1.0 ppm. Protons in the seven-membered ring of **IVa**–**IVf** resonate at δ 6.6–6.8 ppm as doublets (J = 1.5–1.8 Hz). Compounds **IIIa**–**IIIf** show in the ^{1}H NMR spectra a singlet at δ 6.4–6.5 ppm due to 5-H in the tropolone ring.

The structure of 1,3-tropolone **IVe** was proved by X-ray analysis (see figure; hydrogen atom at O^2 was not localized). The molecular structure of compound **IVe** is analogous to the structure of previously studied 5,7-di-*tert*-butyl)-2-(4-chloro-8-methylquinolin-2-yl)-1,3-tropolone (**IVa**) [1] having no nitro group in the quinoline ring. The C^1, C^2, C^3, and C^8 atoms lie in one plane within 0.014 Å (0.019 Å [1]), as well as the C^2, C^8, C^9, and N^1 atoms (0.015 and 0.017 Å, respectively), though the two molecular fragments in **IVe** are turned apart about the C^2–C^8 bond by 13° (4.8° [1]). The position of hydrogen atom on O^2 in molecule **IVe** was not determined, but the distance between the quinoline nitrogen atom and oxygen atom of the hydroxy group in both molecules (**IVe** and **IVa**) is equal to 2.46 Å, indicating formation of especially strong intramolecular hydrogen bond O–H···N. Obviously, such configuration of the central entity reduces steric strain at the C^3 atom, which leads to leveling of the $C^4C^3O^2$ and $C^2C^3O^2$ angles (117 and 118°, respectively, against 113 and 122° in molecule **IVa** [1]). The $N^1C^9C^{10}C^{11}C^{12}C^{13}C^{14}C^{15}C^{16}$ quinoline fragment is planar within 0.04 Å; the N^2 and C^{26} atoms deviate from that plane by 0.15 Å, and the C^{17} and chlorine atoms, by 0.14 and 0.30 Å, respectively, but in the opposite direction. The $N^1C^9C^{10}C^{11}C^{12}C^{13}C^{14}C^{15}C^{16}$ and $N^1C^9C^8C^2$ planes form a dihedral angle of 8.7° along the N^1C^9 line; no such bent was observed in molecule **IVa** [1]. The nitro group is turned with respect to the quinoline ring plane about the C^{12}–N^2 bond, the torsion angle $C^{11}C^{12}N^2O^3$ being 78°. The distances between the chlorine atom, on the one hand, and oxygen atoms of the nitro group, on the other, are 3.11 and 3.30 Å. Molecules **IVe** in crystal are characterized by a shortened contact (d = 3.12 Å) between the chlorine atom and oxygen atom of the hydroxy group along the *a* axis. The C^4 and O^2 atoms lie in the $C^1C^2C^3C^8$ plane (within 0.03 Å), the O^1 atom is located below the plane of the figure by 0.78 Å, while the C^7 atom appears above that plane by 1.08 Å. The seven-membered ring is bent along the $C^1\cdots C^4$ line, so that the $C^4C^1C^6$ plane and the plane including the other atoms form a dihedral angle of 38.9°, and the C^{18} and C^{19} atoms deviate from the $C^4C^1C^6$ plane by 0.38 and 0.30 Å, respectively, in opposite directions, leading to the corresponding deviations of the *tert*-butyl groups from the tropolone ring plane.

To conclude, it should be noted that the described ring expansion in sterically hindered *o*-quinones opens a new synthetic route to previously inaccessible 2-substituted 1,3-tropolones.



Structure of the molecule of 5,7-di-*tert*-butyl-2-(4-chloro-6,8-dimethyl-5-nitroquinolin-2-yl)-1,3-tropolone (**IVe**) according to the X-ray diffraction data.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Varian Unity-300 spectrometer. The mass spectra were run on a Finnigan MAT Incos-50 instrument. The IR spectra were measured on a Specord 75IR spectrometer from samples dispersed in mineral oil.

X-Ray analysis of a single crystal of compound IVe. The unit cell parameters and reflection intensities (a three-dimensional set) were measured on a KUMA Diffraction KM-4 diffractometer (λMoK_α irradiation, graphite monochromator) from an isometric single crystal of a poor quality (indefinite form with a radius of about 0.23 mm). Molecular formula $C_{26}H_{29}ClN_2O_4$; rhombic crystals with the following unit cell parameters: a = 9.621(7), b = 11.276(9), c = 21.96(1) Å; V = 2382(3) Å^3; M 468.9; space group $p2_12_12_1$; Z = 4; d_{calc} = 1.308 g/cm^3. Intensities of 2197 reflections were measured in the independent part of the reciprocal space ($2\theta \le 46.14°$) using $\omega/2\theta$ scanning. After exclusion of systematically cancelled reflections, the working array contained 2120 reflections with $I > 2\sigma(I)$. The structure was solved by the direct method and was refined by the full-matrix least-squares procedure with respect to F^2 in anisotropic approximation for non-hydrogen atoms using SHELXL-97 program [3]. All hydrogen atoms were localized from the geometry considerations (except for the hydrogen atom on O^2), and their positions were not refined because of poor quality of the single crystal.

