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REACTIONS OF SUBSTITUTED *gem*-DICHLOROCYCLOPROPANES WITH PHENOLS

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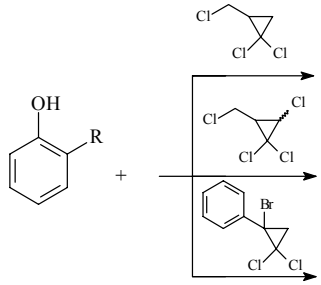
By studying the reactions of substituted phenols with halomethyl-gem-dichlorocyclopropanes in dimethyl sulfoxide, concurrent for both endocyclic and exocyclic chlorine atoms, it was found that cis- and trans-1,1,3-trichloro-2-(chloromethyl)cyclopropanes react with phenols, producing 1,1-disubstituted (chloromethylen)cyclopropanes, as mixtures of Z- and E-configuration isomers, and 1,1-disubstituted 2-chloro-3-methylencyclopropanes. Cyclopropane ring cleaving was observed in reactions of phenols with 2-bromo-2-phenyl-gem-dichlorocyclopropane. The nature of the nucleophile has a significant effect on the yield of the reaction products.



INTRODUCTION

First stage[1] of our research in the synthesis and reactions of substituted *gem*-dichlorocyclopropanes was associated with the development of regio- and stereoselective methods, of adding dichlorocarbene to polyene structures and multifunctional olefins of petrochemical origin (conjugated and unconjugated, linear and cyclic dienes, mono- and polyhaloalkenes etc.).

Transformations of alkenyl-*gem*-dichlorocyclopropanes (alkylation, oxidation, hydration, hydrogenation, etc.), as well as reduction of endocyclic CCl_2- group were previously studied.[2-4]

Continuing this work, we examined the reactions of substituted *gem*-dichlorocyclopropanes with mono- and dibasic phenols.

RESULTS AND DISCUSSION

It is known that interaction of chloride **1a** with phenol in dimethyl sulfoxide in the presence of sodium hydroxide yields substitution products of both exocyclic and endocyclic chlorine atoms, with the latter compounds dominating. [5-8] In the present study we found that in these conditions halomethyl-*gem*-chlorocyclopropanes **1b,c** react with phenols **2a-c** yielding mostly aryloxy derivatives **3a-c** (selectivity over 80%), as well as 1,1-disubstituted methylcyclopropanes **4a-c** (Scheme 1).

In the case of diols **2d** both substitution products are formed for the exocyclic chlorine atom **5,6** and methylenespiro[1,3-benzodioxolan-2,1'-cyclopropane] **7**, as a result of endocyclic chlorine replacement (Scheme 2). Product ratios for **5**, **6** and **7** change from 41:52:4 for **1a** up to 2:28:68 for **1c**.

Trichlorocyclopropanes **8a,b** react with phenols **2a-c** in similar conditions producing 1,1-disubstituted 2-chloro-3-methylencyclopropanes **11a-c** and mixtures of 1,1-disubstituted (chloromethylen) cyclopropanes **9a-c**, **10a-c** (Scheme 3), with a total yield of 79-94%. Substituents in the aromatic ring have little effect on the reaction's regio- and stereoselectivity. Formation of Z-configuration isomer **9a-c** is preferred.

