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Reaction of Substituted Spiro[1,2,3,4-tetrahydronaphthalene-2,3'-(1'-pyrazolines)] with Chlorinating Reagents

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Abstract—In reaction of 4'-arylspiro[1,2,3,4-tetrahydronaphthalene-2,3'-(1'-pyrazolin)]-1-ones with *N*-chlorosuccinimide formed spirocyclic substituted 3-chloro-1-pyrazolines that lost nitrogen at heating transforming into spirocyclic chlorocyclopropanes. The reaction of the same pyrazolines with chlorine led to the formation of spirocyclic gem-dichlorocyclopropanes.

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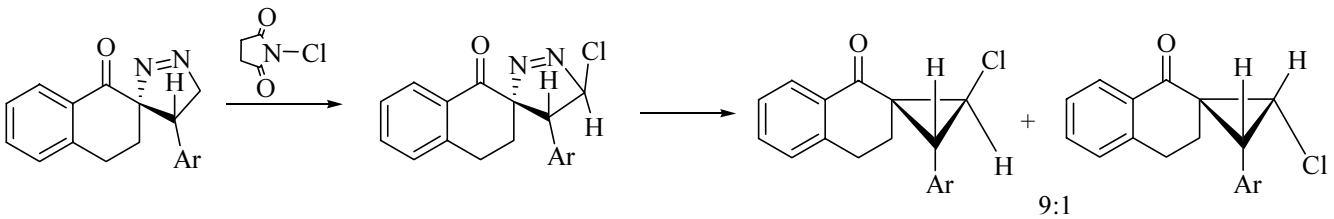
Former studies demonstarated that the reaction of bicyclic and spirocyclic 2-pyrazolines with the halogenating reagents gave rise to 3-halo-1-pyrazolines which on heating eliminated nitrogen resulting in 1-halocyclopropane-1-carboxylates [1–7]. The halogenation of spirocyclic 1-pyrazolines prepared by treating with diazomethane the itaconic acid imide led to the formation of spirocyclic mono- and dihalocyclopropanes [8].

In the present study we investigated the reaction with *N*-chlorosuccinimide and chlorine of 4'-arylspiro[1,2,3,4-tetrahydronaphthalene-2,3'-(1'-pyrazolin)]-1-ones **Ia–Ic** prepared by treating with diazomethane the (*E*)-2-arylmethylene-1-tetralones [9]. From the products obtained in the reaction of compounds **Ia–Ic** with the *N*-chlorosuccinimide we isolated 4'-aryl-3'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,5'-(1'-pyrazolin)]-1-ones **IIa–IIc** in 25–46% yields.

The structure of compounds of **IIa–IIc** was established from the elemental analysis and spectral data. In the ^{1}H NMR spectra of compounds **IIa–IIc** were observed doublet signals at 6.30–7.08 and 4.04–4.41 ppm (*J* 9 Hz) belonging to protons H*3*' and H*4*', respectively, the signals of protons from the CH_2CH_2 group, and also the signals from aromatic protons. In the ^{13}C NMR spectrum of compound **IIa** appeared signals at 26.6 (CH_2), 31.5 (CH_2), 51.0 (CH), 95.6 (CH), 101.0 (C), 190.8 (CO) ppm, and also the signals of carbon atoms from the aromatic rings. The absorption band of the carbonyl group in the IR spectra was observed at 1700 cm^{-1}.

The heating of pyrazolines **IIa–IIc** in toluene at 105–110°C induced nitrogen liberation, and a mixture formed containing stereoisomeric 1'-aryl-2'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-ones **IIIa–IIIc** and **IVa–IVc** in a 51–61% yield at a ratio ~9:1 (Scheme 1). After heating the pyrazolines prepared by the same procedures starting with a mixture of (*Z*)- and (*E*)-1-tetralones (**A** ratio 3:1) the isolated mixture of substances according to the 1H NMR spectrum contained mainly chlorocyclo-propane **V** (Scheme 2).

