Acta Chemica Scandinavica B 32 (1978) 452—456

Chlorinated Polycyclic Compounds. V. Reactions of Chloro-substituted Dibenzobicyclo[3.2.1]octadienes with Sodium Methoxide

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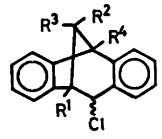
Displacement of chlorine occurred at 4- and 5-positions only when chloro-substituted dibenzobicyclo[3.2.1]octadienes were treated with sodium methoxide in methanol. Hydrolysis of the methoxy derivatives with a mixture of sulfuric acid and acetic acid gave acetates or ketones of the dibenzobicyclo[2.2.2]octadiene series.

Since attempts to synthetize the ketones *7e* and *7f*, required for other studies, by acid catalyzed rearrangement [1,2] of the appropriate dibenzobicyclo[3.2.1]octadiene derivatives [3] were unsuccessful, it was desirable to find a more reactive starting material to effect the conversion. It was found that the chlorides *1o* and *1p*, when treated with sodium methoxide in methanol, gave methoxy derivatives that were readily hydrolyzed to the desired ketones in quantitative yields. In order to locate the methoxy group in the products and to have an idea of the generality of the reaction, all members of the chloride series *1* were subjected to similar reaction conditions. The results are summarized in Table 1.

In spite of the well-known resistance of bridgehead halides toward nucleophilic substitution, reactions of this kind were observed although they occurred at the less hindered side of the molecule only. The inertness of the 8-chlorine atoms must be ascribed to inability to undergo an S_N2 reaction due to steric reasons and to the lack of sufficient stabilisation possibilities required by an S_N1 path. The reactivity of the chlorides unsubstituted at C-5 (*1a*—*1h*) was found to be dependent on the substituent at C-8. Displacement of the 4-chlorine was observed when the *syn*-8-position was unsubstituted (*1a*—*1d*), while the *syn*-8-chloro derivatives (*1e*—*1h*) were only epimerized to the *endo* configuration. The pronounced effect of the *syn*-8-chlorine is in agreement with the idea that the entering group attacks from the *exo* side giving *exo* substitution products, which may then be converted to the *endo* epimers under thermodynamic control.[4,5] When the starting material carried chloro substituents



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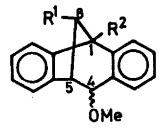
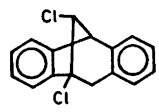


	R^1	R^2
2a	H	H
b	H	Cl
c	Cl	H
d	Cl	Cl

	R^1	R^2	R^3
3a	Cl	H	H
b	Cl	Cl	H
c	Cl	H	Cl
d	H	Cl	Cl
e	Cl	Cl	Cl

	R^1	R^2	R^3
4a	H	Cl	H
b	Cl	H	Cl



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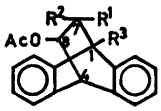

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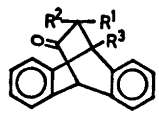
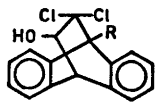
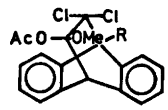


7

	R^1	R^2	R^3
6a,7a	H	H	H
b	H	H	Cl
c	Cl	H	H
d	Cl	H	Cl
e	Cl	Cl	H
f	Cl	Cl	Cl



8a R = H
b R = Cl



9a R = H
b R = Cl

in both the 4- and 5-positions (*1i*–*1p*), the 5-chlorine, giving rise to a more stable carbonium ion, was displaced preferentially. Small amounts of 4,5-dimethoxy compounds were also observed, but not fully characterized. The most important side reaction was reduction of the 4-chlorine to give products of the types *4* and *5*. Examples of dehalogenations with alkoxides appear in the literature but are best known with bromo compounds.[6–8]