5,7-Di-*tert*-butyl-2-(4-chloro-8-methylquinolin-2-yl)-4-nitro-1,3-tropolone (IIIa) and 5,7-di-*tert*-butyl-2-(4-chloro-8-methylquinolin-2-yl)-1,3-tropolone (IVa). A solution of 1.86 g (7 mmol) of 4,6-di-*tert*-

butyl-3-nitro-1,2-benzoquinone (**I**) [2], 0.57 g (3 mmol) of 4-chloro-2,8-dimethylquinoline (**IIa**), and 0.2 g of *p*-toluenesulfonic acid in 10 ml of *o*-xylene was heated for 1 h under reflux. The mixture was cooled and applied to a column (750×20 mm) charged with aluminum oxide, and the column was eluted with hexane–chloroform (1:1). Two bright yellow fractions were collected; the first fraction contained compound **IVa**, and the second, compound **IIIa**. After removal of the solvent, the residues were recrystallized from 2-propanol.

Compound **IIIa**. Yield 0.081 g (6%), yellow crystals, mp 228–230°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 s (9H, 5-*t*-Bu), 1.31 s (9H, 7-*t*-Bu), 2.72 s (3H, 8'-CH_3), 6.38 s (1H, 6-H), 7.51 t (1H, 6'-H, *J* = 7.7 Hz), 7.65 d (1H, 7'-H, *J* = 7.6 Hz), 8.03 d (1H, 5'-H, *J* = 7.6 Hz), 8.34 s (1H, 3'-H), 18.03 br.s (1H, 3-OH). Mass spectrum, *m/z* (I_{rel}, %): 454.9 (10) $[M]^+$, 426 (25), 408 (50), 384 (10), 352 (25), 322 (20), 176 (30), 154 (30), 91 (35), 57 (70), 41(100). Found, %: C 65.91; H 5.88; Cl 7.71; N 6.11. $C_{25}H_{27}ClN_2O_4$. Calculated, %: C 66.00; N 5.98; Cl 7.79; N 6.16. *M* 454.95.

Compound **IVa**. Yield 0.037 g (3%), yellow crystals, mp 189–191°C. IR spectrum: ν(C=O) 1660 cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.24 s (9H, 5-*t*-Bu), 1.37 s (9H, 7-*t*-Bu), 2.72 s (3H, 8'-CH_3), 6.65 d (1H, 4-H, *J* = 1.7 Hz), 6.72 d (1H, 6-H, *J* = 1.7 Hz), 7.41 t (1H, 6'-H, *J* = 7.7 Hz), 7.54 d (1H, 7'-H, *J* = 7.6 Hz), 7.95 d (1H, 5'-H, *J* = 7.6 Hz), 8.23 s (1H, 3'-H), 19.12 s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 409.9 (10) $[M]^+$, 381 (90), 366 (100), 350 (40), 338 (40), 310 (45), 57 (40), 41 (50). Found, %: C 73.22; H 6.71; Cl 8.62; N 3.44. $C_{25}H_{28}ClNO_2$. Calculated, %: C 73.25; H 6.88; Cl 8.65; N 3.42. *M* 409.95.

5,7-Di-*tert*-butyl-2-(4-chloro-6,8-dimethylquinolin-2-yl)-4-nitro-1,3-tropolone (IIIb) and 5,7-di-*tert*-butyl-2-(4-chloro-6,8-dimethylquinolin-2-yl)-1,3-tropolone (IVb) were synthesized as described above for **IIIa** and **IVa** from 0.62 g of quinoline **IIb**.

Compound **IIIb**. Yield 0.084 g (6%), yellow crystals, mp 214–216°C. IR spectrum: ν(C=O) 1633 cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 s (9H, 5-*t*-Bu), 1.31 s (9H, 7-*t*-Bu), 2.65 s (3H, 6'-CH_3), 2.69 s (3H, 8'-CH_3), 6.38 s (1H, 6-H), 7.48 s (1H, 7'-H), 7.80 s (1H, 5'-H), 8.32 s (1H, 3'-H), 18.12 br.s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 468.8 (15) $[M]^+$, 440 (45), 422 (100), 398 (15), 366 (15), 154 (10), 91 (15), 57 (30), 41(35). Found, %: C 66.47; H 6.01; Cl 7.45; N 6.02. $C_{26}H_{29}ClN_2O_4$. Calculated, %: C 66.59; H 6.23; Cl 7.56; N 5.97. *M* 468.98.

