

Bridged polycyclic compounds. 88. Multiple intermediates in solvolysis of certain bridged bicyclic and tricyclic compounds¹

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This paper is dedicated to Professor Arthur N. Bourns

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Acetolyses of 7-chloromethyl- and 7-bromomethylidibenzobicyclo[2.2.2]octatrienes, 8-chloro- and 8-bromo-7-methylenedibenzobicyclo[2.2.2]octa-2,5-dienes, 4-bromo-8-methylenedibenzobicyclo[3.2.1]octa-2,6-diene, and 2-chlorodibenzotricyclo[3.2.2.0^{2,4}]nonadiene were carried out. The results observed were consistent with the involvement of two carbocations interconverting by a Wagner–Meerwein rearrangement, and with the absence of the 1-dibenzosemibullvalenylcarbiny cation, which is the cyclopropylcarbiny isomer of one of the cations produced. The preparations of the reactants and products are described.

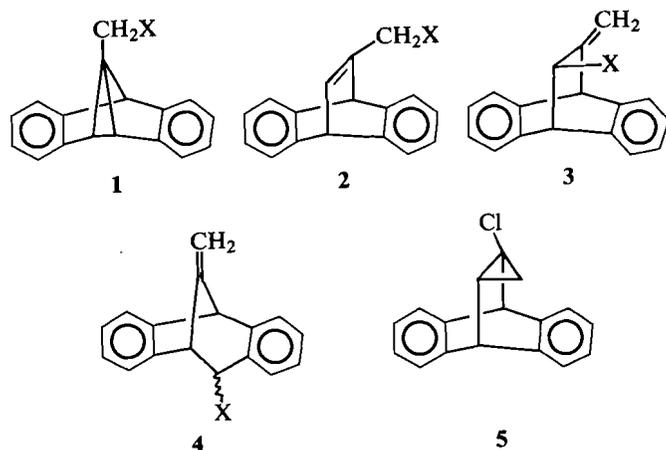
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On a effectué les acétolyses des composés suivants: les chlorométhyl-7 et bromométhyl-7 dibenzobicyclo[2.2.2]octatriènes, les chloro-8 et bromo-8 méthylène-7 dibenzobicyclo[2.2.2]octatriènes-2,5, le bromo-4 méthylène-8 dibenzobicyclo[3.2.1]octadiène-2,6 et le chloro-2 dibenzotricyclo[3.2.2.0^{2,4}]nonadiène. Les résultats observés sont en accord avec un mécanisme impliquant la présence de deux carbocations qui s'interconvertissent par une transposition de Wagner–Meerwein et l'absence du cation dibenzosemibullvalénylcarbiny-1 qui est l'isomère cyclopropylcarbiny-1 de l'un des cations produits. On décrit les préparations des réactifs et des produits.

[Traduit par la revue]

Introduction

Some years ago (2), the solvolysis of **1-Br** (1-bromomethylidibenzosemibullvalene) and the solvolytic deamination of **1-NH₂** were described and the nature of the intermediates involved in the formation of the solvolytic products was discussed. The purpose of this paper is to describe silver-ion assisted acetolyses of the corresponding allylic isomers (**2** and **3**, X = Cl, Br), the homoallylic bromide **4-Br**, and the cyclopropyl chloride **5**, all of which might be expected to share in the manifold of carbocations available to **1**. We were particularly interested in the nature of the products of these ground-state solvolyses, in order to compare them with those of ground-state solvolysis of **1** and those of photosolvolyses of similar or identical compounds.

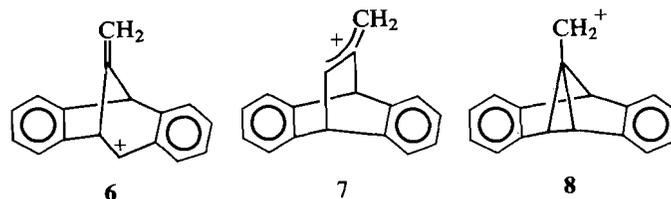


Silver-ion induced acetolysis (2) of **1-Br** gave, in addition to a small amount of 3,4-benzofluorene,³ a mixture of the acetates **2**,