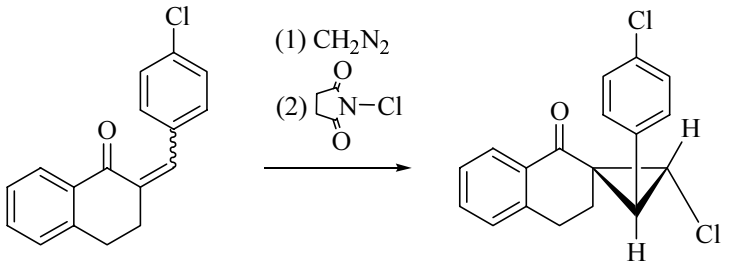
Scheme 1.



Ia–Ic **IIa–IIc** **IIIa–IIIc** **IVa–IVc**

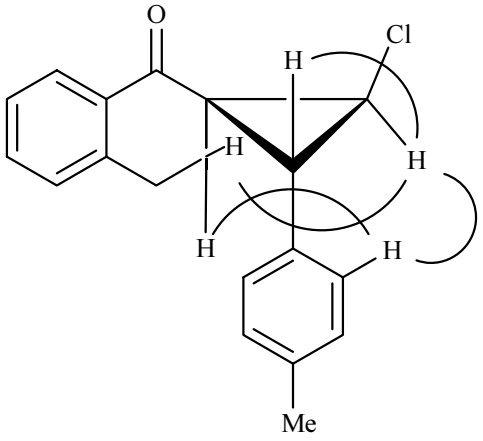
Ar = Ph (**a**), 4-ClC_6H_4 (**b**), 4-MeC_6H_4 (**c**).

Scheme 2.

