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Synthesis of dichlorodiazadiene derivatives based on o- and m-nitrobenzoic aldehyde

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Abstract

Dichlorodiazabutadienes are compounds of interest to synthetics with theirs unique structure. Thus, the presence of a diaza group, heminal dichlorine atoms, a combined heterodiene system in the composition of the compound and good solubility in organic solvents led to their application as synthons suitable for organic synthesis. In previous studies, dichlorodiazadiene derivatives were synthesized on the basis of some N-substituted hydrazones of o-, m- and p-nitrobenzoic aldehydes and many non-covalent bonds (hydrogen, nitrogen-halogen, halogen-chalcogen, halogen-halogen, pnicogenic bonds and etc.) in the obtained compounds have been studied for their role in crystal formation. It was found that the nature of the functional groups and the position in the benzene ring had a significant effect on the crystal design. In this regard, dichlorodiazadienes were synthesized from the reaction of CCl_4 with corresponding phenylhydrazones, synthesized from the reaction of o- and m-nitrobenzaldehyde with 3-halogenphenylhydrazines.

Keywords: N-substituted hydrazone, dichlorodiazadiene, non-covalent bonds.

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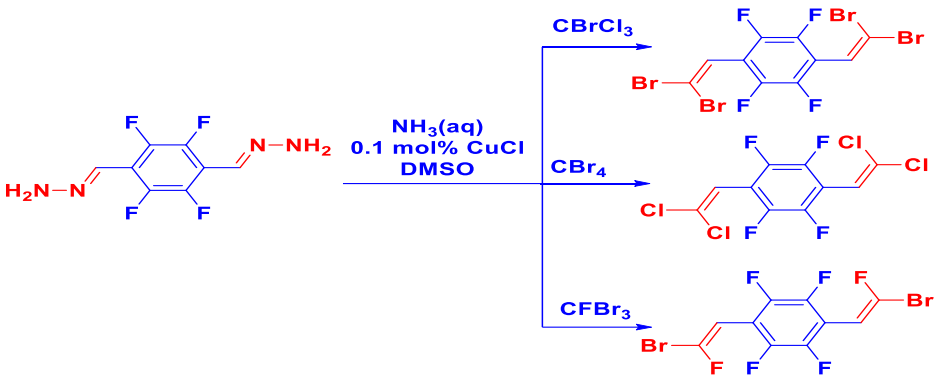
1. Introduction

The importance of non-covalent interactions has long been recognized and is currently attracting the attention of scientists working in various fields of chemistry and technology [1,2]. Non-covalent interactions include hydrogen, halogen, chalcogen and pnicogenic bonds, Van der Vaals forces, π-interactions, and so on. It should be noted that different types of bonds in different fields of chemistry can be considered as "non-covalent". For example, in organic chemistry, coordination bond is considered a non-covalent interaction. In general, non-covalent interactions, which are characterized by a significant reduction in the distance between atoms compared to the sum of the Van der Vaals radii, have a certain direction in space and at the same time have much less energy than a typical "covalent" bond. The weakness of non-covalent interactions and their diverse nature, such as the synthesis of organic, inorganic, coordinated and metal-organic compounds, supramolecular aggregates, are

of great application. With the help of non-covalent interactions, reagents can change the energy profile of the reaction and facilitate its course by providing the appropriate geometric coverage of the fragments entering the reaction. Thus, the formation of numerous non-covalent interactions between reaction products can be considered the main driving force of the process. As an example, we can show the non-covalent bonds formed by halogen atoms in compounds synthesized by us on the basis of catalytic olefinization reaction of N-nonsubstituted and N-substituted hydrazones of tetrafluoride terephthalic aldehyde[3,4] (Scheme 2,3. Figure 1,2.).



Scheme 1. Synthesis of 1,4-bis (2,2-dihalogenvinyl)-2,3,5,6-tetrafluorbenzenes

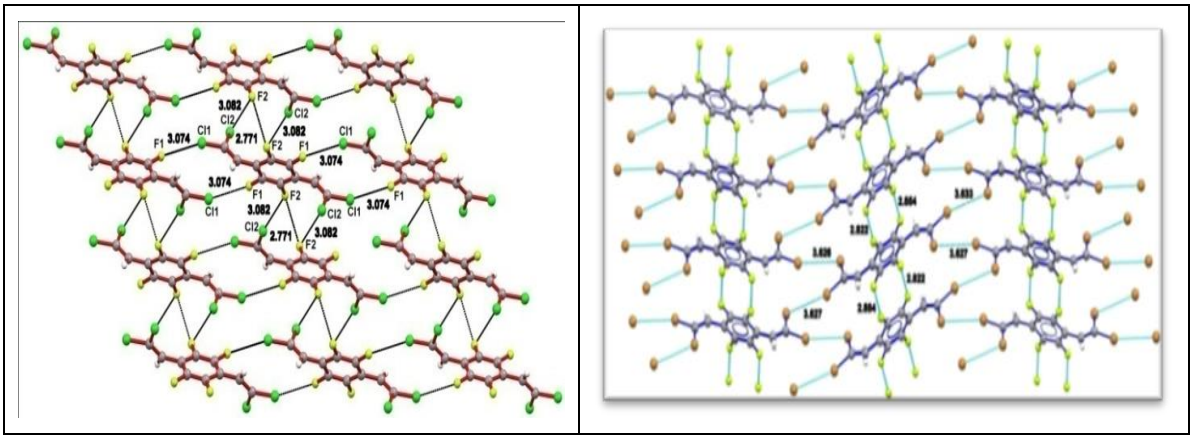
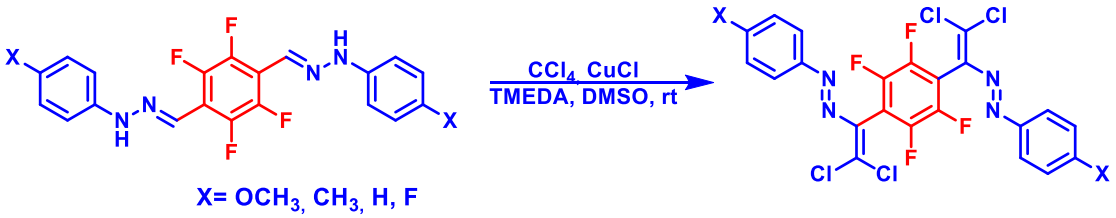


