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Synthesis of Nitroacridinones from 2,1-Benzisoxazole Derivatives

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Abstract—Reaction of 3-aryl-2,1-benzisoxazoles with concentrated nitric acid in chloroform in one stage led to their conversion into acridinone nitro derivatives.

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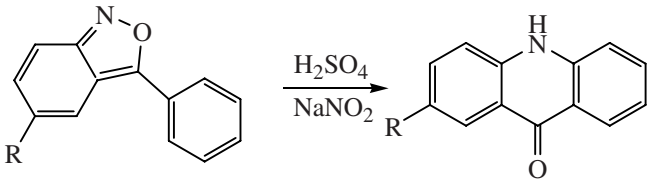
2,1-Benzisoxazoles (anthranils) are fused heteroaromatic systems applied as versatile synthons in the fine organic synthesis. This is due to the easy ring opening at the labile N–O bond and to the capability of 2,1-benzisoxazoles to transform into other heterocyclic systems, for instance, into acridinones extensively used in the production of dyes and pharmaceuticals [1]. With 3-aryl-2,1-benzisoxazoles photorearrangements are known giving 2-R-acridin-9(10*H*)-ones proceeding in acid, neutral, and alkaline media in very low yields [2–5], and also their conversions occurs at high temperature or at treating with sodium nitrite in concn. H_2SO_4 [6, 7].

We established that the latter reaction led to plausible yields only for 5-halo-3-phenyl-2,1-benz-isoxazoles.

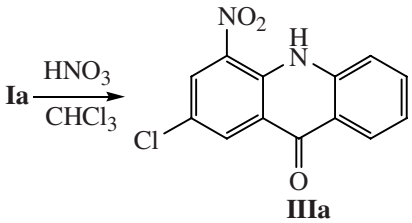


Ia–**Ic** **IIa**–**IIc**

R = Cl (**a**), Br (**b**), I (**c**).

At the use of initial compounds with other substituents the yield of target reaction products considerably decreased due to the alternative concurrent processes.

We formerly established [8] that at treating anthranil **Ia** with concn. HNO_3 or N_2O_4 in chloroform at room temperature as a result of transformation with simultaneous introducing of a nitro group formed 4-nitro-2-chloroacridin-9(10*H*)-one (**IIIa**).



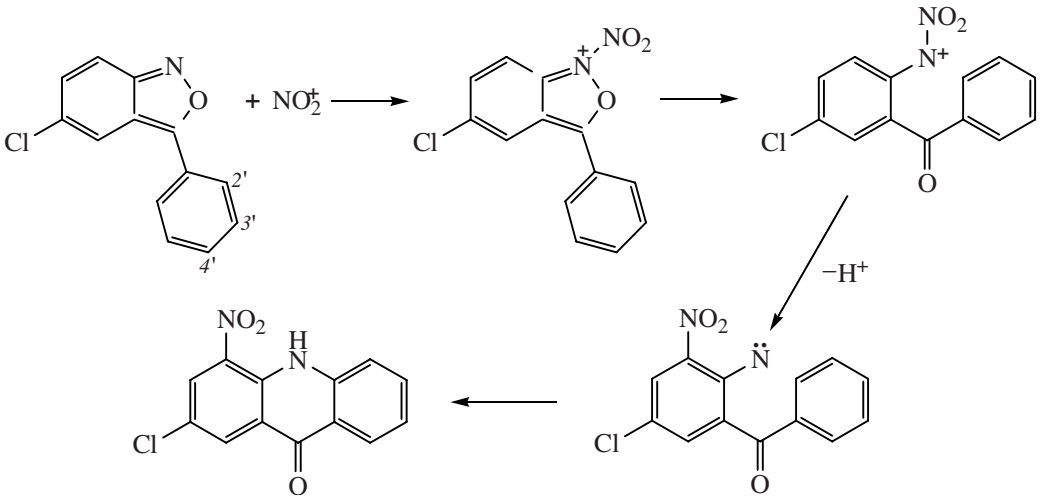
IIIa

To confirm the assumption [8] that under the used conditions first formed 7-nitro-3-(2-nitrophenyl)-5-chloro-2,1-benzisoxazole which suffered further transformation into acridinone **IIIa** we performed a quantum-chemical study of initial anthranil **Ia** by a semiempirical method AM1 (program package MOPAC 7.0).

