verged with $R = 0.075$ and $R_w = 0.056$. A final difference map had no chemically significant features. In the refinement cycles weights were derived from counting statistics, and scattering factor data were taken from ref 28.

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Registry No. 1, 60705-62-6; 3, 103240-05-7; 3·CH_3CN, 103258-02-2.

Supplementary Material Available: Details of molecular geometry (Table 1), final atomic coordinates (Table 2), tables of calculated hydrogen corrdinates (Table 3), and thermal parameters (Table 4) (5 pages). Ordering information is given on any current masthead page.

(28) *International Tables for X-ray Crystallography*; Kynoch: Birmingham, 1974; Vol. IV. (Present distributor: D. Reidel, Dordrecht.)

Trapping of the 6,6-Dimethylbicyclo[3.1.1]hept-2-yl Free Radical by S_H2 Reaction upon Peracid

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The free radical 6,6-dimethylbicyclo[3.1.1]hept-2-yl (**7**) has been obtained by decarboxylation of *cis*- or *trans*-6,6-dimethylbicyclo[3.1.1]heptane-2-peroxycarboxylic acids (**6a** and **6b**). **7** trapped by reaction with the initial peracid gave a stereochemical mixture of α- and β-nopinol (**8a** and **8b**). The ratio **8b**/**8a** is around 12, independent of the initial peracid **6a** or **6b** and its initial concentration. This value is mainly due to the steric effect of one of the methyl groups branched on C_7 in **7**. The structure of **7** is discussed. By a competitive reaction **7** undergoes ring opening to afford 2-(3-cyclohexenyl)-2-propyl free radical (**9**) which by reaction upon peracid leads to 2-(3-cyclohexenyl)-2-propanol (**10**). **7** was successfully trapped because its reaction with peracid is rapid enough. The ratio of the two alcohols **8**/**10** leads to an estimation of 1×10^6 M^{-1} s^{-1} for the rate constant of the reaction of **7** with peracids.

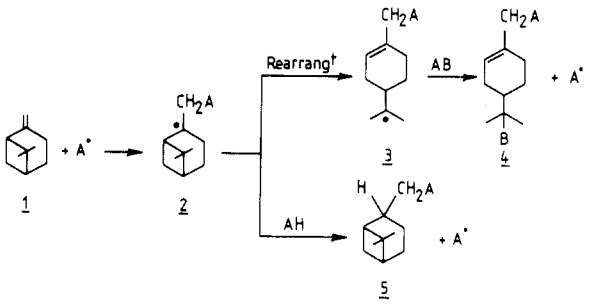
In all examples given in the literature,[1] radicals of type **2** rearrange into radicals of type **3** by opening of the cyclobutane ring and give products of type **4** (see Scheme I).

Nevertheless, the unrearranged product **5** was obtained once[2] when **2** was generated in the presence of thiols: in these conditions H transfer from the thiols by **2** competes with the opening of the cyclobutane ring.

In preceding works[3] we had shown that peracids, RCO_3H, in boiling hydrocarbon solution decarboxylate rapidly into the corresponding alcohols, ROH, through the chain mechanism given in Scheme II.

The rate-determining step of this chain mechanism is step 3. Its activation energy was estimated to be around 4 kcal/mol for the bicyclo[2.2.1]hept-1-yl[3] radical. For cyclohexyl-type radicals the activation energy is still less than 4 kcal/mol.[3b] One more example of the velocity of reaction 3 comes from the retention of configuration observed with the *cis*- and *trans*-decahydronaphthalen-9-yl radicals.[4] The decompositions of peracids **6a** and **6b** have been undertaken in order to study the behavior of the radical **7** in these conditions. The different steps of the reaction are described on Scheme III.