Figure 1. Intermolecular non-covalent F ··· F, F ··· Cl, Cl ··· CI, F··· Br and Br··· Br bonds in 1,4-bis(2,2-dihalogenvinyl)-2,3,5,6-tetrafluorbenzenesare shown by broken lines



Scheme 2. Synthesis of halogenated bis-diazo dyes

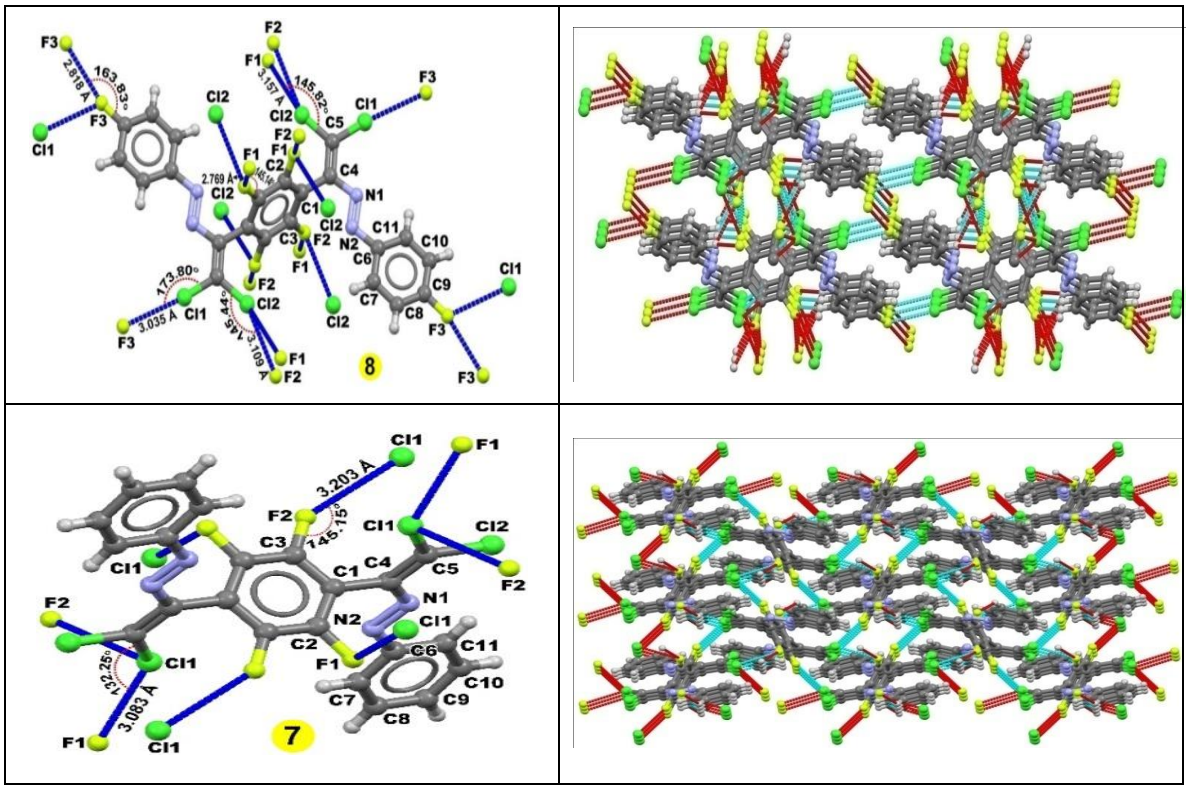
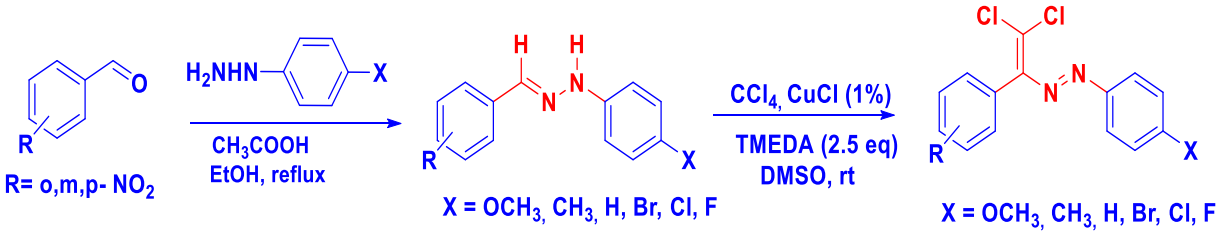
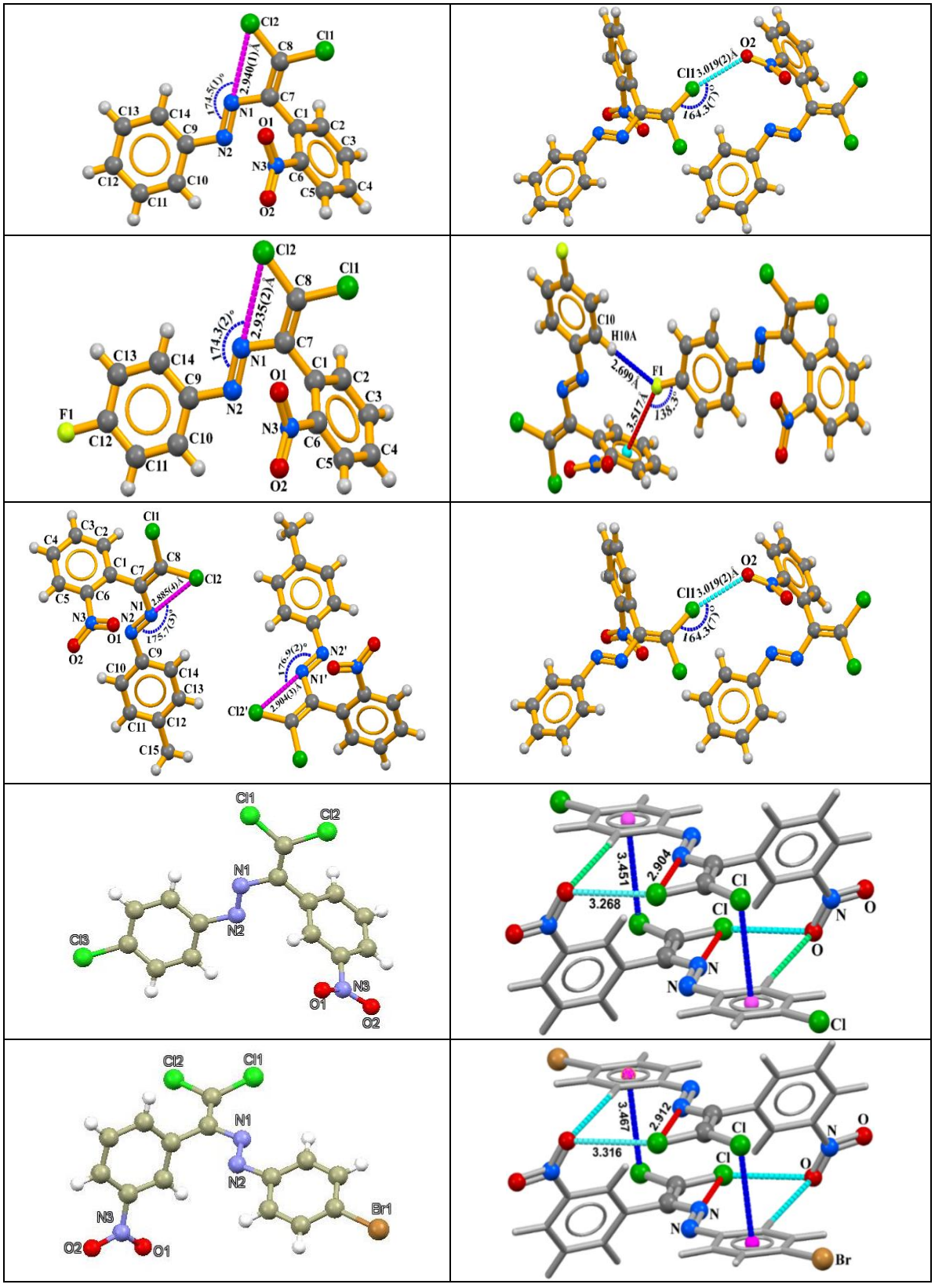


Figure 2. Intermolecular non-covalent bonds are shown by broken lines

It should be noted that in previous studies, it was synthesized dichlorodiazadiene derivatives on the basis of some N-substituted hydrazones of o, m- and p-nitrobenzoic aldehydes, and the role of many non-covalent bonds (halogen, hydrogen, nitrogen-halogen, halogen-chalcogen, halogen-halogen, pnicogenic N...Cl and etc) in crystal formation in the obtained compounds (more than 20) was studied. It was found that the nature of the functional groups and the position in the benzene ring had a significant effect on the crystal design [5-9] (Scheme 3, Figure 3). The role of non-covalent bonds in the formation of crystals of many synthesized compounds was also determined by Hirschfeld's surface analysis [10-17].