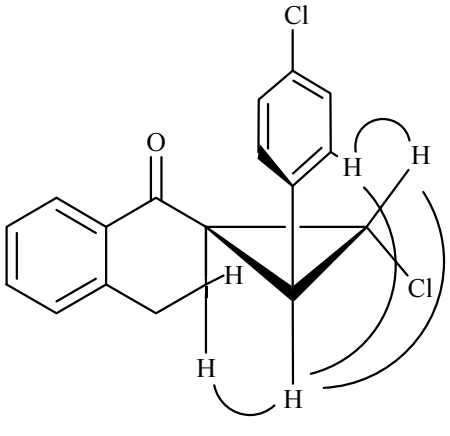


A **V**

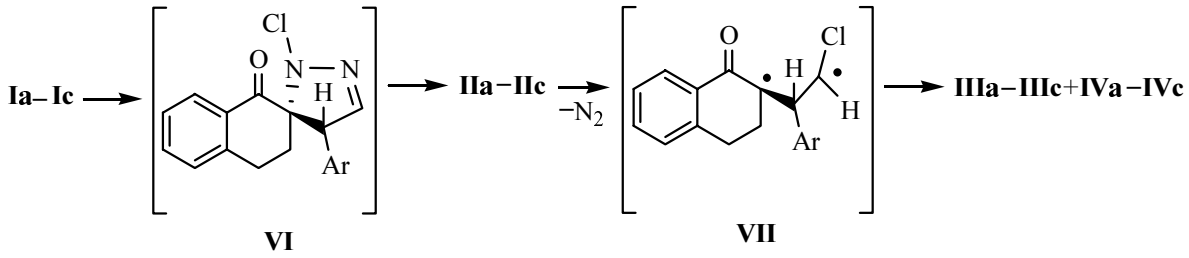
The composition and structure of compounds was established from the elemental analysis and spectral data. In the ^{1}H NMR spectra of stereoisomers **IIIa–IIIc** appeared the doublet signals belonging to cyclopropane protons $H^{1'}$ in the region 3.71–3.75 and $H^{2'}$ in the region 3.79–3.84 ppm with a coupling constant 5 Hz, and also signals of protons from the CH_2CH_2 group and aromatic protons. In the ^{13}C NMR spectrum of compound **IIIc** the signals were observed at 21.5 (CH_3), 28.1 (CH_2), 28.6 (CH_2), 36.5 (CH), 40.4 (C), 43.1 (CH), 192.1 (CO) ppm, and also the signals from the aromatic carbons. In the IR spectra the absorption bands of the carbonyl group appear at 1670 cm^{-1}. In the ^{1}H NMR spectra of stereoisomers **IVa**–**IVc** the doublet signals of cyclopropane protons $H^{1'}$ and $H^{2'}$ are observed at 3.39 and 3.93 ppm with a coupling constants 8 Hz. It is known that in the cyclopropane compounds the larger value of the coupling constant is observed at the *cis*-position of the protons, and the the smaller value corresponds to the *trans*-position [9]. Besides in the ^{1}H–^{1}H NOESY spectrum of compound **IIIc** cross-peaks appear corresponding to the coupling of the protons of the cyclopropane ring with the *ortho*-protons of the phenyl group, to the coupling of CH_2CH_2 group protons of the tetrahydronaphthalene ring with the $H^{2'}$ proton of the cyclopropane ring and the *ortho*-protons of the phenyl group. A cross peak is lacking that should correspond to the coupling of CH_2CH_2 group protons of the tetrahydronaphthalene ring with the $H^{1'}$ of the cyclopropane ring. Therefore isomer **III** has presumably a structure with a *trans*-position of hydrogen atoms, and isomer **IV**, with a *cis*-position. In the ^{1}H NMR spectrum of isomer **V** the doublet signals of the three-membered ring protons are observed at 2.85 ($H^{1'}$, *J* 5.8 Hz) and 4.63 ppm ($H^{2'}$, *J* 5.8 Hz). As seen, the proton signal of the atom in the free-membered ring in the *gem*-position with respect to a chlorine is shifted downfield compared to the signals of the corresponding protons in **IIIb** and **IVb** isomers because of the deshielding effect of the carbonyl group, and the signal of $H^{1'}$proton situated in the *gem*-position relative to aryl group is shifted upfield compared to the corresponding proton signals of **IIIb** and **IVb** isomers. In the ^{13}C NMR spectrum of compound **V** the carbon signals of the three-membered ring are observed at 41.6 (CH), 42.9 (C) and 44.0 (CH) ppm. In the ^{1}H–^{1}H NOESY spectrum of this compound the observed cross-peaks correspond to the coupling of the *ortho*-protons of the phenyl group with the protons of the three-membered ring, to the coupling of protons of C^4H_2 group with the $H^{1'}$ proton in the cyclopropane ring, and the cross-peak of coupling between protons of the



IIIc



V



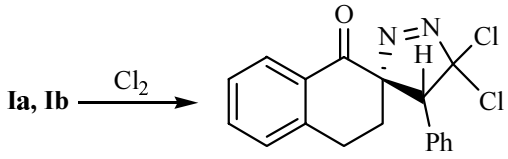
C^4H_2 group and the $H^{2'}$ of the three-membered ring is lacking. These data suggest that compound **V** has a configuration with a *trans*-position of hydrogens in the three-membered ring and a *syn*-position of the aromatic ring and the carbonyl of the tetrahydronaphthalene fragment.

Therefore a mechanism may be suggested where the primarily formed N-chloropyrazoline **VI** isomerizes into 3-chloropyrazoline **II** which loosing a nitrogen molecule forms **VII**; the subsequent cyclization leads to the formation of compounds **III** and **IV**.

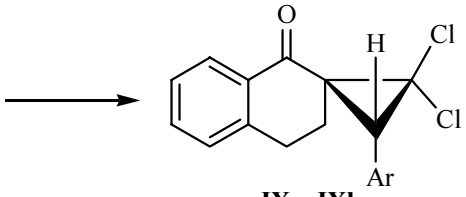
The reaction of 1-pyrazoline **Ia** with excess chlorine in chloroform at cooling with an ice bath gave 4'-phenyl-3',3'-dichlorospiro[1,2,3,4-tetrahydronaphthalene-2,5'-(1'-pyrazolin)]-1-one (**VIII**) in a 24% yield. In the ^{1}H NMR spectrum of the compound appeared a singlet from the $H^{4'}$ proton at δ 4.59 ppm, and also signals of the aromatic protons and the protons of the CH_2CH_2 group. In the ^{13}C NMR spectrum the signals were observed at δ 26.1 (CH_2), 30.8 (CH_2), 56.2 (CH), 101.4 (C), 114.6 (C), 189.7 (CO) ppm, and also the signal of the carbons in the aromatic rings.