The initial anthranil at nitration is subjected to electrophilic attack, therefore its reactivity can be characterized by the structure and the energy of HOMO or by the chrge distribution on the atoms of the molecule.

Comparing the charges on the atoms of the compound under investigation **Ia** (Table 1) and the experimental data it is seen that the charge factor does not influence the direction of the electrophilic attack for the most negatively charged atoms are not reaction site (are not attacked by the electrophile under the conditions of reaction). Therefore we suggested that the process in question is orbital-controlled. The main contribution into the HOMO of anthranil **Ia** makes the nitrogen of the heterocycle. In all likelihood just this atom is subjected to the electrophilic attack leading to the rupture of the labile N–O bond and not the atom $C^{2'}$ as has been presumed before [8]. In the subsequent stages occurred the nitro group migration and the acridine ring formation.

The conversion of 3-phenyl-5-chloro-2,1-benzisoxazole (**Ia**) in acridinone **IIIa** occurred also at the use of a radical species, nitrogen dioxide. In this reaction

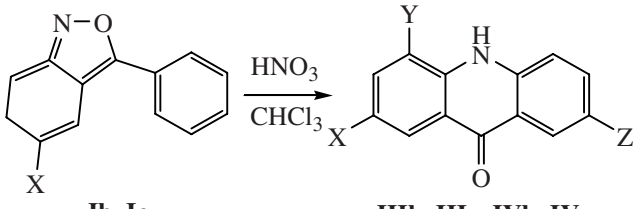


the reactivity of the molecule of anthranil **Ia** is characterized by the energy and structure of LUMO. The maximum contribution into LUMO of 2,1-benzisoxazole **Ia** makes also the nitrogen of the heterocycle which is subjected to the attack of the radical species resulting in the N–O bond rupture.

To reveal the applicability limits of the reaction system concn. HNO_3–chloroform in the synthesis of nitroacridinones we carried out a quantum-chemical simulation in the framework of the semiempirical method AM1, and then the behavior of a series of 2,1-benzisoxazole derivatives was studied under the described conditions.

The consideration of the HOMO structure of compounds under study (Table 2) demonstrated that the main contribution into HOMO of all anthranils originated from the nitrogen of the heterocycle. Therefore they all should be prone to this type of transformations. A special importance has the energy values of HOMO (Table 2) which permit prediction of the reactivity. The highest level of HOMO energy, and thus the reactivity in these processes have 2,1-benzisoxazoles **Ia** and **Id**.

We experimentally established that into the corresponding nitroacridinones anthranils were converted containing in the basic structure various halogen atoms.



Ib, Ic **IIIb, IIIc, IVb, IVc**

I, X = Br (**b**), I (**c**); **III**, X = Br, Y = NO_2, Z = H (**b**); X = I, Y = H, Z = NO_2 (**c**); **IV**, X = Br, Y = Z = NO_2 (**b**); X = I, Y = Z = NO_2 (**c**).

The halogen atoms ambiguously affect the course of the process. Mononitration of 3-phenyl-5-chloro-2,1-benzisoxazole (**Ia**) occurred at any ratio anthranil–nitration agent, and always formed 4-nitro-2-chloroacridin-9(10*H*)-one (**IIIa**). Its further nitration under the studied conditions was not observed. From 5-bromo- and 5-iodo-3-phenyl-2,1-benzisoxazoles (**Ib** and **Ic**), by varying the ratio anthranil–nitration agent it was possible

Table 1. The charges on atoms and contributions of atomic orbitals into HOMO of 3-phenyl-5-chloro-2,1-benzisoxazole (**Ia**) (AM1)

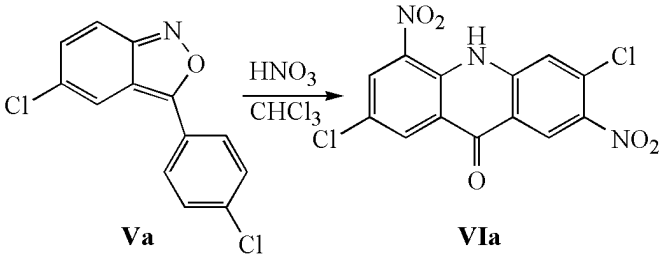
Atoms	Charges on atoms, *e*	C_{Pz}^2
O^1	–0.0319	0.0010
N^2	–0.0192	0.1619
C^3	–0.0865	0.1013
C^4	–0.1038	0.0245
C^6	–0.0696	0.1153
$C^{2'}$	–0.0994	0.0337
$C^{3'}$	–0.1341	0.0074
$C^{4'}$	–0.1073	0.0543