Scheme I



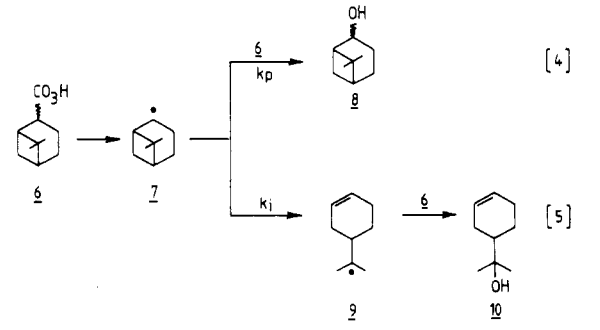
Scheme II

$$R\text{-}CO_3H \longrightarrow R\,CO_2^{\bullet} + {}^{\bullet}OH \quad [1]$$

$$R\text{-}CO_2^{\bullet} \longrightarrow R^{\bullet} + CO_2 \quad [2]$$

$$R^{\bullet} + R\,CO_3H \longrightarrow ROH + RCO_2^{\bullet} \quad [3]$$

Scheme III



This paper reports the first trapping of **7**, by peracid, before opening of the cyclobutane ring. From the ratio of the two alcohol epimers **8a** and **8b**, information concerning the structure of **7** has been obtained. From the relative

(1) (a) Cazaux, M.; Maillard, B.; Lalande, R. *C. R. Seances Acad. Sci., Ser. C* **1972**, *275*, 1133. (b) Gaiffe, A.; Castanet, J. *C. R. Seances Acad. Sci., Ser. C* **1971**, *272*, 96. (c) Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* **1971**, 461. (d) Filliatre, C.; Villenave, J.; Prevot, J. *Bull. Soc. Chim. Fr.* **1979**, 473. (e) Villenave, J.; Filliatre, C.; Prevot, J. *Bull. Soc. Chim. Fr.* **1979**, 479. (f) Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* **1972**, 1887. (g) Paskoff, B.; Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* **1970**, 624. (h) Cazaux, M.; Lalande, R. *Bull. Soc. Chim. Fr.* **1972**, 1984. (i) Lallemand, J. *Tetrahedron Lett.* **1975**, 1217. (j) Kang, Y.; Kice, J. *J. Org. Chem.* **1984**, *49*, 1507. (k) Battisteux, D.; Haseldine, D. *Synth. Commun.* **1984**, *14*, 293. (l) Brace, N. *J. Org. Chem.* **1962**, *27*, 3027.

(2) Claisse, J.; Davies, D.; Parfitt, L. *J. Chem. Soc. C* **1970**, 258.

(3) (a) Fossey, J.; Lefort, D. *Tetrahedron* **1980**, *36*, 1023. (b) Lefort, D.; Fossey, J.; Gruselle, M.; Nedelec, J. Y.; Sorba, J. *Tetrahedron* **1985**, *41*, 4237.

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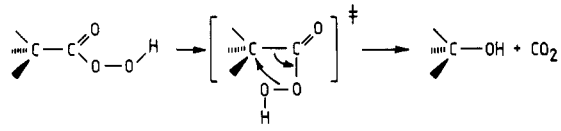
Table I. Alcohol Distribution Obtained by Decomposition of *cis*- or *trans*-Peroxymyrtanic Acids[a-c]

peracids conc, M		alcohol 10	α-nopinol[d] (8a)	β-nopinol[d] (8b)	8b/8a	(8a + 8b)/10
0.1	trans	89	0.4 ± 0.2	9.2 ± 0.4	24 ± 8	0.11
	cis	82	1.1 ± 0.2	17 ± 0.5	15 ± 2	0.22
0.5	trans	76	2.3 ± 0.5	21 ± 2	9 ± 2	0.31
	cis	78	2.1 ± 0.5	21 ± 2	10 ± 2	0.29
1.0	trans	63	2.6 ± 0.4	34.0 ± 0.9	13 ± 2	0.58
	cis	68	2.5 ± 0.5	30.0 ± 0.8	15 ± 2	0.48
2.0	trans	46	4.3 ± 0.2	50.0 ± 0.5	11.7 ± 0.5	1.18

[a] In cyclohexane at reflux. [b] Results in mol %. [c] Moreover, there is obtained about 1% of cyclohexanol (solvent transfer) and 10% of myrtanic acid (arising from free radical chain process[3b,18]). For this last one radical 7 is not implied. [d] Nopinol oxidation to nopinone was not observed.

Scheme IV



quantity of alcohols 8 (**8a** + **8b**) and **10**, evaluation of the rate constant k_p (cf. Scheme III) has been reached.