The heating of pyrazoline **VIII** at 60–65°C induced a nitrogen liberation giving rise to 2'-phenyl-1',1'-dichlorospiro(1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan)-1-one (**IXa**) in a 55% yield. The composition and structure of the compound was established from the elemental analysis and spectral data. The ^{1}H NMR spectrum contained a singlet from the $H^{2'}$ proton at 4.03 ppm, and also signals of the methylene and aromatic protons. In the ^{13}C NMR spectrum appeared signals at 27.8 (CH_2), 28.0 (CH_2), 38.6 (CH), 44.4 (C), 66.8 (C), 190.9 (CO) ppm, and also the signals of the carbons in the aromatic rings. The carbonyl group vibrations gave rise in the IR spectrum to an absorption band at 1690 cm^{-1}; the absorption bands from the saturated and aromatic fragments were also observed.

The reaction of 1-pyrazoline **Ib** with chlorine in chloroform at cooling with an ice bath gave a compound detected by TLC which on warming to the room temperature eliminated nitrogen. As a result we isolated in a 19% yield 1',1'-dichloro-2'-(4-chlorophenyl)spiro(1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan)-1-one (**IXb**). The composition and structure of compound **IXb** was established from the elemental analysis and spectral data. The ^{1}H NMR spectrum contained a singlet from the $H^{2'}$



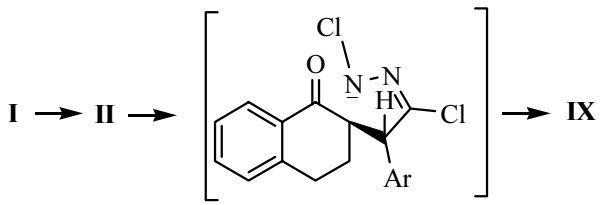
VIII



IXa, IXb

proton at δ 3.97 ppm, and also signals of the methylene and aromatic protons. In the ^{13}C NMR spectrum appeared signals at 27.7 (CH_2), 27.9 (CH_2), 37.9 (CH), 44.4 (C), 66.5 (C) ppm, and also the signals of the carbons in the aromatic rings. The carbonyl group vibrations gave rise in the IR spectrum to an absorption band at 1726 cm^{-1}; the absorption bands from the aromatic fragments were also observed.

The formation of dichloropyrazolines is tentatively presented on the following scheme.



Ar = Ph (**a**), 4-ClPh (**b**).

EXPERIMENTAL

IR spectra of compounds were recorded on a spectrophotometer UR-20 or Specord 75 IR from 2% solutions

in $CHCl_3$. ^1H and ^{13}C NMR spectra were registered on a spectrometer Bruker DPX-300 at operating frequencies 300.13 and 75.47 MHz respectively. The purity of substances was checked and the reaction mixtures were analyzed by TLC on Silufol UV-254 plates.

4'-Arylspiro[1,2,3,4-tetrahydronaphthalene-2,3'-(1'-pyrazolin)]-1-ones were prepared by procedure [10].

4'-Aryl-3'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,5'-(1'-pyrazolin)]-1-ones IIa–IIc. To a mixture of 20 ml of chloroform and 10 ml of acetic acid was added 8 mmol of 1-pyrazoline **Ia–Ic** and 1.3 g (10 mmol) of *N*-chlorosuccinimide. The reaction mixture was heated for 2 h at 60°C. Within this time the *N*-chlorosuccineimide dissolved completely, and the solution turned greenish. The reaction mixture was cooled to room temperature, washed with water, with a solution of $NaHCO_3$, and again with water. The organic phase was dried over Na_2SO_4 for 2 h (the long drying resulted in the decomposition of the product). Chloroform was evaporated, 2 ml of ethanol was added, and the precipitate was filtered off.