Table 2. HOMO energy and contribution of atomic orbitals of N^2 atoms into HOMO of 2,1-benzisoxazoles (AM1)

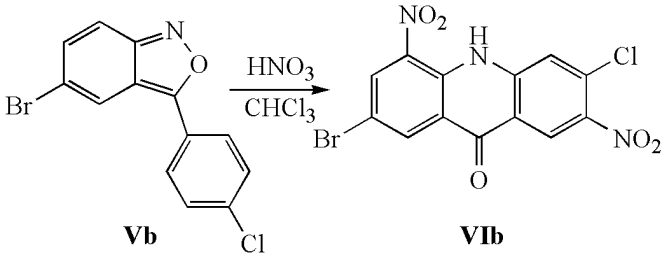
Compound no.	C_{Pz}^2	HOMO energy, eV
Ia	0.1619	–8.659
Ib	0.1669	–8.708
Ic	0.1726	–8.729
Id	0.1811	–8.616
Va	0.1542	–8.743
IX	0.1685	–8.787

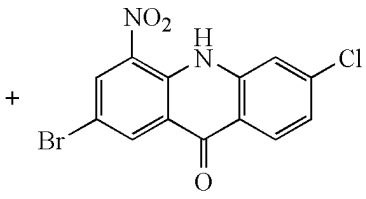
to obtain the corresponding nitro- and dinitroacridinones **IIIb, IIIc** and **IVb, IVc**. Therewith the nitro group in iodonitroacridinone **IIIc** is located in another position.

In the presence of a chlorine in the *para*-position of the phenyl substituent (compounds **Va–Vc**) the pattern of the reaction between initial anthranil and nitric acid in chloroform complicated. In reaction of 5-chloro-3-(4-chlorophenyl)-2,1-benzisoxazole (**Va**) independent of the ratio anthranil–nitration agent always formed 2,5-dinitro-3,7-dichloro-acridin-9(10*H*)-one (**VIa**).



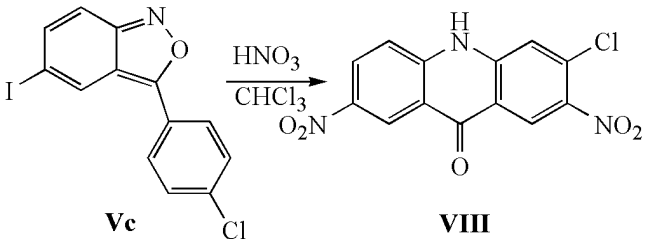
From 5-bromo-3-(4-chlorophenyl)-2,1-benzisoxazole (**Vb**) under the same conditions a mixture was obtained of 2-bromo-4-nitro-7-chloro- and 7-bromo-2,5-dinitro-3-chloroacridin-9(10*H*)-ones (**VII**) and (**VIb**) in a ratio 2:1.



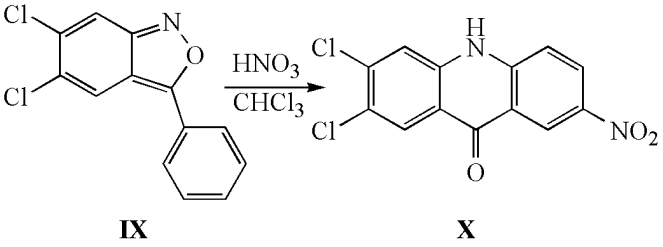


VII

In the reaction of 5-iodo derivative **Vc** alongside the heterocycles transformation accompanied by nitration a formal displacement of iodine by a nitro group occurred presumably through oxidative elimination of iodine.

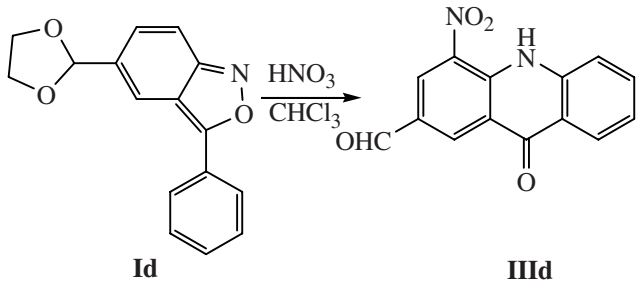


The nitration of 5,6-dichloro-3-phenyl-2,1-benzisoxazole (**IX**) also resulted in nitroacridinone.