3, and **4** (X = OAc). The ratios of the acetate products (rationalized to 100) are given in Table 1, as are corresponding results for **2-Br**, **3-Br**, and **4-Br**. The latter compounds gave no benzofluorene, consistent with the idea³ that the progenitor of this hydrocarbon is a species not reached in the carbocation manifold from **2-Br**, **3-Br**, or **4-Br**. The ratios of products are, within experimental error, identical for the isomeric allylic compounds **2-Br** and **3-Br**. It was first assumed that the experiments conducted at 100°C for approximately an hour were kinetically controlled ones involving silver acetate. However, experiments on corresponding chlorides (see below) made clear that the experiments, which involved addition of silver acetate after the solution had been brought to temperature, allowed for substantial acetolysis and epimerization before solvolysis, and allowed for partial acid-catalyzed *exo-endo* transformation of the benzylic acetates **4** and for a partial conversion of **4-OAc** to the more stable **2-OAc** and **3-OAc** isomers.

When the acetate mixtures were allowed to stand in acetic acid containing 0.5 M perchloric acid for 90 h, conversion to **3-OAc** was substantially complete (95% could be isolated) (2).

It seems possible to conclude from these experiments that the carbocations **6** and **7** are involved, **6** leading to *exo* and *endo*-**4** acetates and **7** to **2**- and **3**-acetates. Reproducibility of conditions is not precise enough, in our opinion, to allow confidence in the assumption (2) that **6** and **7** are interconvertible at a rate substantially slower than capture, as this assumption would lead to substantially different mixtures from the benzylic and the allylic isomers, although the slightly greater amount of **4-OAc**



¹For previous paper in series, see ref. 1.

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³Plausible reaction paths for the formation of this hydrocarbon were discussed earlier (2), as were results on the deamination of **1-NH₂**.

TABLE 1. Silver-ion induced acetolysis of certain bromides

Substrate	Temperature, °C	Time, min	Order of AgOAc addition ^a	Composition of acetate product, %		
				2-OAc	3-OAc	4-OAc ^c
1-Br	100	65	Last	28	29	43
	90	45	Last	26	29	43
2-Br	100	45	Last	35	37	28
3-Br	100	45	Last	39	32	29
	97	840	Last	32	44	24
	23	300	Last	34	30	36
4-Br ^b	100	1 ^d	First	23	30	47
	90	45	Last	21	23	56

^aFor those samples marked "last," the solutions of alkyl bromide were brought to temperature and silver acetate was then added; for that marked "first," the alkyl halide was added to a solution of silver acetate already at temperature.

^bComposition of product computed from that of a reactant mixture of 72% 4-Br, 10% 2-Br, and 18% 3-Br.

^cThe major portion of these product mixtures was *exo*; the fraction of *endo* epimer increased with increasing time and (or) severity.

^dTime of heating after RBr added.

TABLE 2. Solvolyses of certain bicyclic and tricyclic chlorides in acetic acid at reflux

Substrate	Time	Order of AgOAc addition	RCl recovered	Composition of acetate product, %		
				2-OAc	3-OAc	4-OAc
2-Cl	2 h	None	32% 2-Cl, 28% 3-Cl	27	43	30
	35 min ^a	Last	0	26	43	31
	<1 min ^a	Last	19% 2-Cl, 18% 3-Cl	29	41	30
	1 min ^b	First	8% 3-Cl	20	39	42
3-Cl	3 min ^b	First	62% 3-Cl, trace 2-Cl	21	35	44
	1 min ^b	First	36% 3-Cl	22	34	44
5-Cl	23 h	Last	66% 5-Cl	25	37	39
	72 h	Last	48% 5-Cl	25	43	32
	9 d	Last	—	23	53	24

^aTime of heating after AgOAc added.

^bTime of heating after RCl added.

from 4-Br suggests this. It is clear, however, from the absence of 1-OAc and benzofluorene, that interconversion with 8 (homoallyl → cyclopropylcarbonyl isomerization) is not important in these experiments.