Scheme 3. Synthesis of (E)-1-(4-substitutesphenyl)-2-(2,2-dichloro-1-(nitro-phenyl)vinyl)diazene


Cl2
Cl1
C8
C7
C1
C2
C3
C4
C5
C6
N1
N2
N3
O1
O2
C9
C10
C11
C12
C13
C14
2.940(1)Å
174.5(1)°
Cl1 3.019(2)Å
164.3(7)°
2.935(2)Å
174.3(2)°
F1
C10
H10A
2.699Å
3.517Å
138.2°
C3
C2
C4
C1
C7
C8
Cl2
C5
C6
N1
N2
N3
O1
O2
C9
C10
C14
C13
C11
C12
C15
2.885(4)Å
175.7(3)°
176.9(2)°
N2'
N1'
Cl2'
2.904(3)Å
Cl1
Cl2
N1
N2
Cl3
O1
N3
O2
3.451
2.904
3.268
Cl
O
N
3.467
2.912
3.316
Br1
Br

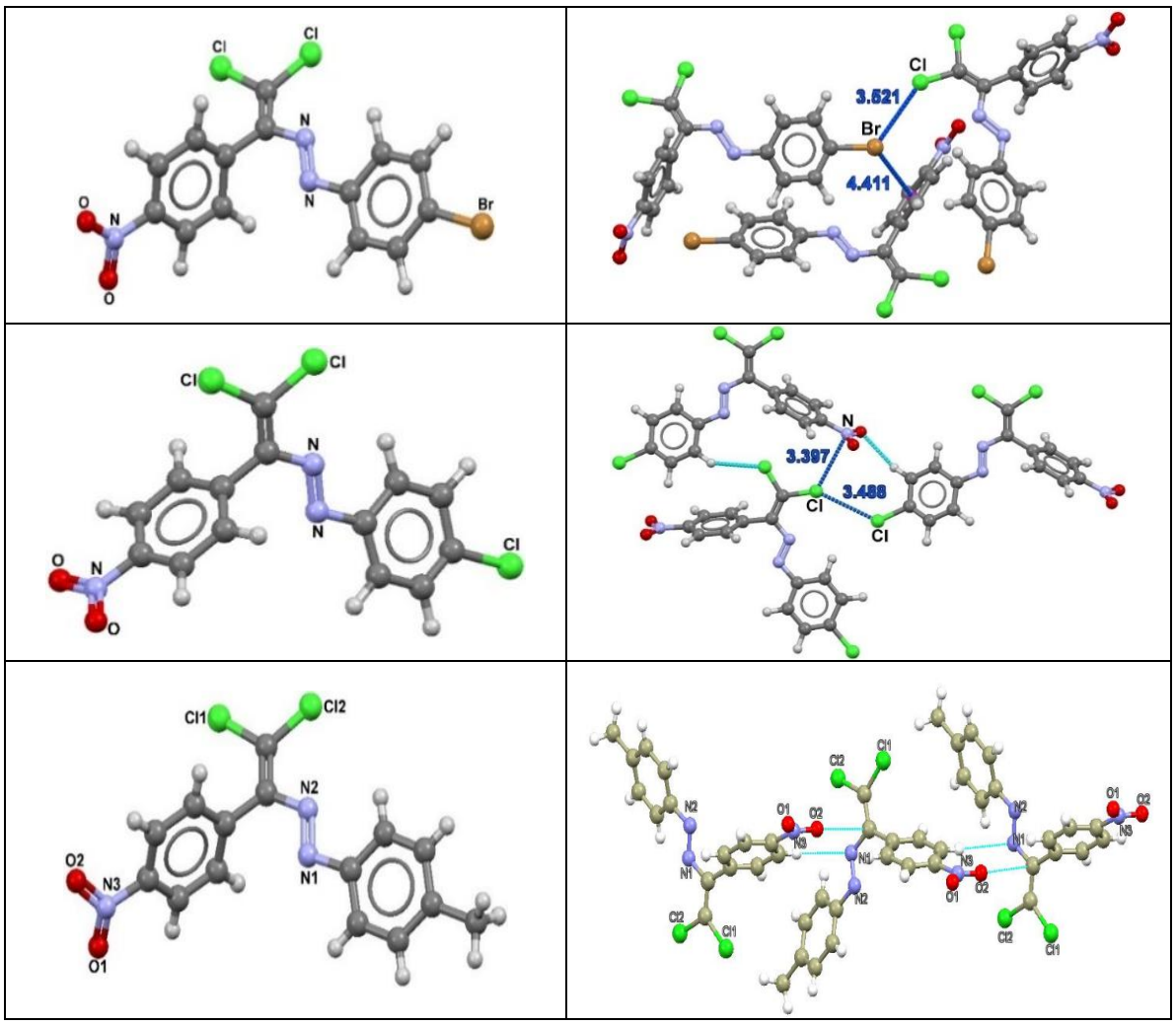
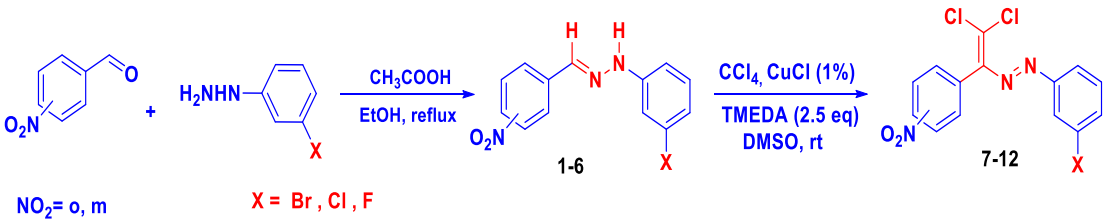


Figure 3. Intermolecular non-covalent bonds are shown by broken lines

It should be noted that the synthesized dichlorodiazabutadienes are compounds of interest to synthetics with their unique structure. Thus, the presence of a diaza group, heminal dichlorine atoms, a combined heterodiene system in the compound led to their application as synthons suitable for organic synthesis [18-21].