The introduction of one more chlorine atom into the para-position of the phenyl moiety of the substrate prevented the transformation, and from the reaction mixture the initial 5,6-dichloro-3-(4-chlorophenyl)-2,1-benzisoxazole was recovered.

In the presence of a dioxolane fragment in the position *5* of the anthranil (compound **Id**) simultaneously with introducing the nitro group and transformation into acridinone occurred the degradation of the dioxolane ring into a carbonyl function.



The appearance of methoxy group in the basic structure of anthranil complicated the transformation under the chosen conditions. For instance, from 3-(4-methoxyphenyl)- and 3-(3,4-dimethoxyphenyl)-5-chloro-2,1-benzisoxazoles formed resinous mixtures which after numerous recrystallizations permitted isolation of traces of initial compounds.

Symmetric binuclear anthranils with an oxide [3-phenyl-5-(3-phenyl-2,1-benzisoxazol-5-yloxy)-2,1-benzisoxazole] or sulfide [3-phenyl-5-(3-phenyl-2,1-

benzisoxazol-5-ylsulfanyl)-2,1-benzisoxazole] bridging fragments in the system in question suffered destructive processes (bridge rupture) resulting in intractable tars.

The applied reaction conditions proved to be suitable for transformation of an anthranil ring fused with a benzofuran (compound **XI**).

In all cases under consideration no nitroanthranils were detected in the reaction products.

EXPERIMENTAL

^{1}H NMR spectra were registered on a spectrometer Bruker AC-300 (300.13 MHz) in DMSO-d_6, internal reference TMS. IR spectra were recorded on a spectrophotometer Specord M-80 from mulls in mineral oil. Elemental composition was determined on an analyzer CHN-1 (Czechia). Mass spectra were measured on MKh-1310 instrument at ionizing electrons energy 70 eV. The composition of the reaction mixtures was monitored and the purity of compounds was checked by TLC on Silufol UV-254 plates, development under UV irradiation. The melting points were measured on PG HS 30 A/G instrument (DDR).

2,1-Benzisoxazoles were synthesized by procedure [9].

Acridinones IIa–IIc. To a solution of 10 mmol of an appropriate 2,1-benzisoxazole in 100 ml of concn. H_2SO_4 was added at cooling while stirring 10 g (0.15 mol) of sodium nitrite. The mixture was stirred for 20 h at room temperature, then poured into water, the precipitate was filtered off, washed with water, dried, and recrystallized from AcOH.

2-Chloroacridin-9(10*H*)-one (IIa). Yield 1.8 g (78%), mp >300°C. IR spectrum, ν, cm^{-1}: 3264 (NH), 1628 (C=O). ^{1}H NMR spectrum, δ, ppm: 11.79 s (1H, NH), 8.22 d.d (1H, H^3), 8.16 d (1H, H^1), 7.72–7.78 m (2H, H^{6,8}), 7.55–7.60 m (2H, H^{4,5}), 7.28 m (1H, H^7). Mass spectrum, *m/z* (I_{rel}, %): 229 (100) $[M]^+$, 201 (10.2) $[M - CO]^+$, 166 (21.1), 139 (13.2). Found, %: C 67.98; H 3.46; N 5.97. $C_{13}H_8ClNO$. Calculated, %: C 67.99; H 3.51; N 6.10. *M* 229.66.

2-Bromoacridin-9(10*H*)-one (IIb). Yield 1.6 g (58%), mp >300°C. IR spectrum, ν, cm^{-1}: 3266 (NH), 1626 (C=O). ^{1}H NMR spectrum, δ, ppm: 11.75 s (1H, NH), 8.20 d.d (1H, H^3), 8.13 d (1H, H^1), 7.71–7.80 m (2H, H^{6,8}), 7.52–7.59 m (2H, H^{4,5}), 7.27 m (1H, H^7). Mass spectrum, *m/z* (I_{rel}, %): 273 (100) $[M]^+$. Found, %: C 56.92; H 2.88; N 5.19. $C_{13}H_8BrNO$. Calculated, %: C 56.96; H 2.94; N 5.11. *M* 274.12.

