noketal **44** (892 mg, 3.84 mmol) was allowed to react with the acyl nickel complex from 2.5 mL (19 mmol) of nickel tetracarbonyl and 15.4 mL (15.4 mmol) of propyllithium solution (1.0 M) in ether, in a mixture of ether (60 mL) and THF (25 mL), at −50 °C for 1 h. Dilution with 10 mL of HMPA and addition of 2.8 mL (31 mmol) of allyl iodide, followed by stirring at 23 °C for 14 h, gave conversion to three products (TLC (ether) R_f 0.43, 0.48, 0.30). Isolation as before followed by flash chromatography (40 g of silica, eluting with 2:1 ether–petroleum ether) provided the major component **50** as a yellow solid, yield 1.086 g (82%). Two recrystallizations from ethyl acetate–hexane (1:1) gave colorless prisms, mp 113.5–114 °C, identical with the chromatographed material by NMR and IR spectral analysis. ^{1}H NMR ($CDCl_3$) δ 7.70 (dd, 1 H, J = 7, 2 Hz), 7.13 (dd, 1 H, J = 8.0, 1.5 Hz), 7.40 (t, 1 H, J = 7.0 Hz), 6.0–5.55 (m, 1 H), 5.2–4.9 (m, 2 H), 4.5–4.0 (m, 4 H), 3.88 (s, 3 H), 3.65 (d, 1 H, J = 6.5 Hz), 3.15–2.25 (m, 5 H), 1.7–1.35 (m, 2 H), 0.90 (t, 3 H, J = 7.0 Hz); IR ($CHCl_3$) 3080 (w), 3000 (m), 2970 (s), 2840 (w), 1710 (s), 1690 (s), 1640 (w), 1590 (s), 1480 (s), 1270 (s) cm^{-1}. Anal. C, H.

Preparation of Keto Hydroquinone 51. Diketo ketal **50** was hydrolyzed exactly as described for **46**. Thus a solution of 444 mg (1.29 mmol) of **50** in 12 mL of dioxane and 6 mL of 6 N HCl was stirred at 23 °C for 2 days. Analytical TLC showed two components (2:1 ether–petroleum ether; R_f 0.58 and 0.32), the more polar of which was desired hydroquinone **51**. On a smaller scale, using recrystallized material, the less polar component was not observed. Isolation as before gave a labile, amber oil, yield 376 mg (97%), contaminated with a trace of dioxane but otherwise homogeneous by NMR spectral analysis. ^{1}H NMR ($CDCl_3$) δ 0.99 (t, 3 H, J = 6 Hz, CH_3), 1.48–1.81 (m, 2 H, $CH_2CH_2CH_3$), 2.82 (t, 2 H, J = 6 Hz), 2.33 (dt, 2 H, J = 5, 1 Hz, $CH_2C{=}C$), 3.96 (s, 3 H, OCH_3), 4.95–5.25 (m, 2 H, allyl $C{=}CH_2$), 5.72–6.15 (m, 1 H, allyl), 6.72 (dd, J = 6, 1 Hz, Ar), 7.30 (t, 1 H, J = 7 Hz, Ar), 7.70 (dd, 1 H, J = 6, 1 Hz, Ar); IR (CCl_4) 3400 (m, OH), 2960 (m), 2930 (m), 1690 (m), 1450 (m), 1400 (s), 1260 (s), 1060 (s) cm^{-1}; mass spectral mol wt 300.1365, calcd for $C_{18}H_{20}O_4$ 300.1361.

Preparation of Triol 52. To a solution of ketohydroquinone **51** (376 mg, 1.25 mmol) in THF (10 mL) under argon was added solid sodium borohydride (110 mg, 2.9 mmol) at 23 °C. The mixture was allowed to stir for 15 h, then diluted with 1.0 mL of water, and carefully saturated with potassium carbonate. The mixture was partioned between 10 mL of degassed brine and 30 mL of degassed 2:1 ether/dichloromethane. The aqueous layer was further extracted with one 10-mL portion of 2:1 ether/dichloromethane. The combined organic solutions were washed with degassed brine, dried (Na_2SO_4), filtered, and concentrated in vacuo. The crude triol **52** was obtained as an amber oil, 369 mg, 98%. ^{1}H NMR ($CDCl_3$) δ 9.5 (s, 2 H), 7.7 (br d, 1 H, J = 8.0 Hz), 7.25 (br t, 1 H, J = 8.0 Hz), 6.73 (br d, 1 H, J = 8.0 Hz), 6.2–5.7 (br m, 1 H), 5.3–4.8 (m, 3 H), 4.03 (s, 3 H), 4.2–3.8 (m, 2 H), 3.54 (br d, 1 H, J = 7.0 Hz), 2.30–0.8 (m, 7 H); IR (neat) 3350 (br, s), 3080 (w), 2970 (s), 2870 (m), 1630 (w), 1610 (m), 1580 (m), 1450 (s), 1380 (vs), 1250 (vs).