Compound **IVb**. Yield 0.038 g (3%), yellow crystals, mp 198–201°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.23 s (9H, 5-*t*-Bu), 1.37 s (9H, 7-*t*-Bu), 2.51 s (3H, 6'-CH_3), 2.68 s (3H, 8'-CH_3), 6.65 d (1H, 4-H, *J* = 1.88 Hz), 6.73 d (1H, 6-H, *J* = 1.88 Hz), 7.43 s (1H, 7'-H), 7.78 s (1H, 5'-H), 8.23 s (1H, 3'-H), 19.19 s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 423.9 (8) $[M]^+$, 395 (88), 380 (100), 352 (25), 57 (50), 41 (45). Found, %: C 73.61; H 7.02; Cl 8.12; N 3.34. $C_{26}H_{30}ClNO_2$. Calculated, %: C 73.66; H 7.13; Cl 8.36; N 3.30. *M* 423.98.

5,7-Di-*tert*-butyl-2-(4-chloro-7,8-dimethylquinolin-2-yl)-4-nitro-1,3-tropolone (IIIc) and 5,7-di-*tert*-butyl-2-(4-chloro-7,8-dimethylquinolin-2-yl)-1,3-tropolone (IVc) were synthesized as described above for compounds **IIIa** and **IVa** from 0.62 g of compound **IIc**. The products were isolated by column chromatography on aluminum oxide (800×20 mm) using hexane–chloroform (2:1) as eluent.

Compound **IIIc**. Yield 0.113 g (8%), yellow crystals, mp 200–202°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 s (9H, 5-*t*-Bu), 1.31 s (9H, 7-*t*-Bu), 2.54 s (3H, 7'-CH_3), 2.61 s (3H, 8'-CH_3), 6.36 s (1H, 6-H), 7.43 d (1H, 6'-H, *J* = 8.4 Hz), 7.92 d (1H, 5'-H, *J* = 8.4 Hz), 8.28 s (1H, 3'-H), 17.93 br.s (1H, 3-OH). Found, %: C 66.48; H 6.15; Cl 7.49; N 6.01. $C_{26}H_{29}ClN_2O_4$. Calculated, %: C 66.59; H 6.23; Cl 7.56; N 5.97.

Compound **IVc**. Yield 0,045 g (3.5%), yellow crystals, mp 174–176°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.23 s (9H, 5-*t*-Bu), 1.37 s (9H, 7-*t*-Bu), 2.52 s (3H, 7'-CH_3), 2.63 s (3H, 8'-CH_3), 6.64 d (1H, 4-H, *J* = 1.86 Hz), 6.68 d (1H, 6-H, *J* = 1.86 Hz), 7.37 d (1H, 6'-H, *J* = 8.5 Hz), 7.89 d (1H, 5'-H, *J* = 8.5 Hz), 8.20 s (1H, 3'-H), 19.31 s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 423.9 (2) $[M]^+$, 395 (88), 380 (100), 352 (28), 338 (12.5), 57 (35), 41 (37.5). Found, %: C 73.63; H 6.94; Cl 8.21; N 3.23. $C_{26}H_{30}ClNO_2$. Calculated, %: C 73.66; H 7.13; Cl 8.36; N 3.30. *M* 423.98.

5,7-Di-*tert*-butyl-2-(4-chloro-8-methyl-5-nitroquinolin-2-yl)-4-nitro-1,3-tropolone (IIId) and 5,7-di-*tert*-butyl-2-(4-chloro-8-methyl-5-nitroquinolin-2-yl)-1,3-tropolone (IVd) were synthesized as described above for compounds **IIIa** and **IVa** from 0.71 g of compound **IId**. The products were isolated by column chromatography on aluminum oxide (800×20 mm) using hexane–chloroform (2:1) as eluent.

Compound **IIId**. Yield 0.105 g (7%), yellow crystals, mp 250–252°C. IR spectrum: ν(C=O) 1647 cm^{-1}. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 s (9H,

5-*t*-Bu), 1.31 s (9H, 7-*t*-Bu), 2.78 s (3H, 8'-CH_3), 6.52 s (1H, 6-H), 7.58 d (1H, 7'-H, *J* = 8.5 Hz), 7.68 d (1H, 6'-H, *J* = 8.5 Hz), 8.40 s (1H, 3'-H), 18.40 br.s (1H, 3-OH). Found, %: C 60.02; H 5.11; Cl 7.13; N 8.29. $C_{25}H_{26}ClN_3O_6$. Calculated, %: C 60.06; H 5.24; Cl 7.09; N 8.40.