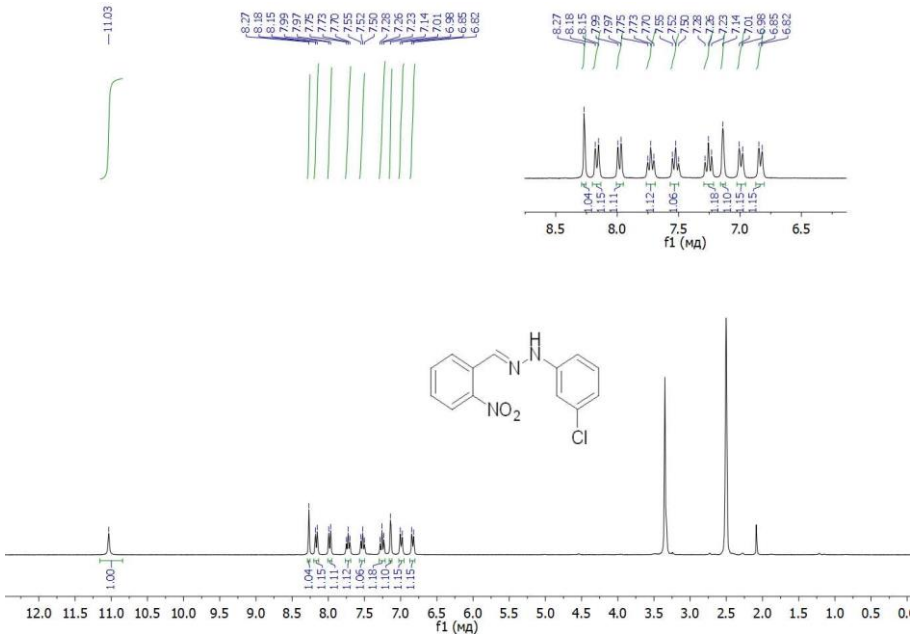
At the same time, dichlorodiazadienes are well soluble in organic solvents, so it increases their relevance as suitable synthons in drugs, dyes and other various fields of application. In view of all this, corresponding phenylhydrazones (1-6) were synthesized from the reactions of o- and m-nitrobenzaldehyde with 3-halogenphenylhydrazines, and dichlorodiazadienes (7-12) were obtained from the reaction of the latter with CCl_4 (Scheme4).



Scheme 4. Synthesis of (E)-1-(3-halogenphenyl)-2-(2,2-dichloro-1-(nitro-phenyl)vinyl)diazene

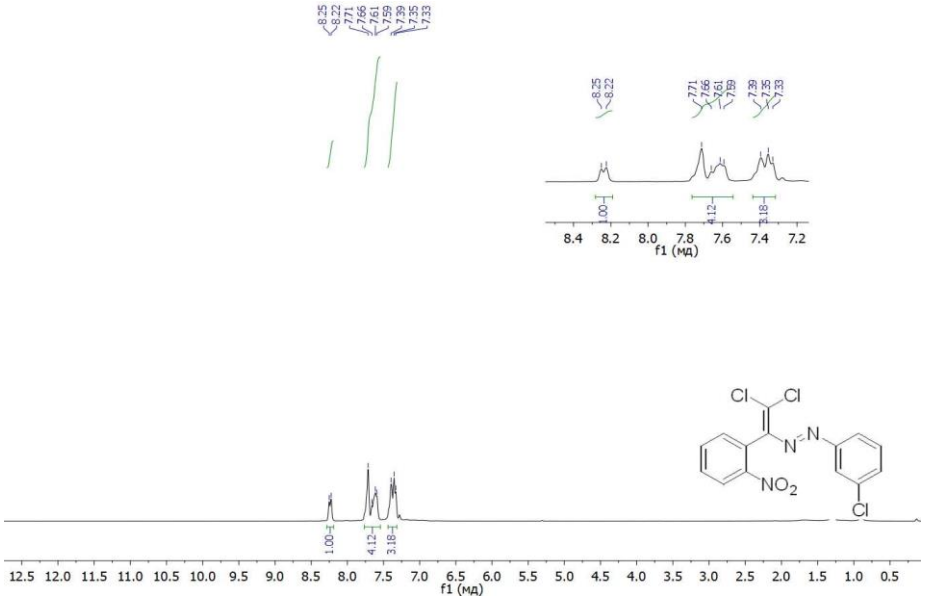
The structure of synthesized hydrazones and dichlorodiazadienes (^1H and ^{13}C) was determined on the basis of NMR spectra.

Hydrazone resonates in the DMSO-d6 (dimethylsulfoxide-D) solvent in the 1H NMR spectrum at room temperature δ6.82–7.28, which indicates protons in the aromatic part. At the same time, a singlet was observed at δ8.28, which proves the presence of the H atom in the CH group. In the ^{13}C NMR spectrum of the compound, all carbon atoms were also found in the expected areas. The interpretation of the spectra proves the structure of the compounds. The known spectrum is shown below.