Preparation of Hydroxy Quinone 53. The crude triol **52** (312 mg, 1.03 mmol) was stirred in a mixture of DDQ (224 mg, 0.99 mmol) and potassium bicarbonate (117 mg, 1.17 mmol) in 10 mL of methyl alcohol under argon at 0 °C for 1.0 h. Dilution with dichloromethane and filtration through alumina gave crude naphthoquinone **53** as an orange oil, 242 mg. Flash chromatography on silica gel (15 g; elution with 4:1 ether–petroleum ether) gave pure **53** as an orange oil, 134 mg, 45% yield overall from **50**. ^{1}H NMR ($CDCl_3$) δ 7.8–7.5 (m, 2 H), 7.25 (dd, 1 H, J = 8.0, 2.5 Hz), 6.1–5.6 (m, 1 H), 5.3–5.0 (m, 2 H), 4.9–4.6 (m, 1 H), 4.00 (s, 3 H), 3.72 (d, 1 H, J = 11 Hz), 3.38 (br d, 2 H, J = 3.5 Hz), 2.2–1.3 (m, 4 H), 0.95 (t, 3 H, J = 6.0 Hz); IR (neat) 3500 (br, s), 3080 (w), 2960 (s), 2880 (m), 2840 (m), 1650 (d, vs), 1620 (s), 1590 (vs), 1470 (vs), 1450 (vs), 1280 (vs) cm^{-1}; mass spectrum, m/e 300 (M^+, 58%), 282 (30), 271 (28), 257 (100), 239 (33), 229 (19), 214 (12).

Preparation of Pyrano Ester 54a,b. The hydroxynaphthoquinone **53** (174 mg, 0.58 mmol) was stirred with a mixture of bis(acetonitrile)palladium dichloride (14 mg, 0.058 mmol), anhydrous cupric chloride (171 mg, 1.27 mmol), and methyl alcohol (3 mL) under carbon monoxide (1.0 atm) for 3.3 h and gave essentially one component detected by analytical TLC (ether on silica gel, R_f 0.35). The mixture was concentrated at oil pump pressure, triturated with benzene (5 mL), and purified by flash chromatography on silica gel (5 g). Elution with 7:2 ether–petroleum ether gave as the main component an orange viscous oil, 144 mg (70%). Analysis by HPLC (Poracil column, 8:1 hexane–ethyl acetate, 5 mL/min; R_v trans 8.85 mL, R_v cis 10.3 mL) indicated a mixture of isomers in the ratio 75:25 later identified as the trans/cis mixture **54a,b**. The chromatographed material was crystallized from 3 mL of warm 3:1 hexane–ethyl acetate to provide pure **54a** as dark orange needles, mp 128–133 °C. Recrystallization gave the analytical sample, mp 134–136 °C. ^{1}H NMR ($CDCl_3$) δ 7.7–7.4 (m, 2 H), 7.13 (dd, 1 H, J = 7.5, 2.5 Hz), 4.80 (br t, 1 H, J = 5.0 Hz), 4.4–4.1 (m, 1 H), 3.97 (s, 3 H), 3.70 (s, 3 H), 2.85–2.58 (m, 3 H), 2.22 (ddd, 1 H, J = 17.0, 10.0, 1.0 Hz), 1.9–1.2 (m, 4 H), 0.95 (t, 3 H, J = 6.5 Hz); IR ($CHCl_3$) 3030 (m), 2960 (m), 2880 (m), 2840 (m), 1735 (s), 1660 (vs), 1590 (s), 1470 (m), 1440 (s), 1280 (vs) cm^{-1}. Anal. C, H. The cis isomer **54b** was previously characterized.[5a]