Results and Discussion

cis- or *trans*-peroxymyrtanic acids (**6a** or **6b**) have been decomposed in boiling cyclohexane at different concentrations (from 0.1 to 2.0 M). The reaction is over after 30–40 min ($t_{1/2}$ 8–10 min). Products are identified and analysed by VPC. Results are quoted in Table I.

Reactivity of 7. First of all, whatever the configuration of the initial peracid (cis or trans) **6a** or **6b** and whatever their initial concentrations, the ratio **8b**/**8a** is more or less constant (12 ± 3, cf. Table I).

These results show that (i) **8a** or **8b** are not formed in a concerted mechanism (cf. Scheme IV), which would imply a retention of configuration and no lifetime for **7** and (ii) decompositions of **6a** or **6b** pass through the same intermediate, i.e., **7**. This confirms previous results about the peracid decomposition[3] and the mechanism shown in Scheme IV.

Even at low initial concentration in peracid (0.1 M) **8a** and **8b** are observed. The reaction of **7** with peracid is, then, fast enough to enter in competition with its own isomerization into **9**. The quasi-absence of cyclohexanol in the products (less than 1%) indicates that **7** or **9** does not abstract hydrogen from the cyclohexane; the cyclohexyl radical formed would have reacted with peracid to give the cyclohexanol.[3] This confirms former works[3] which had shown that a secondary radical or a tertiary one (except cyclopropyl radicals[5] and the bicyclo[2.2.1]hept-1-yl[3]) reacts more rapidly with peracids than they transfer H from the cyclohexane, used as solvent, even at low initial concentrations.

In agreement with Scheme III, the ratio **8**/**10** increases with the initial peracid concentration. Consequently, following a methodology already described[6] which has been adapted to the present study[7] the ratio k_p/k_i has been

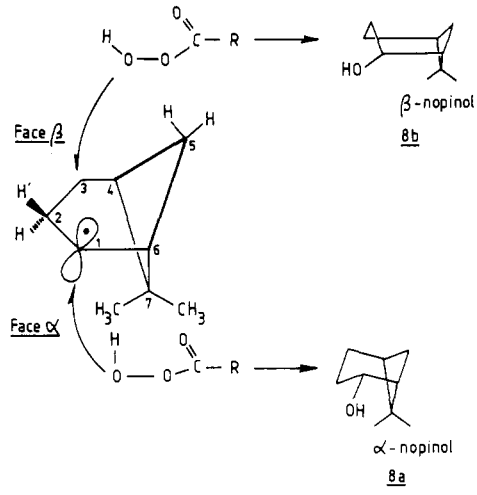


Figure 1.

evaluated, and then $k_p = (1.1 \pm 0.1)k_i$. The rate constant for the opening of 1,2,2-trimethylcyclobutyl was measured by ESR to be equal to 8.7×10^5 s^{-1}.[8] Using that value for k_i, k_p (reaction 4) can be estimated to be around 1×10^6 M^{-1} s^{-1}.

It is interesting to compare this result to those already published for tertiary radicals of type **2**. These were successfully trapped only when they were produced in the presence of methyl mercaptoacetate or thioacetic acid.[2] The energy of activation needed by $^{\bullet}CH_3$ to abstract an H atom from CD_3SH is 4 kcal/mol.[9] The rate constant for H abstraction from thiol by a secondary radical is 1.4×10^6 M^{-1} s^{-1}.[10] When **2** is produced in the presence of CCl_4, only the rearranged product of type **4** (cf. Scheme I) is observed. In such a reaction, the energy of activation needed to transfer a Cl is about 13 kcal/mol,[11] the rate constant being between 5×10^3 and 5.8×10^4 M^{-1} s^{-1} according to the structure of the radical.[12] All these results show that OH transfer from peracid is as fast as H transfer from a thiol; it is the reason why in these conditions **7** has been successfully trapped before isomerization.

Stereochemistry. The stereoselectivity of **8b**/**8a** is independent of the initial peracid, **6a** or **6b**, and of its concentration. That result can be explained by considering the two transition states leading to alcohols **8a** and **8b**. Reaction 3 is very exothermic (more than 40 kcal/mol[3]). Then, according to the Hammond postulate, its transition state should be reactant-like and, more precisely here, free-radical-like. That stereochemical result can be in-

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(7) By applying a classical methodology[6] on Scheme III, the following equation is obtained:

$$R = k_p/(k_i[6]) \qquad \text{where } R = 8/10$$

Formally this equation is correct in the first stage of the reaction. In the present work R is the ratio of alcohols at the end of the reaction. Therefore is is an average value that should be plotted against an average concentration of peracid during the reaction. This average value of **6** was taken as the half of the initial peracid concentration. The slope of this linear relation is 1.1 ± 0.1 with a correlation coefficient of 0.981.