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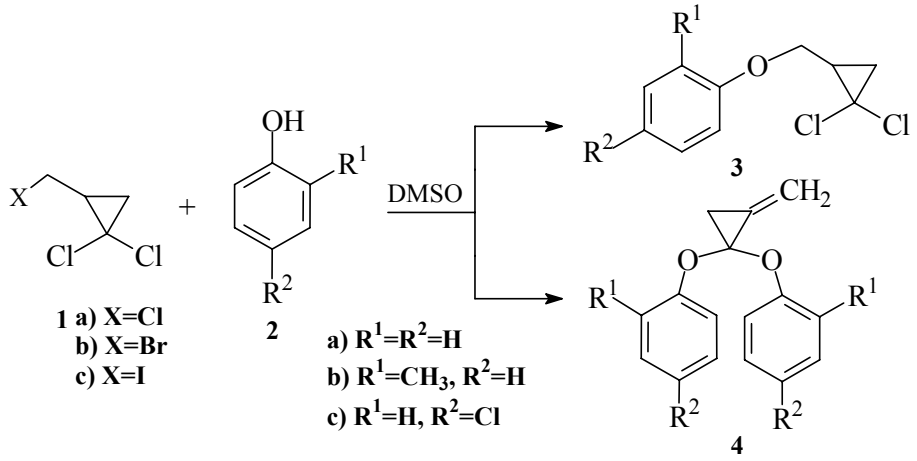
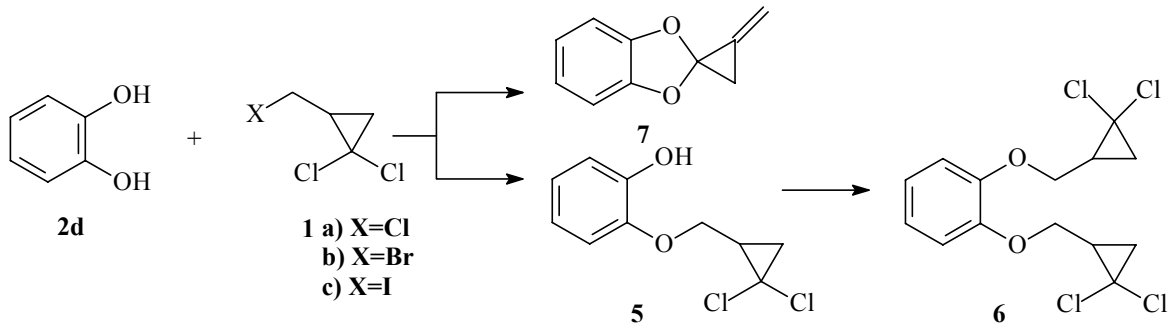
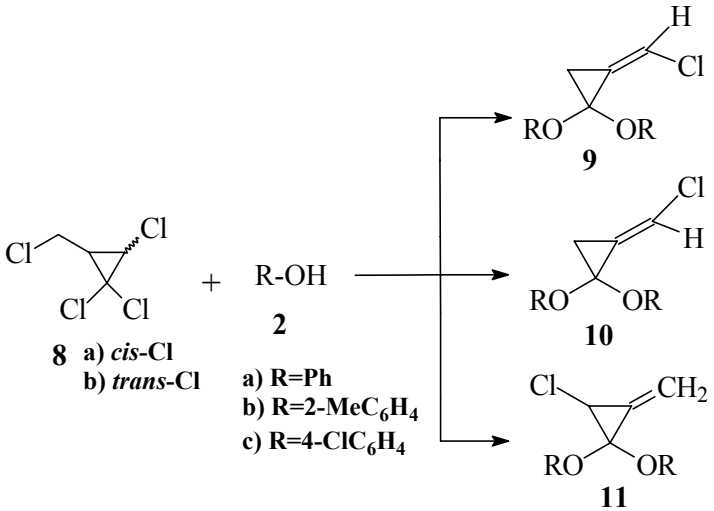


Scheme 1 – Preparation of aryloxy derivatives **3a-c** and 1,1-disubstituted methylcyclopropanes **4a-c**.



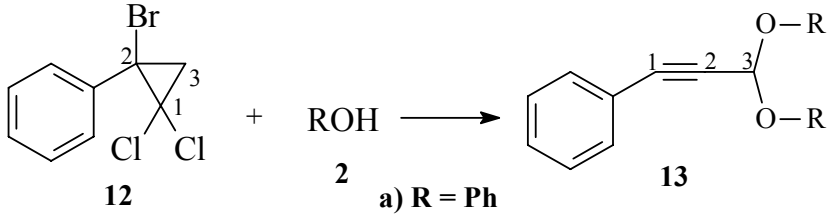
Scheme 2 – Preparation of aryloxy derivatives **5,6** and methylenespiro[1,3-benzodioxolan-2,1'-cyclopropane] **7**.



Scheme 3 – Preparation of 1,1-disubstituted (chloromethylen)cyclopropanes **9a-c**, **10a-c** and 2-chloro-3-methylencyclopropanes **11a-c**.

Composition and structure of the resulting compounds **9a-c**, **10a-c**, **11a-c** have been confirmed by NMR spectroscopy and mass spectrometry. ^{1}H NMR spectra of compounds **9a**, **10a** show not only three-membered ring and aromatic ring protons, but ending chlorine double bond protons as well: for the *Z*-configuration stereoisomer **9a** in the 6.93 ppm area (with spin-spin coupling constants (SSCC) $^4J_{4\text{-}3a}$ and $^4J_{4\text{-}3b}$ 3.2 Hz) and in the 6.37 ppm area for the **10a** *E*-isomer (with SSCC $^4J_{4\text{-}3a}$ and $^4J_{4\text{-}3b}$ 2.6 Hz).