The chlorides 2-Cl, 3-Cl, and 5-Cl were also subjected to acetolysis (Table 2). With 2-Cl, in the absence of silver acetate (or when silver acetate was added after the solution had been brought to reflux and reaction was terminated by pouring into cold water as soon as possible after the addition), there was substantial conversion to the more stable allylic isomer 3-Cl, along with solvolysis. On the other hand, solvolysis of 3-Cl, which was significantly less reactive than 2-Cl, was accompanied by, at most, trace rearrangement to 2-Cl. Thus, like the acetates, 3-Cl is thermodynamically more stable than its allylic isomer 2-Cl.

In the experiments where the alkyl chlorides were added to a refluxing solution of silver acetate in acetic acid, and the reaction was terminated after a few minutes (presumably kinetic control) with 2-Cl, 3-Cl, and 3-Br, the reaction mixture was somewhat richer in 4-OAc (42–47%) and poorer in 2-OAc than

those prepared under more severe conditions. These results make it clear that 4-OAc is converted to both 2-OAc and 3-OAc and, when previous work (2) on these acetates is considered, that, considerably less rapidly, 2-OAc is converted to 3-OAc. This same phenomenon can be noted in the silver-ion promoted acetolysis of 5-Cl, which gives mixtures comparable to those from the other isomers at shorter times, and, at longer times, shows the tendency to give mixtures richer in 3-OAc, consonant with its stability (2). The low reactivity of 5 is obviously a function of its being a chlorocyclopropane (3), and the products are consistent with those of other bridged systems (see, for example, ref. 4), and with the intervention of ions 6 and 7 as the reactive intermediates in the process.

Photochemical results are recorded in the accompanying paper (5), and discussions of the differences between the photoreactions and ground-state reactions are also given there.

Reagents and products

Compound 2-Br was prepared, essentially as described by Shenoy (6), by treatment of 2-OH (2, 6) with phosphorus

tribromide in pyridine. The other allylic isomer, 3-Br, was prepared by a sequence in which allyl chloride was added to anthracene, elimination of hydrogen chloride gave the olefin 3-H, and bromination with *N*-bromosuccinimide gave largely 3-Br. The preparation of 4-Br was carried out by heating 1-Br (2) in xylene; a mixture containing 18% of 3-Br, 10% of 2-Br, and 72% of 4-Br was produced. The lability of 4-Br made purification difficult, hence this mixture was used in the reactions. The acetates 1-OAc, 2-OAc, 3-OAc, and 4-OAc have already been reported (2). 2-Cl was prepared from 2-OH using the method of Young *et al.* (7), for the conversion of primary allylic alcohols into the corresponding chlorides, without allylic rearrangement. We were unable to convert 2-OH or 3-OH into mixtures of allylic chlorides from which pure samples of 3-Cl could be obtained (although it is now clear (see above) that thermolysis of these mixtures would have given 3-Cl). We therefore prepared 3-Cl by treatment of 7-chloro-8-chloromethyldibenzobicyclo[2.2.2]octa-2,5-diene (prepared from anthracene and 1,3-dichloropropene) with base, following the concept of Jarvis (8) regarding elimination in such systems. 5-Cl was prepared by triplet-sensitized irradiation of 2-Cl or 3-Cl (5).

Experimental

The ^1H nmr spectra were determined with a Varian A-60A 60-MHz instrument (bromide work) and a Varian EM-390 (90-MHz) instrument (chloride work). Analyses were by Galbraith Laboratories, Inc., Knoxville, TN.

Preparation of 7-bromomethyldibenzobicyclo[2.2.2]octatriene (2-Br)

A mixture of 250 mg (1.07 mmol) of 2-OH (2), 2.8 g (10.5 mmol) of PBr_3 , 1 mL of pyridine, and 25 mL of reagent-grade benzene was heated and stirred for 20 min at 60°C, following the procedure of Shenoy (6). After being cooled, the mixture was extracted several times with water, aqueous NaHCO_3 , and water. The benzene solution was dried (MgSO_4); evaporation of the solvent left an oil that crystallized from petroleum ether (bp 40–60°C) to give 282 mg (92%) of 2-Br, mp 132–135°C (lit. (6) mp 142–144°C); ^1H nmr (CDCl_3), δ : 7.0 (dt, 1H, H-8, $J_{4,8} = 6.5$ Hz, $J_{8,9} = 0.5$ Hz), 5.28 (s, 1H, H-1), 5.22 (d, 1H, H-4, $J_{4,8} = 6.5$ Hz), 4.40 (d, 2H, H-9, $J_{8,9} = 0.5$ Hz).