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Table II. Influence of γ-CH_3 on Stereoselectivity

epimer alcohol[a]	X	ratio	$\delta G_X^{\ddagger}$ [b]	$\delta G_H^{\ddagger} - \delta G_{CH_3}^{\ddagger}$ [b]
11a/11b	H	80/20	1.0	1.4
12a/12b	CH_3	37/63	−0.4	
13a/13b	H	71/29	0.6	1.4
14a/14b	CH_3	25/75	−0.8	
15a/15b	H	94/6	1.9	1.3
16a/16b	CH_3	70/30	0.6	
17a/17b	H	50/50[d]	0.0	1.7
8a/8b	CH_3	8/92	−1.7	

[a] See Chart I. [b] $\delta G_X^{\ddagger} = \Delta G_a^{\ddagger} - \Delta G_b^{\ddagger}$. $\delta G_X^{\ddagger}$ is the difference (in kcal/mol at 80 °C) of free enthalpy of activation for the two epimers. [c] $\Delta G_H^{\ddagger} - \delta G_{CH_3}^{\ddagger}$ represents the effect of a methyl group in γ-position. [d] Hypothetical value (cf. text).

terpreted as a balance between torsional effects[13] and steric effects. For example, the ratio of axial alcohol to equatorial alcohol obtained from the *tert*-4-butylcyclohexyl[14a] points out that the torsional effects are the determining ones and hide the steric effects which are mainly due to the fact that very little bonding occurs in the transition rates.

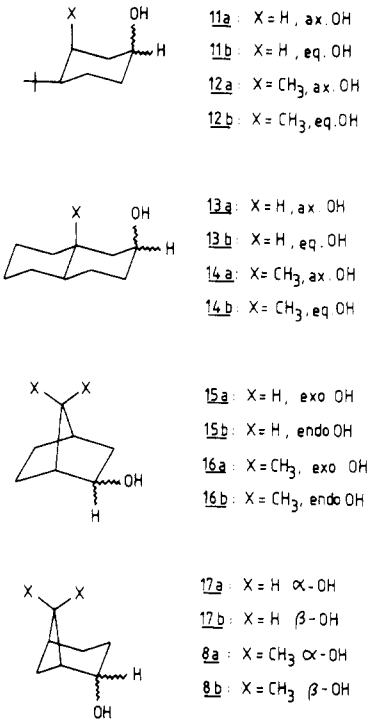
It has been suggested that the norpinane skeleton (6,6-dimethylbicyclo[3.1.1]heptane) must be considered rather as a bridge cyclopentane than as a cyclohexane.[15] From this point of view **7** should be structurally considered as a cyclopentyl radical. For this last one, different authors[16] have proposed a planar structure for the radical center or a pyramidal one that inverts rapidly through the planar structure. These two points of view imply the same consequences for the stereoselectivity. Furthermore, in this type of molecule, the C_2 carbon can just make small displacements up and down out of the plane $C_6C_1C_3C_4$. Then, the conformation can be only a flat boat or a flat chair. Consequently we will first consider that atoms $C_1C_2C_3C_4C_6$ and the H bound to C_1 are coplanar (cf. Figure 1).

In these conditions, torsional effects are more or less equivalent for the two approaches of the hydroxyl peracid group, since, on one hand, the C_2H_2 and C_2H_2' bonds and, on the other hand, the C_6C_5 and C_6C_7 bonds make the same torsional angle with the p orbital of C_2 bearing the single electron. The only difference is the steric hindrance due to the methyl group branched on C_7, which disfavors the α face of **7**. For this reason the β-nopinol (**8b**) (with the OH group in a pseudoequatorial position) is the major product of the ratio **8b/8a**. **8b** adopts a flat boat conformation and **8a** a flat chair one.[15] The ratio **8b/8a** observed here is very different of the ratio at the thermodynamic equilibrium (**8b/8a** = 2.07[17]). Therefore, reaction 4 is under kinetic control, which is in agreement with the exothermicity of the reaction.