At the same time, interaction of 2-bromo-2-phenyl-*gem*-dichlorocyclopropane **12** with phenols **2a-c** in similar conditions demonstrates cyclopropane ring cleaving resulting in phenylpropargyl aldehyde acetals **13a-c** (Scheme 4).



Scheme 4 – Preparation of phenylpropargyl aldehyde acetals **13a-c**.

The unselective formation of acetylenic structures at the condensation of polysubstituted *gem*-dibromocyclopropanes with alcohols had been described before[9-12].

A characteristic feature of the **13a** compound's ^{13}C NMR spectrum recorded in C-H interaction constant modulation mode (JMOD) is the presence of carbon atom signals at the triple bond (95.10 and 85.32) and of the $C(OR)_2$- group quaternary carbon atom signal (103.39).

EXPERIMENTAL

An LHM-8D chromatograph was used for the qualitative and quantitative analysis of starting material and reaction products. The chromatograph was equipped with a thermo-conductivity detector and 2 m column with 5% SE-30 on a Chromaton N-AW support. ^1H and ^{13}C NMR spectra were registered using the Bruker AM-300 spectrometer (300.13 and 75.47 MHz, respectively) in $CDCl_3$ solvent, where Me_4Si was used as an internal standard. Mass-spectra were recorded using the Focus instrument, equipped with a Finnigan DSQ II detector (70 eV, ionization cell temperature 200°C, injector temperature 50-270°C, with a velocity of the temperature rise 10°C/min).

Initial 2-halomethyl-*gem*-dichlorocyclopropanes **1a-c**, 2-brom-2-phenyl-*gem*-dichlorocyclopropane **12** and *cis* and *trans*-1,1,2-trichloro-3-chloromethylcyclopropanes **8a,b** were obtained by the known procedure.[13]

1. Synthesis of aryloxymethyl-gem-dichlorocyclopropanes (**3a-c, 5, 6**) and methylenecyclopropanes (**4a-c, 7, 9a-c, 10a-c, 11a-c**).

A solution of 0.005 M substituted *gem*-dichlorocyclopropane **1a-c, 8a,b** in 1 mL DMSO was added, with intensive stirring, to the mixture of 0.015 M phenol **2a-c** (or 0.01 M catechol **2d**), 0.03 M NaOH (0.02 M in the case of **2d**), 0.0001 M triethylbutylammonium chloride (TEBAC as phase transfer catalyst) (in the case of **8a,b**), 5.3 mL DMSO (2.8mL in the case of **2d**), at the temperature of 55-60°C (0-5°C in the case of **8a,b**). After 2-5 hours (0.2 hours in the case of **8a,b**), the reaction mixture was diluted with water, extracted with chloroform, and the organic layer was washed with 40 mL of a 20% NaOH solution, then with water until neutral, dried with a freshly prepared anhydrous $MgSO_4$, and chloroform was removed at a low pressure; the residue was purified by column chromatography, using silica gel and hexane (cyclohexane), with an increasing ethyl acetate content from 5 to 100%.

The resulting physico-chemical properties, NMR-spectra and mass-spectra of compounds **3a-c, 4a-c** correspond to literature data.[5]

2-[(2,2-dichlorocyclopropyl)methoxy]phenol **(5).** R_f=0.37 (hexane:AcOEt, 3:1). ^1H-NMR ($CDCl_3$, δ ppm, *J*Hz): 1.39 (dd, 1H, C^3H_a, ^2J 7.34, ^3J 7.63); 1.77 (dd, 1H, C^3H_b, ^2J 7.34, ^3J 10.27); 2.13 (m, 1H, C^1H); 4.12 (dd, 1H, $C^{1'}H_a$, ^2J 10.57, ^3J 7.93); 4.26 (dd, 1H, $C^{1'}H_b$, ^2J 10.57, ^3J 5.87); 6.21 (s, 1H, OH); 6.85-7.02 (m, 4H, C_6H_4). MS (70 eV), m/z (*J*., %): 232/234/236 M^+ (7/4/1), 123/125/127 (18/12/2), 110 (100), 100 (6), 87/89 (44/16), 81 (20), 63 (7), 51 (22).