Spectrum 1. ^{1}H spectrum of substance 2

In the corresponding dichlorodiazabutadienes, the protons in the aromatic part resonate in δ7.33-8.25 in the $CDCl_3$ solvent at room temperature. In the ^{13}C NMR spectrum of 7-12 compounds, all carbon atoms were found in the expected areas. The interpretation of the spectra clearly proves the structure of the compounds. One of the known spectra is shown below.



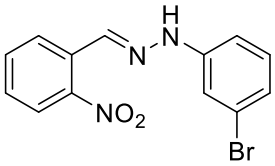
Spectrum 2. ^{1}H spectrum of substance 8

2. Experiments

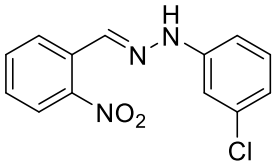
NMR ^{1}H and ^{13}C spectra were recorded on a Brooker Avance 300 MHz spectrometer in $CDCl_3$ and DMSO. $SiMe_4$ was used as an internal standard. TLC was carried out on Silufol plate UV-254, column chromatography - on Merk silica gel.

***General methodology of hydrazone synthesis*:**

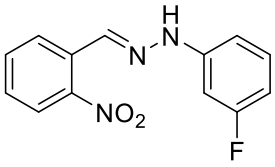
Ethanol (20-50 ml), 0.820 g of CH_3COONa (10 mmol) and 1 mmol of corresponding aromatic aldehydes are added to (5 mmol) phenylhydrazine.The reaction lasts 1 hour. After cooling, 50 ml of water is added to the solution and heated to 60 ^{0}C. The reaction ends.



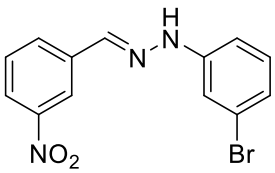
Compound 1. (E)-1-(3-bromophenyl)-2-(2-nitro-benzyliden) hydrazine. $C_{13}H_{10}BrN_3O_2$ (M=320.146), yield 78%, T_{melt}= 167 ^{0}C, ^{1}H NMR (300 MHz, DMSO-d_6) δ 11.02 (s, 1H), 8.26 (s, 1H), 8.16 (d, *J* = 7.9 Hz, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.73 (t, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.28 (s, 1H), 7.20 (t, *J* = 8.0 Hz, 1H), 7.03 (d, *J* = 8.1 Hz, 1H), 6.96 (d, *J* = 7.7 Hz, 1H). ^{13}C NMR (75 MHz, DMSO) δ 147.42, 146.59, 133.70, 132.91, 131.62, 129.87, 129.12, 127.76, 124.98, 122.97, 122.51, 114.95, 111.95.



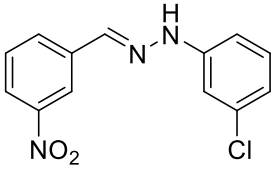
Compound 2. (E)-1-(3-chlorophenyl)-2-(2-nitro-benzyliden) hydrazine. $C_{13}H_{10}ClN_3O_2$ (M=275,046), yield 82%, T_{melt}= 160 ^{0}C, ^{1}H NMR (300 MHz, DMSO-d_6) δ 11.03 (s, 1H), 8.27 (s, 1H), 8.17 (d, *J* = 7.9 Hz, 1H), 7.98 (d, *J* = 8.1 Hz, 1H), 7.73 (t, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.6 Hz, 1H), 7.26 (t, *J* = 8.0 Hz, 1H), 7.14 (s, 1H), 6.99 (d, *J* = 8.1 Hz, 1H), 6.83 (d, *J* = 7.9 Hz, 1H). ^{13}C NMR (75 MHz, DMSO) δ 147.43, 146.49, 134.41, 133.68, 132.86, 131.32, 129.89, 129.11, 127.77, 124.97, 119.61, 112.10, 111.58.



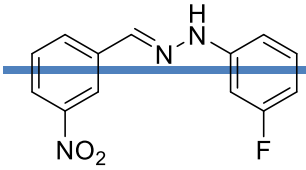
Compound 3. (E)-1-(3-fluorophenyl)-2-(2-nitro-benzyliden) hydrazine. $C_{13}H_{10}FN_3O_2$ (M=259.240), yield 80%, T_{melt}= 171 ^{0}C, ^{1}H NMR (300 MHz, DMSO-d_6) δ 11.06 (s, 1H), 8.26 (s, 1H), 8.17 (d, *J* = 7.9 Hz, 1H), 7.98 (d, *J* = 7.9 Hz, 1H), 7.72 (t, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 7.5 Hz, 1H), 7.26 (q, *J* = 7.7 Hz, 1H), 6.88 (dd, *J* = 16.2, 10.0 Hz, 2H), 6.60 (t, *J* = 8.2 Hz, 1H). ^{13}C NMR (75 MHz, DMSO) δ 133.64, 132.58, 131.38, 131.25, 129.91, 129.06, 127.77, 124.96, 108.99, 106.46, 106.18, 99.58, 99.23.