The structures of the reaction products are based both on chemical reactions and spectral data. Because hydrolysis of the 4-methoxy derivatives with a mixture of sulfuric acid and acetic acid gave acetates (*6*) and the 4-chloro-5-methoxy derivatives gave ketones (*7*), most of which are known,[1,2,9–11] it was evident that the 1,8-face of the molecule had remained intact and only the 4- or 5-chlorine atoms participated in the reactions. As the acid hydrolysis still left the 4- and 5-positions as possible sites of the methoxy group in the compounds *3*, they were tested with silver acetate and acetic acid. Instead of the expected 4-acetates only rearranged products were obtained: *3a*, *3c* and *3d* gave the same ketones as acid hydrolysis but in the case of *3b* and *3e* the methoxy acetates *9a* and *9b* could be isolated. The greater stability of the latter is probably due to steric hindrance caused by the geminal dichloro group. These structures were confirmed by spectroscopic means and by acid catalyzed hydrolysis to the corresponding ketones. Attempts to hydrolyze the acetoxy group in basic media yielded only mixtures while reduction with lithium aluminium hy-

Table 1. Products from the reactions of the chlorides *1a*–*1p* with sodium methoxide.

Starting material					*endo-exo* ratio	Reaction products
No.	R^1	R^2	R^3	R^4		
1a	H	H	H	H	35:65	80 % *exo-2a*
1b	H	H	H	Cl	25:75	65 % *endo-2b*
1c	H	H	Cl	H	50:50	70 % *exo-2c*
1d	H	H	Cl	Cl	10:90	80 % *endo-2d*
1e	H	Cl	H	H	40:60	80 % *endo-1e*
1f	H	Cl	H	Cl	15:85	95 % *endo-1f*
1g	H	Cl	Cl	H	30:70	70 % *endo-1g*
1h	H	Cl	Cl	Cl	10:90	80 % *endo-1h*
1i	Cl	H	H	H	50:50	Complex mixture
1j	Cl	H	H	Cl	0:100	Complex mixture
1k	Cl	H	Cl	H	20:80	25 % *4a*, 65 % *5*
1l	Cl	H	Cl	Cl	50:50	75 % *3d*
1m	Cl	Cl	H	H	60:40	70 % *3a*, 15 % *endo-1m*
1n	Cl	Cl	H	Cl	45:55	65 % *3c*, 20 % *4b*
1o	Cl	Cl	Cl	H	50:50	90 % *3b*
1p	Cl	Cl	Cl	Cl	50:50	85 % *3e*

dride gave the alcohols *8a* and *8b*. As the 4-methoxy derivatives *2* failed to react with silver acetate and acetic acid, it is evident that the methoxy group in the compounds *3* is at C-5.

The stereostructure at C-4 in the compounds *2* is based on the coupling constants between the 4- and 5-protons.[12] In the compounds *3* the 4-proton absorbs in the range δ 5.47 – 5.99, which is consistent with an *exo*-proton. This is clear, if a comparison is made with the spectra of the chlorides *1*, where the chemical shifts are: δ 5.08 – 5.39 (*endo*-4-H), 5.51 – 5.72 (*exo*-4-H).

The predominant *endo* configuration of the reaction products, both methoxy derivatives and recovered starting chlorides, shows that epimerization at C-4 occurs in the presence of a base. Epimeric mixtures were used as starting material, because preliminary experiments with pure epimers showed no significant difference of behaviour under the solvolysis conditions.

EXPERIMENTAL

For general experimental conditions see Ref. 1.

Preparation of the starting materials. The chlorides *1a*,[4] *1b*,[2] *1c*,[13] *1d*,[1] *1e*,[14] *1f*,[1] *1j*,[2] *1l*,[1] *1n*,[1] *1o* [15] and *1p* [3] have been described earlier.