1,2-bis[(2,2-dichlorocyclopropyl)methoxy]benzene **(6).** R_f=0.57 (hexane:AcOEt, 3:1). ^1H-NMR ($CDCl_3$, δ ppm, *J*Hz): 1.39 (dd, 2H, C^3H_a, $C^{3'}H_a$, ^2J 7.34, ^3J 7.63); 1.77 (dd, 2H, C^3H_b, $C^{3'}H_b$, ^2J 7.34, ^3J 10.27); 2.13 (m, 2H, C^1H, $C^{1'}H$); 4.13 (dd, 2H, $C^{1''}H_a$, $C^{1''}H_a$, ^2J 10.57, ^3J 7.93); 4.26 (dd, 2H, $C^{1''}H_b$, $C^{1''}H_b$, ^2J 10.57, ^3J 5.87); 6.70-6.89 (m, 4H, C_6H_4). MS (70 eV), m/z (*J*., %): 356 M^+ (2), 232 (2), 123/125/127 (38/24/4), 110 (100), 100 (18), 87/89 (57/19), 81 (11), 63 (6), 51 (22).

2'-methylenespiro[1,3-benzodioxole-2,1'-cyclopropane] **(7)**. b.p. 79-81°C (4 mm Hg). ^1H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.14 (t, 2H, $C^{3'}H_a$, $C^{3'}H_b$, ^{4}J 2.20); 5.05 (s, 1H, $C^{1''}H_a$, ^{4}J 2.20); 5.33 (s, 1H, $C^{1''}H_b$, ^{4}J 2.20); 6.63-6.91 (m, 4H, C_6H_4). MS (70 eV), m/z (*J*., %): 160 M^+ (81), 145 (37), 134 (100), 121 (3), 103 (4), 92 (7), 76 (6), 67 (18), 51 (14).

1,1'-[[(2Z)-2-(chloromethylene)cyclopropane-1,1-diyl]bis(oxy)] dibenzene (**9a**). R_f 0.30 (hexane:AcOEt, 60:1). ^1H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.21 (d, 2H, C^3H_a, C^3H_b, ^4J 3.23), 6.93 (t, 1H, $C^{1''}HCl$, ^4J 3.23), 7.05-7.37 (m, 10H, Ph). ^{13}C-NMR ($CDCl_3$, δ ppm): 21.34 (C^3H_2); 85.3 (C^1); 113.73 ($C^{1''}HCl$); 117.1, 122.9, 129.5, 156.0 (Ph), 126.2 (C^2). MS (70 eV), m/z (*J*., %): 271/273 $[M-1]^+$ (1), 235 (2), 179/181 $[M-OPh]^+$ (39/14), 151/153 (34/11), 143 (19), 115 (100), 94 (17), 91 (56), 77 (78), 65 (21), 51 (51).

1,1'-[[(2E)-2-(chloromethylene)cyclopropane-1,1-diyl]bis(oxy)] dibenzene (**10a**). R_f 0.21 (hexane:AcOEt, 60:1). ^1H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.13 (d, 2H, C^3H_a, C^3H_b, ^4J 2.47), 6.37 (t, 1H, $C^{1''}HCl$, ^4J 2.6), 7.05-7.37 (m, 10H, Ph). ^{13}C-NMR ($CDCl_3$, δ ppm): 20.49 (C^3H_2); 84.61 (C^1); 115.34 ($C^{1''}HCl$); 117.1, 122.9, 129.5, 156.0 (Ph), 125.99 (C^2);. MS (70 eV), m/z (*J*., %): 271/273 M-1 (1), 235 (2), 179/181 M-OPh (42/13), 151/153 (32/10), 143 (19), 115 (100), 94 (15), 91 (45), 77 (75), 65 (17), 51 (39).

1,1'-[(2-chloro-3-methylenecyclopropane-1,1-diyl)bis(oxy)] dibenzene **(11a).** R_f 0.26 (hexane:AcOEt, 60:1). ^1H-NMR ($CDCl_3$, δ ppm, *J*Hz): 4.03 (t, 1H, C^3HCl, ^4J 2.07), 5.95 (d., 1H, $C^{1''}H_a$, ^2J 1.55), 6.07 (d., 1H, $C^{1''}H_b$, ^2J 1.55), 7.11-7.38 (m, 10H, Ph). ^{13}C-NMR ($CDCl_3$, δ ppm): ($CDCl_3$, δ, м.д.): 36.20 ($C^{1''}HCl$); 82.69 (C^1); 113.32 (C^3H_2); 116.92, 117.29, 122.95, 123.10, 129.52, 129.72, 155.13, 156.11 (Ph), 132.30 (C^2). MS (70 eV), m/z (*J*., %): 272/274 $[M]^+$ (<0.1), 236 (<1), 207 (1), 179/181 $[M-OPh]^+$ (1/0.3), 161 (100), 131 (1), 121 (5), 94 (5), 77 (28), 65 (14), 51 (8).