4'-Phenyl-3'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,5'-(1'-pyrazolin)]-1-one (IIa). Yield 38%, mp 130°C (decomp.). IR spectrum, cm^{-1}: 940, 1160, 1250, 1280 s, 1450, 1600, 1700 v.s, 2940, 3040. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.80 m (1H), 1.93 d.d.d (1H, *J* 14, 5, 4), 2.83 d.t (1H, *J* 17, 4), 3.46 d.d.d (1H, *J* 17, 12, 5), 4.41 d (1H, *J* 9), 6.38 d (1H, *J* 9), 7.16 m (2H), 7.32 m (4H), 7.41 t (1H, *J* 7), 7.58 t (1H, *J* 7), 8.19 d (1H, *J* 7). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 26.6 (CH_2), 31.5 (CH_2), 51.0 (CH), 95.6 (CH), 101.0 (C), 127.6 (CH), 128.5 (CH), 128.7 (CH), 129.3 (CH), 129.35 (CH), 129.41 (CH), 131.8 (C), 133.4 (C), 135.0 (CH), 144.3 (C), 190.8 (CO). Found %: C 69.93; H 4.93; N 8.76. $C_{18}H_{15}ClN_2O$. Calculated %: C 69.57; H 4.86; N 9.01.

3'-Chloro-4'-(4-chlorophenyl)spiro[1,2,3,4-tetrahydronaphthalene-2,5'-(1'-pyrazolin)]-1-one (IIb). Yield 25%. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.84 m (2H), 2.86 m (1H), 3.46 m (1H), 4.40 d (1H, *J* 9), 6.30 d (1H, *J* 9), 7.11 d (2H, *J* 7), 7.30 m (3H), 7.42 t (1H, *J* 8), 7.60 t (1H, *J* 8), 8.18 d (1H, *J* 8).

4'-(4-Methylphenyl)-3'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,5'-(1'-pyrazolin)]-1-one (IIc). Yield 46%, mp 132–133°C. IR spectrum, cm^{-1}: 820, 940, 1280, 1600, 1700 v.s, 2920, 3030. ^1H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.60 m (1H), 2.10 m (1H), 2.26 s (3H), 2.77 m (1H), 3.13 m (1H), 4.04 d (1H, *J* 10), 7.08 d (1H, *J* 10), 7.16 m (4H), 7.38 d (1H, *J* 8), 7.46 t (1H, *J* 7), 7.67 t (1H, *J* 7), 8.04 d (1H, *J* 7). Found %: C 70.39; H 5.20; N 8.41. $C_{19}H_{17}ClN_2O$. Calculated %: C 70.26; H 5.28; N 8.62.

1'-Aryl-2'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-ones III and IV. A solution of 0.48 mmol of pyrazoline **IIa–IIc** was heated in toluene for 2 h at 105–110°C. The solvent was distilled off in a vacuum to obtain 61% of a mixture of compounds **IIIa** and **IVa**, 51% of a mixture of compounds **IIIb** and **IVb**, and 51% of a mixture of compounds **IIIc** and **IVc** in a ratio ~9:1. Compounds **III** and **IV** were separated by column chromatography, eluent ethyl acetate–hexane.

***rel*-(1'*R*,2'*S*,3'*S*)-1'-Phenyl-2'-chlorospiro-[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-one (IIIa).** ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.35 d.t (1H, *J* 14, 4), 2.09 m (1H), 2.91 d.t (1H, *J* 16, 4), 3.19 m (1H), 3.77 d (1H, *J* 5), 3.84 d (1H, *J* 5), 7.23–7.43 m (7H), 7.53 m (1H), 8.19 d (1H, *J* 7). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.1 (CH_2), 28.6 (CH_2), 36.7 (CH), 40.5 (C), 42.9 (CH), 127.3 (CH), 127.7 (CH), 128.3 (CH), 128.9 (CH), 129.0 (CH), 129.2 (CH), 131.2 (C), 133.2 (C), 134.0 (CH), 135.0 (C), 144.1 (C), 191.9 (CO).