Compound **IVd**. Yield 0,068 g (5%), yellow crystals, mp 210–212°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.23 s (9H, 5-*t*-Bu), 1.37 s (9H, 7-*t*-Bu), 2.73 s (3H, 8'-CH_3), 6.68 d (1H, 4-H, *J* = 1.82 Hz), 6.83 d (1H, 6-H, *J* = 1.82 Hz), 7.57–7.64 m (2H, 6'-H, 7'-H), 8.32 s (1H, 3'-H), 18.02 s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 454.7 (7) $[M]^+$, 426 (92), 411 (100), 383 (28), 365 (38), 57 (46), 41 (48). Found, %: C 65.93; H 5.92; Cl 7.84; N 6.03. $C_{25}H_{27}ClN_2O_4$. Calculated, %: C 66.00; H 5.98; Cl 7.79; N 6.16. *M* 454.95.

5,7-Di-*tert*-butyl-2-(4-chloro-6,8-dimethyl-5-nitroquinolin-2-yl)-4-nitro-1,3-tropolone (IIIe) and 5,7-di-*tert*-butyl-2-(4-chloro-6,8-dimethyl-5-nitroquinolin-2-yl)-1,3-tropolone (IVe) were synthesized as described above for compounds **IIIa** and **IVa** from 0.75 g of compound **IIe**.

Compound **IIIe**. Yield 0,093 g (6%), yellow crystals, mp 250–252°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 s (9H, 5-*t*-Bu), 1.31 s (9H, 7-*t*-Bu), 2.43 s (3H, 6'-CH_3), 2.72 s (3H, 8'-CH_3), 6.47 s (1H, 6-H), 7.56 s (1H, 7'-H), 8.35 s (1H, 3'-H), 18.46 br.s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 513.9 (13) $[M]^+$, 485 (100), 467 (63), 437 (44), 91 (37), 57 (78), 41 (82). Found, %: C 60.68; N 5.39; Cl 6.94; N 8.11. $C_{26}H_{28}ClN_3O_6$. Calculated, %: C 60.76; H 5.49; Cl 6.90; N 8.18. *M* 513.97.

Compound **IVe**. Yield 0.042 g (3%), yellow crystals, mp 223–225°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.25 s (9H, 5-*t*-Bu), 1.38 s (9H, 7-*t*-Bu), 2.40 s (3H, 6'-CH_3), 2.70 s (3H, 8'-CH_3), 6.68 d (1H, 4-H, *J* = 1.82 Hz), 6.82 d (1H, 6-H, *J* = 1.82 Hz), 7.50 s (1H, 7'-H), 8.28 s (1H, 3'-H), 18.02 s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 468.8 (5) $[M]^+$, 440 (75), 425 (75), 397 (20), 379 (18), 91 (33), 57 (90), 41 (100). Found, %: C 66.64; H 6.21; Cl 7.41; N 5.92. $C_{26}H_{29}ClN_2O_4$. Calculated, %: C 66.59; H 6.23; Cl 7.56; N 5.97. *M* 468.98.

5,7-Di-*tert*-butyl-2-(4-chloro-7,8-dimethyl-5-nitroquinolin-2-yl)-4-nitro-1,3-tropolone (IIIf) and 5,7-di-*tert*-butyl-2-(4-chloro-7,8-dimethyl-5-nitroquinolin-2-yl)-1,3-tropolone (IVf) were synthesized as described above for compounds **IIIa** and **IVa** from 0.75 g of compound **IIf**.

Compound **IIIf**. Yield 0.108 g (7%), yellow crystals, mp 240–242°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.30 s (9H, 5-*t*-Bu), 1.32 s (9H, 7-*t*-Bu), 2.58 s (3H, 7'-CH_3), 2.65 s (3H, 8'-CH_3), 6.44 s (1H, 6-H), 7.52 s (1H, 6'-H), 8.32 s (1H, 3'-H), 18.29 br.s (1H, 3-OH). Found, %: C 60.66; H 5.45; Cl 6.96; N 8.09. $C_{26}H_{28}ClN_3O_6$. Calculated, %: C 60.76; H 5.49; Cl 6.90; N 8.18.

Compound **IVf**. Yield 0.056 g (4%), yellow crystals, mp 234–236°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.24 s (9H, 5-*t*-Bu), 1.39 s (9H, 7-*t*-Bu), 2.54 s (3H, 7'-CH_3), 2.65 s (3H, 8'-CH_3), 6.67 d (1H, 4-H, *J* = 1.87 Hz), 6.81 d (1H, 6-H, *J* = 1.87 Hz), 7.51 s (1H, 6'-H), 8.25 s (1H, 3'-H), 18.30 s (1H, 3-OH). Mass spectrum, *m*/*z* (I_{rel}, %): 468.8 (5) $[M]^+$, 440 (75), 425 (75), 397 (20), 379 (18), 91 (33), 57 (90), 41 (100). Found, %: C 66.62; H 6.13; Cl 7.52; N 5.93. $C_{26}H_{29}ClN_2O_4$. Calculated, %: C 66.59; H 6.23; Cl 7.56; N 5.97. *M* 468.98.

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