1,1'-[[(2Z)-2-(chloromethylene)cyclopropane-1,1-diyl]bis (oxy)]bis(2-methylbenzene) (**9b**): R_f 0.38 (hexane:AcOEt, 60:1). ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.25 (s, 6H, CH_3; 2H, C^3H_a, C^3H_b), 6.93-7.45 (m, 8H, Ph; 1H, $C^{1'}HCl$). ^{13}C-NMR ($CDCl_3$, δ ppm): 16.42 (CH_3), 21.60 (C^3H_2), 84.66 (C^1), 113.84 ($C^{1'}HCl$), 114.92, 122.56, 126.9, 131.18, 154.28 (Ph), 126.54 (C^2). MS (70 eV), m/z (*J*., %): 300/302 $M^{+\bullet}$ (58/19), 265 $[M-^{\bullet}Cl]^+$ (1), 193/195 (22/8), 165/167 (40/13), 157 (21), 129 (100), 108 (9), 91 (55), 75 (15), 65 (40).

1,1'-[[(2E)-2-(chloromethylene)cyclopropane-1,1-diyl]bis (oxy)]bis(2-methylbenzene) (**10b**): R_f 0.26 (hexane: AcOEt, 60:1). ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.20 (d, 2H, C^3H_a, C^3H_b, ^4J 2.3), 2.26 (s, 6H, CH_3), 6.41 (t, 1H, $C^{1'}HCl$, ^4J 2.3), 6.97-7.66 (m, 8H, Ph). MS (70 eV), m/z (*J*., %): 300/302 $M^{+\bullet}$ (35/12), 265 $[M-^{\bullet}Cl]^+$ (0.5), 193/195 (23/8), 165/167 (34/10), 157 (19), 129 (100), 108 (9), 91 (49), 75 (14), 65 (40).

1,1'-[(2-chloro-3-methylenecyclopropane-1,1-diyl)bis(oxy)]bis(2-methylbenzene) (**11b**): R_f 0.33 (hexane: AcOEt, 60:1). ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.21 (s, 6H, CH_3), 3.96 (t, 1H, C^3HCl, ^4J 2.2), 5.90 (dd, 1H, $C^{1'}H_a$, ^2J 1.5, ^4J 2.2), 6.04 (dd, 1H, $C^{1'}H_b$, ^2J 1.5, ^4J 2.2), 6.92 – 7.43 (m, 8H, Ph). MS (70 eV), m/z (*J*., %): 300/302 $[M]^{+\bullet}$ (<0.1), 265 $[M-^{\bullet}Cl]^+$ (2), 249 (1), 221 (2), 207 (55), 193/195 $[M-^{\bullet}OPh]^+$ (40/14), 178/180 (3/1), 158 (10), 129 (30), 115 (5), 108 (30), 91 (25), 73 (22), 65 (20).

1,1'-[[(2Z)-2-(chloromethylene)cyclopropane-1,1-diyl]bis (oxy)]bis(4-chlorobenzene) (**9c**): R_f 0.35 (hexane:AcOEt, 60:1). ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.15 (d, 2H, C^3H_a, C^3H_b, ^4J 3.2), 6.92 (t, 1H, $C^{1'}HCl$, ^4J 3.2), 7.09-7.30 (m, 8H, Ph). ^{13}C-NMR ($CDCl_3$, δ ppm): 21.06 (C^3H_2), 85.27 (C^1), 114.23 ($C^{1'}HCl$), 118.25, 128.16, 129.48, 154.19 (Ph), 125.23 (C^2). MS (70 eV), m/z (*J*., %): 340/342/344 $M^{+\bullet}$ (1), 339/341/343 $[M-^{\bullet}H]^+$ (1), 305/307/309 (1), 213/215/217 (43/27.5/5), 185/187/189 (45/27.5/5), 177/179 (27/8), 149/151 (100/31), 128/130 (14/4), 125/127 (82/32), 111/113 (46/17), 99/101 (27/10), 85/87 (8.5/3), 75 (75), 63 (14), 50 (15).