Preparation of *1g* and *1h*. The alcohol *8a* (see below) (2.0 g) was refluxed for 10 min with a mixture of 5.0 g of PCl_5 and 5.0 g of $POCl_3$. The hot reaction mixture was carefully decomposed with water, extracted twice with chloroform, the chloroform solution washed with $NaHCO_3$ and water, dried and evaporated. The mixture of the reactions products contained *ca.* 20 % of *endo*-4,8,8-trichlorodibenzobicyclo[3.2.1]octadiene (*1g*), m.p. 85 °C, δ 5.61 (*exo*-4-H), 4.28 (1-H), 3.97 (5-H) + 8 Ar-H, $J_{4,5}$ = 5.0 Hz and 80 % of the *exo* epimer, m.p. 105 °C, δ 5.17 (*endo*-4-H), 4.37 (1-H), 4.08 (5-H) + 8 Ar-H, $J_{4,5} < 1$ Hz. Similarly, the alcohol *8b* gave 10 % of 1-*endo*-4,8,8-tetrachlorodibenzobicyclo[3.2.1[octadiene (*1h*), m.p. 103 °C, δ 5.73 (*exo*-4-H), 4.21 (5-H) + 8 Ar-H, $J_{4,5}$ = 4.8 Hz and 90 % of the *exo* epimer, m.p. 177 °C, δ 5.13 (*endo*-4-H), 4.27 (5-H) + 8 Ar-H, $J_{4,5} < 1$ Hz. In both cases the epimers were separated by TLC (elution with light petroleum) and crystallized from EtOH. Approximative yields are based on ^{1}H NMR.

Preparation of *1i*. The chlorides *1i* were best obtained by treatment of dibenzobicyclo[2.2.2]-octadien-7-one (*7a*) [11] with PCl_5 as described above but using a reaction time of 4 h. The product contained *ca.* 50 % of *endo*-4,5-dichlorodibenzobicyclo[3.2.1]octadiene (*1i*), m.p. 121 °C, δ 2.65 (*syn*-8-H), 2.95 (*anti*-8-H), 5.53 (*exo*-4-H), 3.91 (1-H) + 8 Ar-H and 50 % of the *exo* epimer, δ 2.6 – 3.2 (*syn*- and *anti*-8-H), 5.13 (*endo*-4-H), 3.97 (1-H) + 8 Ar-H, $J_{1,8}$ = 4.3 Hz, $J_{8,8}$ = 10.5 Hz. The epimers could not be separated chromatographically, but the *endo* epimer was obtained by treatment of the corresponding alcohol (see below) with PCl_5 for 10 min and crystallization from EtOH. The structures of the chlorides *1i* were confirmed by successive acetolysis, hydrolysis and oxidation reactions.[1,2] Acetolysis of *1i* gave 25 % of 5-chlorodibenzobicyclo[3.2.1]octadien-*endo*-4-yl acetate, m.p. 159 °C, ν_{max} 1725 cm^{-1}, δ 2.69 (syn-8-H), 2.81 (*anti*-8-H), 6.47 (*exo*-4-H), 3.88 (1-H), 2.03 (OAc) + 8 Ar-H, $J_{1,8}$ = 4.0 Hz, $J_{8,8}$ = 11.4 Hz and 75 % of the *exo* epimer, m.p. 101 °C, ν_{max} 1735 cm^{-1}, δ 2.90 (*syn*-8-H), 2.70 (*anti*-8-H), 6.01 (*endo*-4-H), 3.99 (1-H), 2.09 (OAc) + 8 Ar-H, $J_{1,8}$ = 4.0 Hz, $J_{8,8}$ = 10.0 Hz. Hydrolysis of the acetates gave the corresponding *endo* alcohol, m.p. 120 °C, ν_{max} 3330 cm^{-1}, δ 2.60 (*syn*-8-H), 2.79 (*anti*-8-H), 4.96 (*exo*-4-H), 3.84 (1-H), 2.26 (OH) + 8 Ar-H, $J_{1,8}$ = 4.4 Hz, $J_{8,8}$ = 11.0 Hz and the *exo* epimer, m.p. 121 °C, ν_{max} 3220 cm^{-1}, δ 2.90 (*syn*-8-H), 2.64 (*anti*-8-H), 4.48 (*endo*-4-H), 3.93 (1-H), 2.86 (OH) + 8 Ar-H, $J_{1,8}$ = 4.2 Hz, $J_{8,8}$ = 11.0 Hz. On oxidation both alcohols gave the same ketone, 5-chlorodibenzobicyclo[3.2.1]octadien-4-one.[11]