In Table II are gathered results concerning the effect of a methyl γ to a radical center on the stereoselectivity of reaction 4. It concerns cyclohexyl-type radicals,[14a] bicyclo[2.2.1]hept-2-yl radicals,[14b] and the norpinyl radical. For the first groups the effect of the methyl on the stereoselectivity can be measured by the numbers quoted in column 3 of Table II.

According to Table II, substitution of X = H by X = CH_3 raise the activation barrier by rougly 1.5 kcal/mol.

Chart I. Epimer Alcohols



If one considers that radical **7** without methyl groups in C_7, has a planar structure for $C_1C_2C_3C_4C_6$, it turns out that the two faces α and β are alike; consequently the enantiomeric ratio **17b/17a** should be 50/50. Then, in Table II, the value 1.7 represents the effect of the methyl in C_7 on the activation barrier. This value agrees with what has been observed previously for the effect of a methyl group γ to the radical center and should mean that radical **7** behaves as all the rest of the series.

Conclusion

In this work, results indicate that the transfer of an OH group from a peracid to the radical **7** is a reaction fast enough, at concentration 2 M in peracid, to result in equimolar concentration of nopinols **8** and the open alcohol **10**. This open alcohol is due to the competing isomerization of **7** by opening of cyclobutane ring.

These results are consistent with the former observations on the velocity of reaction of type 3. Competition between the two reactions 4 and 5 allows an estimation of the rate constant of secondary alkyl radical reaction with the O–O bond of a peracid as 1×10^6 M^{-1} s^{-1}.

The stereochemically controlled ratio of nopinols **8a** and **8b** is the result of the steric effect of the CH_3 group at carbon C_7 in the reaction of the peroxy acid with radical **7** which behaves as planar. This influence of a methyl substituent at the γ-position relative to the radical center is of the same magnitude as that observed with cyclohexyl and bicyclo[2.2.1]cycloheptyl-type radicals.

Experimental Section

Cyclohexane was spectroscopic grade. VPC analyses were run on a GIRDEL-75 apparatus with a recording integrator AutoLab (Spectra-Physics). IR and NMR spectra were (in CCl_4 solution) run respectively on a Perkin-Elmer 577 and on a Varian T60. *trans*-Myrtanol [bp 127 °C (22 mm); phthalate derivative, mp 100–111 °C, pure in VPC] has been kindly given by the company "Dérivés résiniques et terpéniques" in Dax (France) Professor B. Waegell (University of Marseille III, France) and Professeur Lalande (University of Bordeaux, France) gave us, respectively, **8a**, **8b**, and some *cis*-myrtanic acid.

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Preparation of *trans*-Myrtanic Acid. *trans*-Myrtanic acid was obtained by permanganate oxidation of *trans*-myrtanol[19] (**18**) purified by distillation under vacuum [bp 107–110 °C (0.5 mm)]. It was transformed into its methyl ester (by reaction with the diazomethane), and its purity was analyzed by VPC (the methyl *cis*- and *trans*-myrtanoate have different retention times (t_r) in VPC (trans t_r/cis t_r = 1.09 on Silar 5C, 15%, 3.6 min at 150 °C).

Preparation of *cis*- and *trans*-Peroxymyrtanic Acids. *cis*- and *trans*-peroxymyrtanic acids were obtained from the corresponding acids, by a method described in Swern et al.[20] and modified by us.[21] The purity of the peracid (90–95%) was determined by iodometric titration.[22]

Peracid Decomposition. Peracid decomposition, at different concentrations, was done as it has been described previously.[4,21] In a reactor (50 mL) with a condenser was placed the peracid solution (10 mL). The external oil bath temperature was fixed at 100 °C. The reaction is followed by iodiometric measurement. At the end of the reaction the solution was treated by diazomethane in order to transform carboxylic acids into the corresponding methyl esters. The product mixture was analyzed on two types of VPC columns, one polar (Carbowax 20 M, 15%, 2 m, 130 °C) and another one nonpolar (Silar 5C, 15%, 3.6 m, 180 °C). The relative retention times (in min) are as follows: [Carbowax] cyclohexanol, 1; **10** 2.48; **8a**, 2.96; **8b**, 3.47; [Silar 5C] cyclohexanol, 1; **10**, 1.50; **8a**, 1.68; **8b**, 1.92. **8a** and **8b** were identified by comparison of IR and ^{1}H NMR spectra (triplet at τ 4.08 for **8b** and multiplet at τ 4.20 for **8a**[15]) and VPC results with those of authentic samples.