2-Iodoacridin-9(10*H*)-one (IIc). Yield 1.5 c (47%), mp >300°C. IR spectrum, ν, cm^{-1}: 3264 (NH), 1632 (C=O). ^{1}H NMR spectrum, δ, ppm: 11.70 s (1H, NH), 8.12 d.d (1H, H^3), 8.10 d (1H, H^1), 7.67–7.72 m (2H, H^{6,8}), 7.25–7.29 m (2H, H^{4,5}), 7.20 m (1H, H^7). Mass spectrum, *m/z* (I_{rel}, %): 321 (100) $[M]^+$. Found, %: C 48.60; H 2.45; N 4.24. $C_{13}H_8INO$. Calculated, %: C 48.63; H 2.51; N 4.36. *M* 321.11.

Nitroacridin. To a solution of 5 mmol of anthranil in 30 ml of $CHCl_3$ was slowly added dropwise at cooling in the course of mononitration 0.1 ml (5 mmol) of HNO_3 (d_4^{20} 1.49 g/cm^3), of dinitration, 0.2 ml (10 mmol) of HNO_3. The reaction mixture was stirred for 1 h at room temperature. The separated precipitate was filtered off, washed with water, and recrystallized from a mixture 2-propanol–DMF, 3:1.

4-Nitro-2-chloroacridin-9(10*H*)-one (IIIa). Yield 1.13 g (82%), mp >300°C. IR spectrum, ν, cm^{-1}: 3330 (NH), 1670 (C=O), 1510 [$\nu_{as}(NO_2)$], 1350 [$\nu_s(NO_2)$]. ^{1}H NMR spectrum, δ, ppm: 12.39 s (1H, NH), 8.95 d (1H, H^3, *J* 2.4 Hz), 8.47 d.d (1H, H^8, *J* 8.0 Hz), 8.15 d (1H, H^1, *J* 2.4 Hz), 7.84 d.d (1H, H^5, *J* 8.0 Hz), 7.61–7.71 m (2H, H^{6,7}). Mass spectrum, *m/z* (I_{rel}, %): 274 (22.1) $[M]^+$, 244 (8.5) $[M - NO]^+$, 228 (16.1) $[M - NO_2]^+$, 164 (14.9), 44 (100). Found, %: C 56.73; H 2.45; N 10.12. $C_{13}H_7ClN_2O_3$. Calculated, %: C 56.85; H 2.57; N 10.20. *M* 274.66.

2-Bromo-4-nitroacridin-9(10*H*)-one (IIIb). Yield 0.78 g (49%), mp >300°C. IR spectrum, ν, cm^{-1}: 3333 (NH), 1667 (C=O), 1512 [$\nu_{as}(NO_2)$], 1353 [$\nu_s(NO_2)$]. ^{1}H NMR spectrum, δ, ppm: 12.09 s (1H, NH), 8.76 d (1H, H^3, *J* 2.4 Hz), 8.51 d.d (1H, H^8, *J* 8.0 Hz), 8.85 d (1H, H^1, *J* 2.4 Hz), 7.78 d.d (1H, H^5, *J* 8.0 Hz), 7.60–7.68 m (2H, H^{6,7}). Mass spectrum, *m/z* (I_{rel}, %): 318 (21.0) $[M]^+$, 273 (100) $[M - NO_2]^+$, 194 (28.9), 166 (32.5), 139 (38.6). Found, %: C 48.79; H 2.43; N 8.61. $C_{13}H_7BrN_2O_3$. Calculated, %: C 48.93; H 2.21; N 8.78. *M* 319.11.

2-Iodo-7-nitroacridin-9(10*H*)-one (IIIc). Yield 0.6 g (33%), mp >300°C. IR spectrum, ν, cm^{-1}: 3332 (NH), 1666 (C=O), 1513 [$\nu_{as}(NO_2)$], 1355 [$\nu_s(NO_2)$]. ^{1}H NMR spectrum, δ, ppm: 12.45 s (1H, NH), 8.94 d (1H, H^8, *J* 2.4 Hz), 8.48 d.d (1H, H^6, *J* 8.0 Hz), 8.46 d (1H, H^1, *J* 2.4 Hz), 8.07 d.d (1H, H^3, *J* 8.0 Hz), 7.66 d (1H, H^5, *J* 8.0 Hz), 7.41 d (1H, H^4, *J* 8.0 Hz). Mass spectrum, *m/z* (I_{rel}, %): 366 (100) $[M]^+$, 320 (33.4) $[M - NO_2]^+$, 240 (72.2), 193 (80.5), 164 (98.6), 139 (64.6). Found, %: C 42.51; H 1.87; N 7.78. $C_{13}H_7IN_2O_3$. Calculated, %: C 42.65; H 1.93; N 7.65. *M* 366.11.