Preparation of *1k*. Hydrolysis [3] of 5-*anti*-8-dichlorodibenzobicyclo[3.2.1]octadien-4-yl acetate [16] and reaction of the epimeric alcohol mixture with PCl_5 for 10 min (see above) gave 10 % of *endo*-4,5-*anti*-8-trichlorodibenzobicyclo-[3.2.1]octadiene (*1k*), δ 4.59 (*syn*-8-H), 5.51 (*exo*-4-H), 4.24 (1-H) + 8 Ar-H and 90 % of the *exo* epimer, m.p. 153 °C, δ 5.19 (*syn*-8-H), 5.32 (*endo*-4-H), 4.24 (1-H) + 8 Ar-H. The *exo* epimer was obtained in a pure state by three recrystallizations from EtOH. The chemical shifts for the *endo* epimer are from the spectrum of the mixture left in the mother liquor.

Preparation of *1m*. The most convenient route to *1m* was *via* acid catalyzed rearrangement [1,2] of 5-*anti*-8-dichlorodibenzobicyclo-[3.2.1]octadien-4-yl acetate [16] to 7-chlorodibenzobicyclo[2.2.2]-octadien-8-one (*7c*) [10,11] and reaction of the latter with PCl_5 for 4 h as described above. This reaction gave *ca.* 15 % of *endo*-4,5-*syn*-8-trichlorodibenzobicyclo[3.2.1]-octadiene (*1m*), m.p. 110 °C, δ 4.81 (*anti*-8-H), 5.57 (*exo*-4-H), 4.06 (1-H) + 8 Ar-H, $J_{1,8}$ = 4.5 Hz and 85 % of the *exo* epimer, m.p. 130 °C, δ 4.79 (*anti*-8-H), 5.08 (*endo*-4-H), 4.14 (1-H) + 8 Ar-H, $J_{1,8}$ = 4.2 Hz. The epimers were separated by TLC (elution with light petroleum) and crystallized from EtOH.

Reactions of the chlorides 1a – 1p *with sodium methoxide*. General method: 5.0 g of clean sodium was dissolved in 50 ml of methanol. The chloride (1.0 g) was added and the mixture refluxed for 24 h. The reaction mixture was poured into water, neutralized with HOAc

and the products isolated by ether extraction. The products were separated by TLC (elution with light petroleum or a 3:1 mixture of light petroleum and chloroform) and purified by crystallization from EtOH. The yields are based on the total amount of the reaction products. The following compounds were obtained:

1a. 80 % of *exo*-4-methoxydibenzobicyclo-[3.2.1]octadiene (*2a*), m.p. 110 °C, δ 2.15 – 2.58 (*syn*- and *anti*-8-H), 4.00 (*endo*-4-H), 3.57 – 3.80 (1-H, 5-H), 3.50 (OMe) + 8 Ar-H.

1b. 65 % of 1-chloro-*endo*-4-methoxydibenzobicyclo[3.2.1]octadiene (*2b*), m.p. 196 °C, δ 2.65 (syn-8-H), 2.92 (anti-8-H), 4.53 (*exo*-4-H), 3.81 (5-H) + 8 Ar-H, $J_{4,5}$ = 5.0 Hz, $J_{5,8}$ = 5.4 Hz.

1c. 70 % of *anti*-8-chloro-*exo*-4-methoxydibenzobicyclo[3.2.1]octadiene (*2c*), m.p. 135 °C, δ 4.83 (*syn*-8-H), 4.16 (*endo*-4-H), 3.99 (1-H), 3.70 (5-H), 3.57 (OMe) + 8 Ar-H, $J_{4,5}$ = 1.8 Hz.

1d. 80 % of 1-*anti*-8-dichloro-*endo*-4-methoxydibenzobicyclo[3.2.1]octadiene (*2d*), m.p. 191 °C, δ 4.57 (*syn*-8-H), 4.58 (*exo*-4-H), 4.09 (5-H), 3.60 (OMe) + 8 Ar-H, $J_{4,5}$ = 5.6 Hz.