***rel*-(1'*R*,2'*S*,3'*R*)-1'-Chloro-2'-(4-chlorophenyl)-spiro-[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-one (IIIb).** ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.31 d.t (1H, *J* 14, 4), 2.08 m (1H), 2.92 d.t (1H, *J* 16, 4), 3.20 m (1H), 3.71 d (1H, *J* 5), 3.79 d (1H, *J* 5), 7.19 d (2H, *J* 8), 7.31 m (3H), 7.39 t (1H, *J* 8), 7.53 t (1H, *J* 7), 8.18 d (1H, *J* 8). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 28.1 (CH_2), 28.6 (CH_2), 35.9 (CH), 40.5 (C), 42.7 (CH), 127.4 (CH), 128.3 (CH), 129.1 (CH), 129.2 (CH), 130.5 (CH), 133.1 (C), 133.5 (C), 133.7 (C), 134.2 (CH), 144.0 (C).

***rel*-(1'*R*,2'*S*,3'*S*)-1'-(4-Methylphenyl)-1-oxo-2'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-one (IIIc).** IR spectrum, cm^{-1}: 905, 1000, 1010, 1050, 1100, 1150, 1260, 1310 v.s, 1350, 1390, 1450 s, 1600 s, 1665 v.v, 2865, 2925, 2950. ^1H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.36 d.t (1H, *J* 13, 4), 2.08 m (1H), 2.36 C (3H), 2.90 d.t (1H, *J* 16, 4), 3.18 d.d.d (1H, *J* 16, 13, 4), 3.75 d (1H, *J* 5), 3.80 d (1H, *J* 5), 7.15 m (4H), 7.28 d (1H, *J* 8), 7.38 t (1H, *J* 8), 7.52 t (1H, *J* 8), 8.19 d (1H, *J* 8). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 21.5 (CH_3), 28.1 (CH_2), 28.6 (CH_2), 36.5 (CH), 40.4 (C), 43.1 (CH), 127.3 (CH), 128.2 (CH), 129.1 (CH), 129.7 (CH), 131.7 (C), 133.3 (C), 134.0 (CH), 137.4 (C), 192.1 (CO). Found, %: C 76.82; H 5.80. $C_{19}H_{17}ClO$. Calculated, %: C 76.89; H 5.77.

***rel*-(1'*R*,2'*R*,3'*S*)-1'-Chloro-2'-(4-chlorophenyl)-spiro-[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-one (IVb).** ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.31 m (1H), 1.97 d.t (1H, *J* 15, 5), 2.20 m (1H), 2.97 m (1H), 3.40 d (1H, *J* 8), 3.93 d (1H, *J* 8), 7.16–7.40 m (6H), 7.54 t (1H, *J* 7), 8.04 d (1H, *J* 7).

***rel*-(1'*R*,2'*R*,3'*S*)-1'-(4-Methylphenyl)-2'-chlorospiro[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-one (IVc).** ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 2.00 m (1H), 2.22 m (1H), 2.37 s (3H), 2.40 m (1H), 3.02 m (1H), 3.40 d (1H, *J* 8), 3.96 d (1H, *J* 8), 7.12–7.30 m (5H), 7.37 t.d (1H, *J* 7, 2), 7.54 t (1H, *J* 7), 8.04 d (1H, *J* 7).