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Structural Effects in Solvolytic Reactions. 51. Examination of the Differences in the Behavior of Secondary and Tertiary U-Shaped Bicyclic Derivatives toward Solvolysis

Herbert C. Brown,* Irvin Rothberg,[1a] and J. Chandrasekharan[1b]

Richard B. Wetherill Laboratory, Purdue University, West Lafayette, Indiana 47907

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The solvolyses (acetolysis) of secondary bicyclic tosylates such as *cis*-bicyclo[3.3.0]oct-2-yl and 5,6-*endo*-trimethylene-8-norbornyl and -9-norbornyl proceed with low exo/endo rate ratios (<10), unlike secondary 2-norbornyl (280) and the analogous tertiary derivatives wherein the exo/endo rate ratios increase progressively with increasing U-shaped character. The possible factors responsible for this difference in behavior are discussed. One possible factor can be nucleophilic solvent participation in the secondary systems, absent both in secondary 2-norbornyl and in tertiary solvolyses. An examination of the products of acetolysis of a number of bicyclic secondary tosylates reveals predominant inversion during acetolysis, suggesting solvent participation.

Winstein and Trifan[2] proposed that the large exo/endo rate ratio observed in the acetolysis of 2-norbornyl tosylate (280) arises from nonclassical stabilization of the exo transition state by σ-participation of the C_1–C_6 electron

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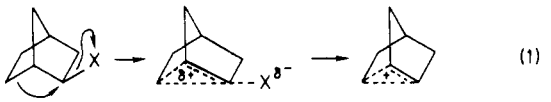
cloud (eq 1).



(1)

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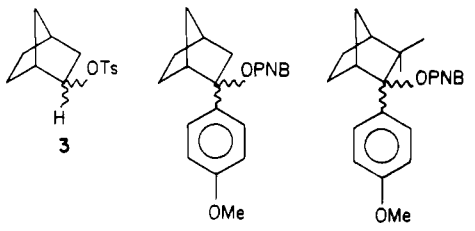
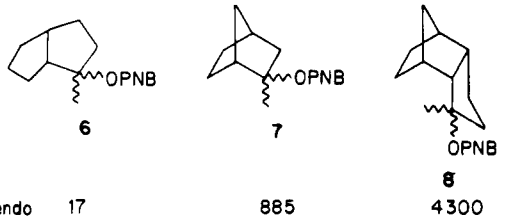
To gain a real understanding of the factors governing the solvolysis of 2-norbornyl derivatives, we began a systematic study of the effects of structure on the rates of solvolysis of 2-norbornyl derivatives. A major attempt in that direction was a study of the solvolysis of tertiary 2-norbornyl derivatives.[3] The exo/endo rate ratios for tertiary norbornyl derivatives were clearly comparable to those of secondary 2-norbornyls **3–5**.



	3	4	5
exo/endo	280	284	44 000

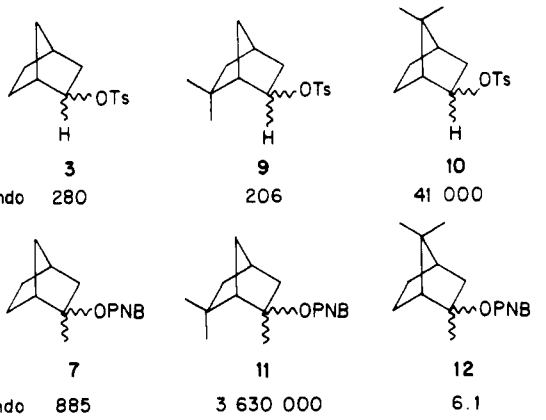
Interestingly, even those tertiary derivatives that would lead to highly stabilized cationic intermediates exhibited large exo/endo rate ratios. In fact, elaborate studies revealed that tertiary 2-norbornyl cations are classical.[4] Since this means that large exo/endo rate ratios do not require nonclassical participation per se, we considered steric retardation of ionization of the endo isomer as an alternative explanation for these large exo/endo rate ratios.[5] The basic philosophy behind this proposal was that in U-shaped systems, such as *endo*-norbornyl, there is an increase in strain as the locus of the departing group moves into the endo cavity. To verify this proposal, we systematically studied the effect of U-shape character on the rates of solvolysis of representative tertiary U-shaped bicyclic systems and found that the exo/endo rate ratio increases with increasing U-shape character (**6–8**). More