Registry No. **6a**, 103533-22-8; **6b**, 103533-23-9; **7**, 103533-24-0; **8a**, 53767-58-1; **8b**, 51703-63-0; **10**, 90645-55-9; methyl *cis*-myrtanoate, 103617-37-4; methyl *trans*-myrtanoate, 54164-10-2.

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Product Study of Some One-Electron Oxidations of Bibenzyl and 4-Ethylbibenzyl. Evidence against Carbon–Carbon Bond Cleavage of the Bibenzyl Radical Cation in Solution

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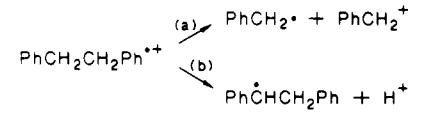
The oxidations of bibenzyl (**1**) and (or) 4-ethylbibenzyl (**2**) have been investigated under a variety of conditions, all of which should involve the intermediacy of bibenzyl radical cations: (a) reaction with ceric ammonium nitrate (CAN) in AcOH or CH_3CN–H_2O; (b) anodic oxidation in AcOH–CH_3CN or $(CH_3)_2CO$–H_2O; (c) photochemical oxidation by CAN in CH_3CN; (d) photochemical autoxidation catalyzed by 9,10-dicyanoanthracene or by CAN in CH_3CN. Nearly exclusive formation of side chain substituted products is observed for the chemical and electrochemical oxidations when the reactions are carried out in AcOH, CH_3CN, or AcOH–CH_3CN, whereas extensive formation of C–C bond cleavage products occurs in the same processes when aqueous solvents are used. In the photochemical reactions, autoxidation produces both cleavage and side chain substituted products, whereas only the latter forms in the CAN-induced reaction in the absence of dioxygen. Evidence based on product analysis suggests that in these reactions no significant C–C bond breaking takes place at the state of bibenzyl radical cation. Cleavage products, where observed, nearly certainly derive from first-formed side chain substitution products.

The fragmentation of bibenzyl radical cation through carbon–carbon bond cleavage (Scheme I, path a) is a very important process in the gas phase.[1] A similar process, of course, is in principle possible in solution as well, where, however, it has to compete with deprotonation (Scheme I, path b), which is favored by the extremely high solvation energy of the proton.

Claims in favor of the occurrence of process a in solution have come from studies concerning the oxidation of bibenzyl by one-electron oxidants, i.e., processes suggested to involve the intermediacy of the bibenzyl radical cation. Thus, the observation that the oxidation of bibenzyl induced by cerium(IV) ammonium nitrate (CAN) in aqueous acetonitrile[2] or photochemically in acetonitrile[3] leads exclusively to fragmentation products has been interpreted as an indication that the bibenzyl radical cation undergoes C–C bond cleavage.

However, formation of cleavage products is by no means a general outcome of the oxidations of bibenzyls involving a radical cation intermediate. Accordingly, recent studies

Scheme I



have shown that oxidation of bibenzyl by cerium(IV) ammonium nitrate in AcOH[4] or by $S_2O_8^{2-}$ in aqueous acetonitrile[5] leads nearly exclusively to side chain substituted derivatives. Moreover no C–C cleavage occurs when methoxy-substituted bibenzyls are reacted with tris(4-bromophenyl)ammoniumyl hexachloroantimoniate,[6] a process which should lead to bibenzyl radical cations.

In this situation further investigation is desirable, which might rationalize these conflicting results and provide information on the factors influencing the competition between path a and path b for reactions in solution. This is true also in view of the fact that bibenzyl is a coal model[7]

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(7) See, for example: Poutsma, M. L.; Douglas, E. C.; Leach, J. E. *J. Am. Chem. Soc.* **1984**, *106*, 1136–1137 and references therein.

0022-3263/86/1951-3587$01.50/0