4-Nitroacridin-9(10*H*)-one-2-carbaldehyde (IIId). Yield 0.84 g (63%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 12.59 s (1H, CHO), 10.03 s (1H, NH), 8.87 d (1H, H^3, *J* 2.4 Hz), 8.69 d (1H, H^1, *J* 2.4 Hz), 8.46 d.d (1H, H^8, *J* 8.0 Hz), 8.16 d.d (1H, H^5, *J* 8.0 Hz), 7.61–7.67 m (2H, H^{6,7}). Mass spectrum, *m/z* (I_{rel}, %): 268 (100) $[M]^+$, 238 (20.4) $[M - NO]^+$, 222 (41.4) $[M - NO_2]^+$, 193 (40.7), 164 (42.6), 139 (20.6). Found, %: C 62.52; H 3.14; N 10.31. $C_{14}H_8N_2O_4$. Calculated, %: C 62.70; H 3.01; N 10.44. *M* 268.23.

2-Bromo-4,7-dinitroacridin-9(10*H*)-one (IVb). Yield 0.89 g (49%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 11.81 d (1H, NH), 8.92 d (1H, H^3, *J* 2.4 Hz), 8.78 d (1H, H^8, *J* 2.4 Hz), 8.67 d (1H, H^1, *J* 2.4 Hz), 8.56 d.d (1H, H^6, *J* 8.0 Hz), 8.28 d.d (1H, H^5, *J* 8.0 Hz). Mass spectrum, *m/z* (I_{rel}, %): 363 (100) $[M]^+$, 335 (18.6) $[M - NO]^+$, 319 (21.4) $[M - NO_2]^+$, 271 (30.1), 192 (13.8), 164 (32.0). Found, %: C 43.03; H 1.48; N 11.73. $C_{13}H_6BrN_3O_5$. Calculated, %: C 42.88; H 1.66; N 11.54. *M* 364.11.

2-Iodo-4,7-dinitroacridin-9(10*H*)-one (IVc). Yield 0.76 g (37%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 12.36 s (1H, NH), 8.98 d (1H, H^8, *J* 2.4 Hz), 8.55 d.d (1H, H^6, *J* 8.0 Hz), 8.47 d (1H, H^3, *J* 2.4 Hz), 7.73 d (1H, H^5, *J* 8.0 Hz), 7.69 d (1H, H^1, *J* 2.4 Hz). Mass spectrum, *m/z* (I_{rel}, %): 411 (100) $[M]^+$, 365 (12.4) $[M - NO_2]^+$. Found, %: C 38.12; H 1.31; N 10.39. $C_{13}H_6IN_3O_5$. Calculated, %: C 37.98; H 1.47; N 10.22. *M* 411.11.

2,5-Dinitro-3,7-dichloroacridin-9(10*H*)-one (VIa). Yield 1.1 g (62%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 11.71 s (1H, NH), 8.76 d (1H, H^1, *J* 2.4 Hz), 8.71 d (1H, H^6, *J* 2.4 Hz), 8.53 d (1H, H^8, *J* 2.4 Hz), 8.51 d (1H, H^4, *J* 2.4 Hz). Mass spectrum, *m/z* (I_{rel}, %): 353 (30.4) $[M]^+$, 323 (18.7) $[M - NO]^+$, 277 (10.1), 261 (24.5), 198 (69.9), 162 (38.6), 111 (33.3). Found, %: C 44.21; H 1.29; N 11.72. $C_{13}H_5Cl_2N_3O_5$. Calculated, %: C 44.09; H 1.42; N 11.87. *M* 354.11.