	6	7	8
exo/endo	17	885	4300

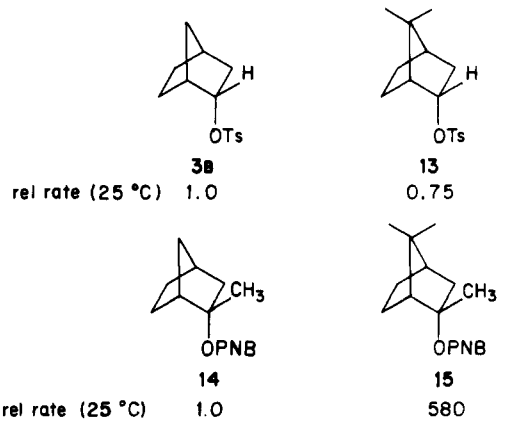
importantly, we studied a host of other reactions and found that the relative exo/endo face selectivity increases with increasing U-shape character, thus demonstrating that there is nothing unusual about the solvolysis and that it is part of a general steric phenomenon.

However, it has been pointed out that there are significant differences between secondary and tertiary derivatives and, consequently, the conclusions reached with tertiary bicyclic systems need not apply to the secondary systems. For example, Schleyer has pointed out that the secondary and tertiary 2-norbornyl substrates do not respond similarly to increase in steric strain[6] (**3**, **9**, **10**, **7**, **11**, **12**).



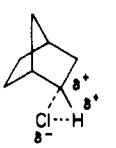
	3	9	10	7	11	12
exo/endo	280	206	41 000	885	3 630 000	6.1

The tertiary esters are much more sensitive to steric effects, as illustrated by the comparisons in **3B** and **13–15**.



	3B	13	14	15
rel rate (25 °C)	1.0	0.75	1.0	580

On the basis of these, Schleyer concludes:[6] "Steric hindrance to ionization is a plausible explanation for the behavior of tertiary 2-norbornyl systems. The locus of departure of an endo leaving group can be restricted by a 2-*exo*-methyl or -phenyl substituent. I doubt if a much smaller hydrogen can function similarly. Another possible difference should be considered. During ionization, part of the developing charge will be transferred to the α-hydrogen which should then *attract* rather than *repel* the leaving group (**16**) in a kind of hydrogen-bonding interaction. Tertiary systems could not enjoy this effect to the same extent."



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Consequently, in this paper we plan to compare the behavior of secondary and tertiary U-shaped bicyclic systems toward solvolysis.

Results and Discussion

On the basis of our studies of the tertiary U-shaped bicyclic systems,[5] we compiled the rate data for the acetolysis of *cis*-bicyclo[3.3.0]oct-2-yl,[7] 2-norbornyl, 5,6-

(1) (a) Postdoctoral research associate, 1963–1965. (b) Postdoctoral research associate on a grant from the National Science Foundation, 1981–1984.

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(6) See Schleyer's comments in: Brown, H. C. (with comments by Schleyer, P. v. R.) "The Nonclassical Ion Problem"; Plenum Press: New York, 1977; pp 146–149.

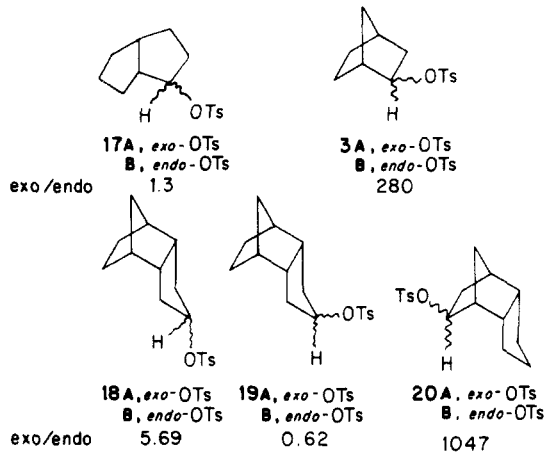
(7) Closson, W. D.; Kwiatkoski, J. H. *Tetrahedron Lett.* **1966**, *52*, 6430.