Compound 4. (E)-1-(3-bromophenyl)-2-(3-nitro-benzyliden) hydrazine. $C_{13}H_{10}BrN_3O_2$ (M=320.146), yield 78%, T_{melt}= 167 ^{0}C, ^{1}H NMR (300 MHz, DMSO-d_6) δ 10.84 (s, 1H), 8.43 (s, 1H), 8.12 (d, *J* = 7.8 Hz, 2H), 7.98 (s, 1H), 7.67 (t, *J* = 7.9 Hz, 1H), 7.28 (s, 1H), 7.19 (t, *J* = 8.0 Hz, 1H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.94 (d, *J* = 7.7 Hz, 1H). ^{13}C NMR (75 MHz, DMSO) δ 148.73, 146.80, 137.82, 135.88, 132.14, 131.61, 130.64, 122.91, 122.88, 122.17, 120.44, 114.84, 111.82.



Compound 5. (E)-1-(3-chlorophenyl)-2-(3-nitro-benzyliden) hidrazine. $C_{13}H_{10}ClN_3O_2$ (M=275,046), yield 82%, T_{melt}= 160 ^{0}C, ^{1}H NMR (300 MHz, DMSO-d_6) δ 10.85 (s, 1H), 8.42 (s, 1H), 8.11 (d, *J* = 7.7 Hz, 2H), 7.97 (s, 1H), 7.65 (t, *J* = 7.9 Hz, 1H), 7.24 (t, *J* = 7.9 Hz, 1H), 7.14 (s, 1H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.80 (d, *J* = 7.6 Hz, 1H).^{13}C NMR (75 MHz, DMSO) δ 162.32, 148.71, 146.69, 137.84, 135.79, 134.35, 132.12, 131.25, 130.58, 122.83, 120.42, 119.25, 111.99, 111.44.

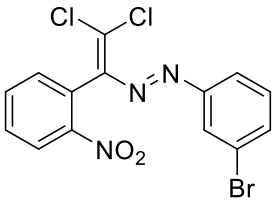


Compound 6. (E)-1-(3-fluorophenyl)-2-(4-nitrobenzyliden) hydrazine. $C_{13}H_{10}FN_3O_2$ (M=259.240), yield 83%, T_{melt}= 115 ^{0}C,

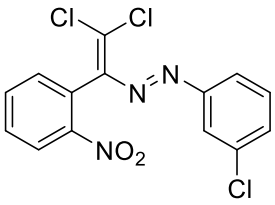
^{1}H NMR (300 MHz, DMSO-d_6) δ 10.87 (s, 1H), 8.44 (s, 1H), 8.12 (d, *J* = 7.5 Hz, 2H), 7.98 (s, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.26 (d, *J* = 7.4 Hz, 1H), 6.90 (t, *J* = 9.7 Hz, 2H), 6.57 (t, *J* = 8.0 Hz, 1H).^{13}C NMR (75 MHz, DMSO) δ 148.73, 137.90, 135.56, 132.14, 131.20, 130.60, 122.81, 120.40, 108.89, 106.12, 105.83, 99.48, 99.13.

General method of synthesis of dichlorodiazabutadienes

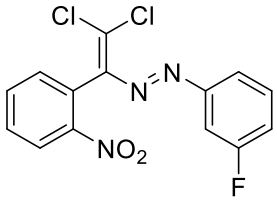
1 mmol of starting hydrazone is added to the flask, followed by 10-12 ml of DMSO, followed by (290 mg; 1.25 mol / eq) TMEDA. Then CuCl (6 mg; 3 mol%) is added. CCl_4 (4-5 mol / eq; 1.5 g) is added to the latter. At the end of the reaction, which lasts 3 hours, the extraction process begins - methylene chloride (3 * 15 ml), NaCl (1 * 50 ml), Na_2SO_4. Solvents used in column chromatography - hexane: dichloromethane 1: 5.



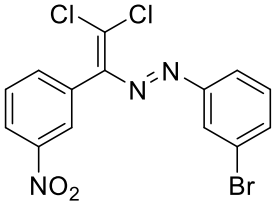
Compound 7. (E)-1-(3-bromophenyl)-2-(2,2-dichloro-1-(2-nitrophenyl)vinyl)diazene, $C_{14}H_8BrCl_2N_3O_2$ (M=401.041), yield 51%, T_{melt}= 87 ^{0}C, ^{1}H NMR (300 MHz, Chloroform-*d*)) δ 7.92 (t, *J* = 2.0 Hz, 1H), 7.86 (dd, *J* = 7.5, 2.0 Hz, 1H), 7.64 (tt, *J* = 7.7, 2.0 Hz, 3H), 7.56 (td, *J* = 7.5, 2.0 Hz, 1H), 7.39 – 7.27 (m, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.85, 149.53, 143.54, 136.19, 133.89, 132.53, 130.21, 129.69, 129.00, 124.77, 123.72, 122.47, 120.15.