7-Bromo-2,5-dinitro-3-chloroacridin-9(10*H*)-one (VIb). Yield 0.42 g (21%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 11.70 s (1H, NH), 8.78 d (1H, H^6, *J* 2.4 Hz), 8.73 d (1H, H^1, *J* 2.4 Hz), 8.59 d (1H, H^8, *J* 2.4 Hz), 8.51 d (1H, H^4, *J* 2.4 Hz). Mass spectrum, *m/z* (I_{rel}, %): 397 (13.6) $[M]^+$, 354 (58.7) $[M - NO_2]^+$, 308 (41.2), 198 (39.9), 164 (100), 111 (23.2). Found, %: C 44.31; H 1.56; N 7.82. $C_{13}H_6BrClN_2O_3$. Calculated, %: C 44.16; H 1.71; N 7.92. *M* 398.55.

2-Bromo-4-nitro-6-chloroacridin-9(10*H*)-one (VII). Yield 0.66 g (37%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 12.34 s (1H, NH), 8.75 d (1H, H^3, *J* 2.4 Hz), 8.19 d (1H, H^5, *J* 2.4 Hz), 7.92 d.d (1H, H^7, *J* 8.0 Hz), 7.64 d (1H, H^1, *J* 2.4 Hz), 7.48 d.d (1H, H^8, *J* 8.0 Hz). Mass spectrum, *m/z* (I_{rel}, %): 352 (61.5) $[M]^+$, 306 (45.4) $[M - NO_2]^+$. Found, %: C 39.29; H 1.09; N 10.68. $C_{13}H_5BrClN_3O_5$. Calculated, %: C 39.18; H 1.26; N 10.54. *M* 353.55.

2,7-Dinitro-3-chloroacridin-9(10*H*)-one (VIII). Yield 1.26 g (79%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 12.71 s (1H, NH), 8.83 d (1H, H^1, *J* 2.4 Hz), 8.75 d (1H, H^8, *J* 2.4 Hz), 8.52 d.d (1H, H^6, *J* 8.0 Hz), 7.68 d (1H, H^4, *J* 2.4 Hz), 7.67 d.d (1H, H^5, *J* 8.0 Hz). Mass spectrum, *m/z* (I_{rel}, %): 319 (98.1) $[M]^+$, 289 (32.6) $[M - NO]^+$, 274 (51.2), 227 (100), 164 (82.3), 149 (48.8), 137 (22.4). Found, %: C 48.68; H 2.02; N 13.29. $C_{13}H_6ClN_3O_5$. Calculated, %: C 48.85; H 1.89; N 13.15. *M* 319.66.

7-Nitro-2,3-dichloroacridin-9(10*H*)-one (X). Yield 0.79 g (51%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 12.50 s (1H, NH), 8.91 d (1H, H^8, *J* 2.4 Hz), 8.49 d.d (1H, H^6, *J* 8.0 Hz), 8.28 d (1H, H^1), 7.77 d (1H, H^4), 7.67 d.d (1H, H^5, *J* 8.0 Hz). Mass spectrum, *m/z* (I_{rel}, %): 308 (100) $[M]^+$, 278 (19.6) $[M - NO]^+$, 262 (47.2) $[M - NO_2]^+$, 222 (20.2), 199 (22.3), 164 (24.8), 121 (82.4). Found, %: C 50.34; H 2.09; N 9.19. $C_{13}H_6Cl_2N_2O_3$. Calculated, %: C 50.51; H 1.96; N 9.06. *M* 309.11.

4-Nitro-3-chlorobenzofuro[2,3-*a*]acridin-13(5*H*)-one (XII). Yield 1.4 g (77%), mp >300°C. ^{1}H NMR spectrum, δ, ppm: 9.41 s (1H, NH), 8.38–8.42 m (2H, H^{7,8}), 8.40 d.d (1H, H^1, *J* 8.0 Hz), 7.82–7.88 m (2H, H^{6,11}), 7.80 d.d (1H, H^2, *J* 8.0 Hz), 7.52–7.62 m (2H, H^{9,10}). Mass spectrum, *m/z* (I_{rel}, %): 364 (20.1) $[M]^+$, 318 (9.1) $[M - NO_2]^+$, 290 (58.3), 149 (24.3), 139 (69.5), 111 (100). Found, %: C 62.41; H 2.61; N 7.49. $C_{19}H_9ClN_2O_4$. Calculated, %: C 62.57; H 2.49; N 7.68. *M* 364.74.

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