Preparation of 7-methylene-8-bromodibenzobicyclo[2.2.2]octadiene (3-Br)

A solution of 7.73 g (35.4 mmol) of 7-methylenedibenzobicyclo[2.2.2]octadiene (9), 7.9 g (35 mmol) of *N*-bromosuccinimide, and a catalytic amount of azobisisobutyronitrile in 90 mL of reagent-grade carbon tetrachloride was irradiated with a 100-W unfrosted tungsten lamp for 3½ h. The heat from the lamp caused the solution to reflux gently. The mixture was cooled and the insoluble succinimide was filtered. The filtrate was evaporated *in vacuo*. The resulting oil was chromatographed on Merck 71707 alumina and eluted rapidly with petroleum ether, bp 40–60°C. The major fraction was collected. Several recrystallizations from petroleum ether (bp 40–60°C) produced 6.2 g (60%) of 3-Br, mp 121–122°C; ^1H nmr (CDCl_3), δ : 5.40 (d, 1H, H-9, $J_{8,9} = 1.5$ Hz), 5.23 (d, 1H, H-9, $J_{8,9} = 2.0$ Hz), 5.90 (m, 1H, H-8, $J_{4,8} = 3.0$, $J_{8,9} = 2.0$, 1.5 Hz), 4.72 (s, 1H, H-1), 4.54 (d, 1H, H-4, $J_{4,8} = 3.0$ Hz).

Anal. calcd. for $\text{C}_{17}\text{H}_{13}\text{Br}$: C 68.70, H 4.41; found: C 68.75, H 4.57.

Thermal isomerization of 1-bromomethyldibenzobicyclo[3.3.0.0^{2,8}]octadiene (1-Br)

A solution of 70 mg (0.24 mmol) of 1-Br in 50 mL of *m*-xylene was heated at reflux for 15 h. The *m*-xylene was removed *in vacuo* to give a yellow oily residue; ^1H nmr analysis showed 46% of *exo*-4-Br, 26% of *endo*-4-Br, 18% of 3-Br, and 10% of 2-Br. The mixture was analysed from the area of the following absorptions in the ^1H nmr spectrum: H-4, δ 5.90 of *endo*-4-Br; H-4, δ 5.65 of *exo*-4-Br; H-9, δ 4.40 of

2-Br; H-1, s, δ 4.72 and H-4, d, δ 4.54 of 3-Br. This mixture resisted attempts at separation and purification by either chromatography or crystallization due to the lability of the compounds. The yield of rearranged products was quantitative. The ^1H nmr spectra of *exo*- and *endo*-4-Br reported are those provided by analysis of the spectrum of the mixture: *exo*-4-Br: ^1H nmr (CDCl_3), δ : 5.65 (d, 1H, H-4, $J_{4,5} = 2.2$ Hz), 5.07, 5.03 (2s, 2H, H-9), 4.40 (s, 1H, H-1); *endo*-4-Br: ^1H nmr (CDCl_3), δ : 5.90 (d, 1H, H-4, $J_{4,5} = 5.5$ Hz), 4.98, 4.92 (2s, 2H, H-9), 4.40 (s, 1H, H-1), 4.05 (d, 1H, H-5, $J_{4,5} = 5.5$ Hz).

Silver-assisted solvolysis of 7-bromomethyldibenzobicyclo[2.2.2]octatriene (2-Br)

To a solution of 500 mg (1.68 mmol) of 2-Br in 15 mL of acetic acid at 100°C was added, with stirring, 300 mg (1.8 mmol) of solid silver acetate. The reaction mixture was stirred for 45 min, cooled, and filtered. Dilution of the filtrate with 150 mL of cold water produced a cloudy precipitate, which was extracted with several 50-mL portions of ether. The combined ether layers were washed with water and saturated sodium bicarbonate solution until neutral, then dried (MgSO_4). Evaporation of the solvent *in vacuo* produced 460 mg (98%) of an oil whose ^1H nmr spectrum⁴ indicated it contained 35% of 2-OAc, 37% of 3-OAc, 22% of *endo*-4-OAc, and 6% of *exo*-4-OAc. This analysis was confirmed, as was that of all other solvolyses of the bromides, by reduction with LiAlH_4 to the corresponding alcohols and analysis of the ^1H nmr spectra of that mixture.