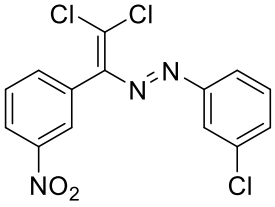
Compound 8. (E)-1-(3-chlorophenyl)-2-(2,2-dichloro-1-(2-nitrophenyl)vinyl)diazene. $C_{14}H_8Cl_3N_3O_2$ (M=356.587), yield 53%, T_{melt}= 75 ^{0}C, ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.24 (d, *J* = 8.0 Hz, 1H), 7.76 – 7.54 (m, 4H), 7.44 – 7.32 (m, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 153.10, 150.40, 147.99, 136.73, 135.12, 133.83, 132.19, 131.56, 130.35, 130.12, 128.15, 124.59, 122.48.



Compound 9. (E)-1-(3-fluorophenyl)-2-(2,2-dichloro-1-(2-nitrophenyl)vinyl)diazene. $C_{14}H_8$ $FCl_2N_3O_2$ (M=340.135), yield 43%, T_{melt}= 65 ^{0}C, ^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.86 (dd, *J* = 7.5, 2.0 Hz, 1H), 7.65 (dd, *J* = 7.4, 2.1 Hz, 1H), 7.57 (td, *J* = 7.5, 1.9 Hz, 1H), 7.45 (ddt, *J* = 10.7, 8.7, 2.0 Hz, 2H), 7.41 (td, *J* = 7.4, 5.6 Hz, 1H), 7.35 (td, *J* = 7.5, 2.0 Hz, 1H), 7.18 (ddt, *J* = 9.3, 7.4, 2.0 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.86, 150.70, 150.64, 149.53, 143.54, 136.19, 133.89, 132.53, 129.69, 123.72, 123.58, 119.08, 115.31, 110.68

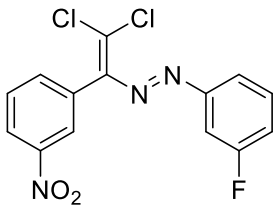


Compound 10. (E)-1-(3-bromophenyl)-2-(2,2-dichloro-1-(3-nitrophenyl)vinyl)diazene, $C_{14}H_8BrCl_2N_3O_2$ (M=401.041), yield 56%, T_{melt}= 91 ^{0}C, ^{1}H NMR (300 MHz, Chloroform-*d*)) δ 8.33 (t, *J* = 2.0 Hz, 1H), 8.19 (dt, *J* = 7.5, 2.0 Hz, 1H), 7.79 – 7.70 (m, 2H), 7.64 (ddt, *J* = 6.9, 4.3, 2.0 Hz, 2H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.33 (t, *J* = 7.5 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δδ 150.80, 147.58, 145.84, 135.51, 133.98, 133.48, 130.37, 130.19, 129.00, 127.37, 126.40, 124.98, 122.40, 120.17.



Compound 11. (E)-1-(3-chlorophenyl)-2-(2,2-dichloro-1-(3-nitrophenyl)vinyl)diazene. $C_{14}H_8Cl_3N_3O_2$ (M=356.587), yield 59%, T_{melt}= 69 ^{0}C, ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.33 (t, *J* = 2.0 Hz, 1H), 8.19 (dt, *J* = 7.3, 2.0 Hz, 1H), 7.69 – 7.61 (m, 3H), 7.57 (dt, *J* = 7.5, 2.0 Hz, 1H), 7.50 (t, *J* = 7.5 Hz, 1H), 7.38 (t, *J* = 7.4 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ^{13}C NMR (125 MHz, Common NMR

Solvents) δ 150.54, 147.58, 145.84, 135.51, 135.08, 133.98, 133.48, 130.64, 130.37, 130.06, 127.37, 126.40, 122.15, 121.57.



Compound 12. (E)-1-(3-fluorophenyl)-2-(2,2-dichloro-1-(3-nitrophenyl)vinyl)diazene. $C_{14}H_8FCl_2N_3O_2$ (M=340.135), yield 39%, T_{melt}= 72 ^{0}C, ^{1}H NMR (300 MHz, Chloroform-*d*) δ 8.33 (t, *J* = 2.0 Hz, 1H), 8.19 (dt, *J* = 7.5, 2.0 Hz, 1H), 7.64 (dt, *J* = 7.5, 2.0 Hz, 1H), 7.57 – 7.47 (m, 2H), 7.42 (td, *J* = 7.5, 5.6 Hz, 1H), 7.56 (dt, *J* = 8.8, 2.0 Hz, 1H), 7.19 (ddt, *J* = 9.3, 7.5, 2.0 Hz, 1H), ^{13}C NMR (75 MHz, $CDCl_3$) δ 163.88, 150.89, 147.58, 145.84, 135.51, 133.98, 133.48, 130.37, 127.37, 126.40, 123.52, 119.06, 115.15, 110.52.

3. Conclusion

In previous studies, the synthesis of dichlorodiazadiene derivatives on the basis of some N-substituted hydrazones of o-, m- and p-nitrobenzoic aldehydes was carried out, and many non-covalent bonds (hydrogen, nitrogen-halogen, halogen-chalcogen, halogen-halogen, pnicogenic bonds and etc) in the obtained compounds have been studied for their role in crystal formation. It was found that the nature of the functional groups and the position in the benzene ring had a significant effect on the crystal design. Continuing research in this direction, dichlorodiazadienes were synthesized from the reaction of $CCl_4$with corresponding phenylhydrazones, obtained from the reaction of o- and m-nitrobenzaldehyde with meta-halogenated phenylhydrazines. The structure of the synthesized compounds was determined by the NMR method.

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