The chlorides *1e – 1h* gave mainly the *endo* epimer of the starting material. The chlorides *1i* and *1j* gave complex mixtures containing *ca.* 60 – 70 % of methoxy derivatives (according to ^{1}H NMR). These mixtures were not fractionated.

1k. 65 % of 5-*anti*-8-dichlorodibenzobicyclo-[3.2.1]octadiene (*5*), m.p. 117 °C, δ 4.62 (*syn*-8-H), 3.40 (*endo*-4-H), 3.60 (*exo*-4-H), 4.17 (1-H) + 8 Ar-H, $J_{4,4}$ = 17.0 Hz and 25 % of *anti*-8-chloro-5-methoxydibenzobicyclo[3.2.1]-octadiene (*4a*), m.p. 130 °C, δ 4.63 (*syn*-8-H), 2.82 (*endo*-4-H), 3.60 (*exo*-4-H), 4.13 (1-H), 3.57 (OMe) + 8 Ar-H, $J_{4,4}$ = 16.0 Hz.

1l. 75 % 1-*endo*-4-*anti*-8-trichloro-5-methoxydibenzobicyclo[3.2.1]octadiene (*3d*), m.p. 190 °C, δ 4.70 (*syn*-8-H), 5.65 (*exo*-4-H), 3.54 (OMe).

1m. 70 % of *endo*-4-*syn*-8-dichloro-5-methoxydibenzobicyclo[3.2.1]octadiene (*3a*), m.p. 138 °C, δ 4.90 (*anti*-8-H), 5.47 (*exo*-4-H) 4.11 (1-H), 3.40 (OMe) + 8 Ar-H, $J_{1,8}$ = 4.8 Hz and 15 % of *endo-1m.*

1n. 65 % of 1-*endo*-4-*syn*-8-trichloro-5-methoxydibenzobicyclo[3.2.1]octadiene (*3c*), m.p. 158 °C, δ 4.96 (*anti*-8-H), 5.50 (*exo*-4-H) 3.47 (OMe) + 8 Ar-H and 20 % of 1-*syn*-8-dichloro-5-methoxydibenzobicyclo[3.2.1]octadiene (*4b*), m.p. 147 °C, δ 4.82 (*anti*-8-H), 2.70 (*endo*-4-H), 3.34 (*exo*-4-H), 3.34 (OMe) + 8 Ar-H, $J_{4,4}$ = 17.0 Hz.

1o. 90 % of *endo*-4,8,8-trichloro-5-methoxydibenzobicyclo[3.2.1]octadiene (*3b*), m.p. 158 °C, δ 5.87 (*exo*-4-H), 4.40 (1-H), 3.85 (OMe) + 8 Ar-H, *m/e* 338(34), 303(100).

1p. 85 % of 1-*endo*-4,8,8-tetrachloro-5-methoxydibenzobicyclo[3.2.1]octadiene (*3e*), m.p. 203 °C, δ 5.99 (*exo*-4-H), 3.87 (OMe) + 8 Ar-H, *m/e* 372 (13), 337(100).

Hydrolysis of the methoxy derivatives 2a – 2d *and* 3a – 3e. The methoxy compound (0.05 – 0.2 g) was refluxed for 40 min with a mixture of 1.5 g of H_2SO_4 and 3.5 g of HOAc. The mixture was poured into water and the product isolated by ether extraction. ^{1}H NMR and TLC examination showed that the reaction was essentially quantitative in each case. Following compounds were obtained (starting material, hydrolysis product): *2a, 6a*;[9] *2b, 6b*;[2] *2c, 6c*;[10] *2d, 6d*,[11] *3a, 7c*;[10,11] *3b, 7e*; *3c, 7d*;[1] *3d, 7d*;[1] *3e, 7f.*

The new compounds had the following properties: 7,7-dichlorodibenzobicyclo[2.2.2]octadien-8-one, (*7e*) m.p. 142 °C, ν_{max} 1758 cm^{-1}, δ 4.92 (1-H), 4.79 (4-H) + 8 Ar-H, *m/e* 288(8), 178(100) and 1,7,7-trichlorodibenzobicyclo-[2.2.2]octadien-8-one (*7f*), m.p. 158 °C, ν_{max} 1760 cm^{-1}, δ 4.87 (4-H) + 8 Ar-H, *m/e* 322(2), 212(100).