Table I. Rates of Acetolysis of Representative U-Shaped Secondary Bicyclic Tosylates

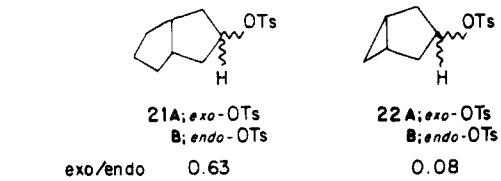
tosylate	temp, °C	$10^6 k_1$, s^{-1}	exo/endo rate ratio
cyclopentyl[a]	75	541 (1.58)[b]	
cis-bicyclo[3.3.0]oct-2-yl[c] (exo)	75	116	1.32
cis-bicyclo[3.3.0]oct-2-yl[c] (endo)	75	87.3	
cis-bicyclo[3.2.0]hept-3-yl[a] (exo)	75	95	0.63
cis-bicyclo[3.2.0]hept-3-yl[a] (endo)	75	150	
cis-bicyclo[3.1.0]hex-3-yl[a] (exo)	75	51	0.08
cis-bicyclo[3.1.0]hex-3-yl[a] (endo)	75	690	
2-norbornyl (exo)[d]	25	23.3	280
2-norbornyl (endo)[d]	25	0.0828	
endo-5,6-trimethylene-2-norbornyl[e,f] (exo)	25	~9[g]	1047
endo-5,6-trimethylene-2-norbornyl[e,f] (endo)	25	0.00860	
endo-5,6-trimethylene-8-norbornyl[f,h] (exo)	25	0.0888	5.69
endo-5,6-trimethylene-8-norbornyl[f,i] (endo)	25	0.0156	
endo-5,6-trimethylene-9-norbornyl[f,j] (exo)	25	0.448	0.623
endo-5,6-trimethylene-9-norbornyl[f,k] (endo)	25	0.719	

[a] From ref 9. [b] Value in parentheses for 25 °C; taken from ref 19. [c] From ref 7. [d] From ref 2. [e] From ref 8. [f] Rate constants were determined at higher temperatures and extrapolated for 25 °C. [g] Estimated indirectly by NMR; on solvolysis the compound isomerizes to 5,6-*exo*-trimethylene derivative. [h] $k_{50} = 0.357 \times 10^{-5}$ s^{-1}; $k_{75} = 8.44 \times 10^{-5}$ s^{-1}; $\Delta H^{\ddagger} = 27.7$ kcal mol^{-1}; $\Delta S^{\ddagger} = 2.0$ eu. [i] Taken from ref 17. [j] $k_{50} = 1.34 \times 10^{-5}$ s^{-1}; $k_{75} = 33.9 \times 10^{-5}$ s^{-1}; $\Delta H^{\ddagger} = 25.4$ kcal mol^{-1}; $\Delta S^{\ddagger} = 2.4$ eu. [k] $k_{50} = 1.98 \times 10^{-5}$ s^{-1}; $k_{75} = 33.9 \times 10^{-5}$ s^{-1}.

endo-trimethylene-2-norbornyl, -8-norbornyl, and -9-norbornyl tosylates[8] (**3**, **17**, **18**, **19**, **20**), and related systems



such as bicyclo[3.2.0]hept-3-yl[9] and bicyclo[3.1.0]hexyl[9] (**21** and **22**) (Table I). The results reveal interesting differences in the behavior of the secondary and tertiary derivatives except in the case of 2-norbornyl (compare **6** vs. **17**, **3** vs. **7**, and **8** vs. **18**). The exo/endo rate ratios are large for both secondary and tertiary 2-norbornyl derivatives. On the other hand, in the case of *cis*-bicyclo[3.3.0]octyl and 5,6-*endo*-trimethylene-8-norbornyl and -9-norbornyl systems, the exo/endo rate ratios are dramatically lower for the secondary derivatives. Moreover, they do not show any effect of the U-shaped character of these systems. It is of interest to consider the possible factors responsible for the differences in the behavior of the tertiary vs. secondary U-shaped systems on one hand and of 2-norbornyl vs. the other secondary bicyclic systems on the other.