Silver-assisted solvolysis of 4-bromo-8-methylenedibenzobicyclo[3.2.1]octadiene (4-Br)

To a solution of 70 mg (0.24 mmol) of the mixture of bromides from the thermolysis of 1-Br described above, in 30 mL of acetic acid heated at 90°C, was added 100 mg (0.6 mmol) of silver acetate. The reaction was stirred at 90°C for 45 min, cooled, and filtered. The clear filtrate was diluted with 150 mL of water and extracted with several portions of ether. The combined ether layers were washed with water and saturated sodium bicarbonate solution until neutral, and dried (MgSO_4). Evaporation of the solvent gave 66 mg of an oil (100%) whose ^1H nmr spectrum⁴ indicated a composition of 56% 4-OAc, 23% of 3-OAc, and 21% of 2-OAc.

Silver-assisted solvolysis of 7-methylene-8-bromodibenzobicyclo[2.2.2]octadiene (3-Br)

A suspension of 1.24 g (4.18 mmol) of 3-Br in 33 mL of acetic acid was heated to 100°C, after which silver acetate (0.70 g, 4.18 mmol) was added. The reaction mixture was heated at 100°C for 45 min, cooled, and filtered. Dilution of the filtrate with 150 mL of cold water resulted in a white precipitate, which was extracted with several 50-mL portions of ether. The combined ether portions were washed with water and saturated sodium bicarbonate solution. The ether was then dried (MgSO_4) and evaporated *in vacuo* to yield 1.65 g (97%) of an oil. The ^1H nmr spectrum of the mixture indicated the formation of 24% of *exo*-4-OAc, 5% of *endo*-4-OAc, 32% of 3-OAc, and 39% of 2-OAc.

Preparation of 8-chloro-7-methylenedibenzobicyclo[2.2.2]octadiene (3-Cl)

To a solution of 4.0 g (14 mmol) of 7-chloro-8-chloromethyldibenzobicyclo[2.2.2]octadiene (10) in 100 mL of dry dimethyl sulfoxide was added 2.3 g (21 mmol) of potassium *tert*-butoxide. The reaction mixture was stirred for 12 h at room temperature, and was then quenched by addition of 500 mL of water. The resulting suspension was extracted with several portions of ether. The combined ether layers were washed with 1 *M* hydrochloric acid and with water, then dried (MgSO_4). Evaporation of the solvent produced a yellow solid. Recrystallization from water–acetone yielded 2.5 g (71%) of crystalline 3-Cl, mp 132–133°C; ^1H nmr (CDCl_3), δ : 5.35 (d, 1H, H-9, $J_{8,9} = 2.0$ Hz), 5.17 (d, 1H, H-9, $J_{8,9} = 1.8$ Hz), 4.71 (s, 1H, H-1), 4.70 (m, 1H, H-8, $J_{4,8} = 3.0$, $J_{8,9} = 2.0$, 1.8 Hz), 4.48 (d, 1H, H-4, $J_{4,8} = 3.0$ Hz). Anal. calcd. for $\text{C}_{17}\text{H}_{13}\text{Cl}$: C 80.79, H 5.19; found: C 80.91, H 5.01.

⁴The properties of the acetate products and of the corresponding alcohols as well have already been reported (2).