1,1'-[[(2E)-2-(chloromethylene)cyclopropane-1,1-diyl]bis (oxy)]bis(4-chlorobenzene) (**10c**): R_f 0.24 (hexane:AcOEt, 60:1). ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.12 (d, 2H, C^3H_a, C^3H_b, ^4J 2.5), 6.39 (t, 1H, $C^{1'}HCl$, ^4J 2.5), 7.09-7.30 (m, 8H, Ph). MS (70 eV), m/z (*J*., %): 340/342/344 $M^{+\bullet}$ (1), 339/341/343 $[M-^{\bullet}H]^+$ (1), 305/307/309 (1), 213/215/217 (45/30/5), 185/187/189 (48/30/5), 177/179 (30/10), 149/151 (100/31), 128/130 (12/4), 125/127 (80/27), 111/113 (48/16), 99/101 (27/10), 85/87 (9/3), 75 (78), 63 (10), 50 (16).

1,1'-[(2-chloro-3-methylenecyclopropane-1,1-diyl)bis (oxy)]bis (4-chlorobenzene) (**11c**): R_f 0.29 (hexane:AcOEt, 60:1). ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 3.93 (t, 1H, C^3HCl, ^4J 2.1), 5.93 (dd, 1H, $C^{1'}H_a$, ^2J 0.8, ^4J 2.1), 6.0 (dd, 1H, $C^{1'}H_b$, ^2J 0.8, ^4J 2.1), 7.06-7.31 (m, 8H, Ph). MS (70 eV), m/z (*J*., %): 340/342/344 $M^{+\bullet}$ (5/3/0.5), 215/217/219 (35/18/5), 213/217/219 (13/9/1.5), 178/180 (100/33), 149/151 (42/15), 128/130 (34.5/10.5), 115/117 (44/15), 111/113 (16/5), 99/101 (16.5/5.5), 85/87 (7/2), 75 (25), 63 (11), 50 (11).

2. Acetals synthesis with phenylpropargyl aldehyde **13a-c**

The solution of 0.003 M 2-brom-2-phenyl-*gem*-dichlorocyclopropane **12** in 1 mL DMF was added dropwise, with intensive stirring to the mixture of 0.007 M phenol **2a-c**, 0.02 M NaOH, 2.5 mL DMF. After 2-4 hours, the reaction mass was diluted with water, extracted with chloroform, washed with water, the solvent was evaporated, the residue was purified by column chromatography, using silica gel, hexane: ethyl acetate 9:1 as eluent.

1,1'-[(1-phenylprop-1-yne-3,3-diyl)bis(oxy)]dibenzene **(13a).** R_f 0.22. ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 7.05-7.40 (m, 15H, Ph; 1H, CH). ^{13}C-NMR ($CDCl_3$, δ ppm): 85.32 (C^2), 95.10 (C^1), 103.39 (C^3), 126.58, 127.61, 128.10, 128.35, 129.00, 131.02, 158.37 (Ph). MS (70 eV), m/z (*J*., %): 300 $[M]^{+\bullet}$ (<0.1), 207 $[M-^{\bullet}OC_6H_5]^+$ (2), 175 (100), 158 (91), 144 (13), 131 (15), 116 (28), 102 (19), 91 (12), 77 (10), 63 (11).

1,1'-[(1-phenylprop-1-yne-3,3-diyl)bis(oxy)]bis(2-methylbenzene) **(13b).** R_f 0.27. ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 2.2 (s, 6H, CH_3), 6.7-7.5 (m, 13H, Ph; 1H, CH). MS (70 eV), m/z (*J*., %): 328 $[M]^{+\bullet}$ (<0.1), 175 (100), 158 (87), 144 (12), 131 (15), 116 (29), 102 (21), 91 (12),77 (10), 63 (12), 51 (4).

1,1'-[(1-phenylprop-1-yne-3,3-diyl)bis(oxy)]bis(4-chlorobenzene) **(13c).** R_f 0.24. ^{1}H-NMR ($CDCl_3$, δ ppm, *J*Hz): 6.90-7.48 (m, 13H, Ph, 1H, CH). MS (70 eV), m/z (*J*., %): 368/370/372 $[M]^{+\bullet}$ (<0.1), 175 (76), 158 (100), 144 (17), 131 (18), 116 (28), 102 (28), 91 (18), 77 (15), 63 (16) 51 (17).

CONCLUSIONS

Using halomethyl-*gem*-dichlorocyclopropanes, corresponding aroxymethyl derivatives and cyclopropanone ketals were obtained. It was demonstrated that catechol reacts through both endocyclic and exocyclic carbon-halogen bonds. For *cis-, trans*-1,1,3-trichloro-2-chloromethylcyclopropanes only CCl_2 group is involved in alkoxy group substitution.

In these conditions presence of bromine atom in the cycle leads to cleaving of the cyclopropane ring producing phenylpropargyl aldehyde acetals.

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