4'-Phenyl-3',3'-dichlorospiro[1,2,3,4-tetrahydronaphthalene-2,5'-(1-pyrazolin)]-1-one (VIII). In 20 ml of chloroform was dissolved 1.4 g (5 mmol) of pyrazoline **IIIa**, the solution was cooled by a salt-ice mixture, and a flow of dry chlorine was passed through the solution (about 30 min, TLC monitoring). Chloroform was evaporated, and a mixture of ethyl acetate with hexane was added. The separated precipitate was filtered off, dried, and used without further purification. Yield 0.4 g (24%), mp 84–85°C**.** ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.32 m (1H), 2.63 d.t (1H, *J* 14, 4), 3.02 m (1H), 3.30 m (1H), 4.59 C (1H), 7.42 m (7H), 7.70 t (1H, *J* 7), 8.00 d (1H, *J* 8). ^{13}C NMR spectrum (DMSO-d_6), δ, ppm: 26.1 (CH_2), 30.8 (CH_2), 56.2 (CH), 101.4 (C), 114.6 (C), 128.2 (CH), 129.1 (CH), 129.4 (CH), 129.6 (CH), 130.2 (CH), 130.8 (C), 131.0 (C), 132.3 (C), 136.1 (CH), 145.1 (C), 189.7 (CO).

2'-Phenyl-1',1'-dichlorospiro[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-one (IXa). In 5 ml of toluene was dissolved 0.30 g (0.8 mmol) of pyrazoline **VIII**. The solution was heated at 63–65°C for 40 min (TLC monitoring). The reaction occurred with gas liberation.Toluene was distilled off in a vacuum, the residue was recrystallized from ethanol. Yield 0.12 g (55%), mp 98"99°C. IR spectrum, cm^{-1}: 895, 915, 1030, 1070, 1150, 1310 s, 1335, 1390, 1450, 1500, 1505, 1605, 1680 v.s, 2950. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 2.00 d.t (1H, *J* 14, 4), 2.56 m (1H), 2.98 m (1H), 3.43 m (1H), 4.03 s (1H), 7.29–7.40 m (7H), 7.57 t (1H, *J* 7), 8.19 d (1H, *J* 7). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 27.8 (CH_2), 28.0 (CH_2), 38.6 (CH), 44.4 (C), 66.8 (C), 127.4 (CH), 128.0 (CH), 128.6 (CH), 128.9 (CH), 129.4 (CH), 130.5 (CH), 132.1 (C), 132.4 (C), 134.6 (CH), 144.3 (C), 190.9 (CO). Found, %: C 68.11; H 4.48. $C_{18}H_{14}Cl_2O$. Calculated, %: C 68.16; H 4.45.

1',1'-Dichloro-2'-(4-chlorophenyl)spiro[1,2,3,4-tetrahydronaphthalene-2,3'-cyclopropan]-1-one (IXb). In 10 ml of chloroform was dissolved 0.70 g (1.8 mmol) of pyrazoline **IIIb**, the solution was cooled by a salt-ice mixture, and a flow of dry chlorine was passed through the solution for 30 min (TLC monitoring). On removing the cooling and warming the mixture to room temperature a spontaneous gas liberation was observed accompanied by a self-heating. Chloroform was evaporated, ethanol was added to the residue. The separated precipitate was filtered off and recrystallized from ethanol. Yield 0.14 g (19%), mp 149–150°C. IR spectrum, cm^{-1}: 865, 920, 980, 1020 s, 1095, 1150, 1295 s, 1310, 1375, 1400, 1460, 1500, 1505, 1605 s, 1685 v.s, 1725 v.s, 2950, 3000. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 1.96 d.t (1H, *J* 14, 4), 2.53 t.d (1H, *J* 14, 4), 2.98 d.t (1H, *J* 17, 3), 3.42 m (1H), 3.97 s (1H), 7.26 m (2H), 7.37 m (4H), 7.57 t.d (1H, *J* 7, 1), 8.17 d (1H, *J* 7). ^{13}C NMR spectrum ($CDCl_3$), δ, ppm: 27.7 (CH_2), 27.9 (CH_2), 37.9 (CH), 44.4 (C), 66.5 (C), 127.7 (CH), 128.6 (CH), 129.2 (CH), 129.4 (CH), 130.8 (C), 131.9 (CH), 132.0 (C), 134.1 (C), 134.7 (CH), 144.2 (C). Found, %: C 61.41; H 3.86. $C_{18}H_{13}Cl_3O$. Calculated, %: C 61.48; H 3.73.

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