Preparation of 7-chloromethyldibenzobicyclo[2.2.2]octatriene (2-Cl)

To a solution of 3.5 g (15 mmol) of 7-hydroxymethyldibenzobicyclo[2.2.2]octatriene 2-OH (2) and 1.5 g (19 mmol) of pyridine in 200 mL of anhydrous ethyl ether was added (dropwise) a solution of 2.0 g (17 mmol) of thionyl chloride dissolved in 50 mL of anhydrous ether. After stirring overnight, the reaction mixture was poured into 350 mL of cold water. The ether layer was separated, and the water layer extracted twice with ether. The combined ether extracts were washed twice with sodium bicarbonate solution and twice with water. After the solution was dried (MgSO₄), removal of the solvent by distillation left a solid residue (3.8 g, 100%). Recrystallization from petroleum ether (bp 40–60°C) produced more than 90% of 2-Cl, mp 147–148°C; ¹H nmr, δ: 7.41–6.91 (m, 8H, aromatic H), 6.80 (dt, 1H, H-8, *J*_{4,8} = 6 Hz, *J*_{8,9} = 1 Hz), 5.10 (s, 1H, H-1), 5.03 (d, 1H, H-4, *J*_{4,8} = 6 Hz), 4.22 (d, 2H, H-9, *J*_{8,9} = 1 Hz). *Anal.* calcd. for C₁₇H₁₃Cl: C 80.79, H 5.19; found: C 80.70, H 5.35.

Preparation of 2-chlorodibenzotricyclo[3.2.2.0^{2,4}]nonadiene (5)

A solution of 1.9 g (7.5 mmol) of 2-Cl and 12 mL of acetophenone in 65 mL of acetonitrile in a Pyrex tube was deoxygenated by nitrogen bubbling for 20 min. It was then irradiated at 300 nm for 4 days with an Hanovia 500-W lamp (pyrex filter). The solvent was distilled off and the residual oil was chromatographed on 600 mL of a 60–200 mesh silica gel column. Elution with 10% benzene in petroleum ether (bp 40–60°C) gave 5, contaminated with acetophenone. Steam distillation removed most of the acetophenone. The residue (1.2 g, 63%) contained 80% product and 20% acetophenone. Recrystallization from hexane gave pure 5, mp 121–122°C; ¹H nmr (CDCl₃), δ: 7.40–7.00 (m, 8H, aromatic protons), 4.51 (s, 1H, H-1), 4.37 (d, 1H, H-5, *J*_{4,5} = 5 Hz), 1.80 (dt, 1H, H-4, *J*_{4,5} = 5 Hz, *J*_{3anti,4} = 9 Hz, *J*_{3syn,4} = 5 Hz), 1.17 (dd, 1H, H-3_{anti}, *J*_{3anti,4} = 9 Hz, *J*_{3anti,3syn} = 7 Hz), 0.55 (dd, 1H, H-3_{syn}, *J*_{3anti,3syn} = 7, *J*_{3syn,4} = 5 Hz). *Anal.* calcd. for C₁₇H₁₃Cl: C 80.79, H 5.19; found: C 80.59, H 5.13.

Thermal rearrangement of 2-Cl to 3-Cl

A solution of 66 mg (0.26 mmol) of 2-Cl and 31 mg of anhydrous ferric chloride in 10 mL of carbon tetrachloride was heated at reflux for 2 h. After dilution with more carbon tetrachloride, the solution was washed with water, then chromatographed on a cold short silica-gel column to remove traces of ferric chloride. Elution with hexane gave a solution that, upon removal of the solvent by distillation, left a residue whose ¹H nmr spectrum was essentially that of 3-Cl.

Acetolysis of 2-Cl in acetic acid

A solution of 18 mg (0.07 mmol) of 2-Cl in 10 mL of acetic acid was heated at reflux for 2 h. After work-up similar to that described above for the bromides, ¹H nmr analysis indicated 32% of 2-Cl remaining, 28% had been isomerized to 3-Cl, while acetolysis was also seen (17% of 3-OAc, 11% of 2-OAc, and 12% of 4-OAc).

Silver-assisted solvolyses of 2-Cl, 3-Cl, and 5-Cl

These experiments were generally conducted with about 0.01–0.04 *M* alkyl chloride in glacial acetic acid, and with 1.5–10 times the equivalent amount of silver acetate. Recoveries were close to quantitative. Conditions and results are tabulated in Table 2. When silver acetate is described as added "last," the alkyl chloride solution was heated to reflux before addition of silver acetate. In the "first" cases, a solution or suspension of silver acetate in acetic acid was brought to reflux before addition of solid alkyl chloride.

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