Reduction of the ketones 7e *and* 7f *with sodium borohydride and acetylation of the resulting alcohols.* A solution of 10 mmol of the ketone (2.89 g of *7e* or 3.24 g of *7f*) and 0.38 g (10 mmol) of $NaBH_4$ in 100 ml of EtOH was stirred for 80 min at room temperature. The solution was poured into water, HCl added and the aqueous solution extracted twice with ether. The ethereal solution was dried and evaporated. The analytical samples were crystallized from 80 % aqueous EtOH. The ketone *7e* gave 7,7-dichlorodibenzobicyclo[2.2.2]octadien-8-ol (*8a*), m.p. 131 °C, ν_{max} 3420, 3520 cm^{-1}, δ 4.73 (1-H) 4.22 (4-H), 4.27 (8-H), 1.77 (OH) + 8 Ar-H, $J_{4,8}$ = 2.6 Hz and *7f* gave 1,7,7-trichlorodibenzobicyclo[2.2.2]octadien-8-ol (*8b*), m.p. 172 °C, ν_{max} 3540, 3560 cm^{-1}, δ 4.20 (4-H), 4.37 (8-H), 2.04 (OH), $J_{4,8}$ = 2.6 Hz.

The alcohol *8a* (0.2 g) refluxed for 80 min with a mixture of 10 ml of Ac_2O and 1 ml of pyridine, gave the acetate *6e*, m.p. 139 °C, ν_{max} 1743 cm^{-1}, δ 4.70 (1-H), 4.23 (4-H), 5.29 (8-H), 2.00 (OAc) + 8 Ar-H, $J_{4,8}$ = 2.6 Hz. Similarly, *8b* gave the acetate *6f*, m.p. 170 °C, ν_{max} 1754 cm^{-1}, δ 4.23 (4-H), 5.44 (8-H), 2.02 (OAc) + 8 Ar-H, $J_{4,8}$ = 2.6 Hz. The acetates were crystallized from EtOH.

Acetolysis of the methoxy derivatives 3a – 3e. The compound *3b* (0.68 g, 2.0 mmol) was refluxed for 10 min with 0.40 g (2.4 mmol) of AgOAc and 20 ml of HOAc. Acetic acid was removed under reduced pressure, the residue dissolved in acetone, the solution filtered and evaporated. According to ^{1}H NMR, the mixture contained *ca.* 30 % of unreacted *3b*, 20 % of the ketone *7e* and 50 % of an unknown acetate. The components were separated by TLC (elution with a 1:1 mixture of light petroleum and chloroform). The last compound was crystallized from EtOH to give 7,7-dichloro-8-methoxydibenzobicyclo[2.2.2]octadien-8-yl acetate (*9a*), m.p. 203 °C, ν_{max} 1740 cm^{-1} δ 4.62 (1-H), 5.33 (4-H), 3.35 (OMe), 1.81 (OAc) + 8 Ar-H, *m/e* 319 (M-43, 8), 178 (100).

Similarly, *3e* gave 30 % of unreacted starting material, 10 % of the ketone *7f* and 60 % of 1,7,7-trichloro-8-methoxydibenzobicyclo[2.2.2]-octadien-8-yl acetate (*9b*), m.p. 160 °C, ν_{max} 1750 cm^{-1}, δ 5.42 (4-H), 3.41 (OMe), 1.85 (OAc) + 8 Ar-H, *m/e* 353 (M-43, 0.7), 212(100).

The only reaction products from the compounds *3a*, *3c* and *3d* were the ketones *7c*, *7d* and *7d*, respectively.

Acknowledgements. The author wishes to express his thanks to Professor Jarl Gripenberg (Emeritus) and to Professor Tapio Hase, for valuable comments.

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Received February 27, 1978.