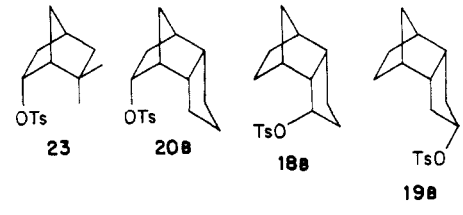


I. Steric Factors. Applicability of the Foote–Schleyer Correlation to the Solvolysis of Secondary U-Shaped Bicyclic Tosylates. The low exo/endo rate ratio in the secondary tosylates **17**, **18**, and **19** might mean that steric retardation of ionization is not important in these systems. If we can calculate the solvolysis rate constant without accommodating the steric retardation of ionization and if the calculated and observed rate constants agree, obviously steric retardation of ionization could not be a major factor. On the other hand, if there is a disagreement, steric retardation of ionization could be an important factor. The Foote–Schleyer correlation[10] was proposed as a means of making such a calculation (eq 2).

$$\log k_{rel} = \frac{1715 - \nu_{CO}}{8} + 1.32k(1 + \cos 3\phi_2) + \text{inductive term} + \frac{GS_{strain} - TS_{strain}}{1.36} \quad (2)$$

Since the major point in the proposal of steric retardation of ionization is that strain increases as the reaction proceeds, i.e., $TS_{strain} \neq 0$, it was decided to test the generality of the assumption that GS_{strain} involving the leaving group can be assumed to vanish in the transition state.

Accordingly, in collaboration with Schleyer,[11] we undertook to test the ability of the correlation to predict the rates of representative endo U-shaped bicyclic derivatives **18B**, **19B**, **20B**, and **23**. Discrepancies of 8000 for **23**,



10 000 for **20B**, and 100 000 for **18B** between the calculated and observed rates were realized. On the other hand, in less hindered systems such as **19B**, there is an agreement between the calculated and observed values.[11] This suggested to us that steric factors are important in some of these secondary systems and also that the Foote–Schleyer correlation, as commonly applied, is not equipped to handle situations wherein $TS \neq 0$.

II. Role of Solvent Participation. Nucleophilic solvent participation plays a critical role in the acetolysis of unhindered secondary tosylates.[12] On the other hand,

(8) Data for 5,6-*endo*-trimethylene-2-norbornyl tosylates taken from: Takeuchi, K.; Oshika, T.; Koga, Y. *Bull. Chem. Soc. Jpn.* **1965**, *38*, 1318.
(9) Meinwald, J.; Anderson, P.; Tufariello, J. J. *J. Am. Chem. Soc.* **1966**, *88*, 1301.
(10) (a) Foote, C. S. *J. Am. Chem. Soc.* **1964**, *86*, 1853. (b) Schleyer, P. v. R. *Ibid.* **1964**, 1854, 1856.
(11) Brown, H. C.; Rothberg, I.; Schleyer, P. v. R.; Donaldson, M. M.; Harper, J. J. *Proc. Natl. Acad. Sci. U.S.A.* **1966**, *56*, 1653.

Table II. Products in the Acetolysis of Representative Secondary Bicyclic Tosylates

tosylate	products, % exo-OAc	endo-OAc	eliminatn product	misc
cis-bicyclo[3.3.0]oct-2-yl[a,b] (exo)	24.6	14.7	52.0	*cis*-bicyclo[3.3.0]-oct-1-yl OAc (0.9); bicyclo[3.2.1]oct-8-yl OAc (7.9)
cis-bicyclo[3.3.0]oct-2-yl[a,b] (endo)	51.3	0	47.0	*cis*-bicyclo[3.3.0]oct-1-yl OAc (1.7)
cis-bicyclo[3.2.0]hept-3-yl[c,d] (exo)	6.0	94.0		
cis-bicyclo[3.2.0]hept-3-yl[c,f] (endo)	93.0	7.0		
bicyclo[3.1.0]hex-3-yl[c,d] (exo)	0	100.0		
bicyclo[3.1.0]hex-3-yl[c,d] (endo)	...	98.0		
5,6-*endo*-trimethylene-8-norbornyl[e,f] (exo)	2.4	0.1	92.6	5,6-*endo*-trimethylene-5-norbornyl OAc (1.2); 5,6-*endo*-trimethylene-9-norbornyl OAc (exo) (0.6); its endo isomer (1.0)
5,6-*endo*-trimethylene-8-norbornyl[e,f] (endo)	40	<5	48.6	5,6-*endo*-trimethylene-2-norbornyl (9.0)
5,6-*endo*-trimethylene-9-norbornyl[e,f] (exo)	3.5	13.9	80.2	
5,6-*endo*-trimethylene-9-norbornyl[e,f] (endo)	48.3	0.2	45.2	

[a] From ref 7. [b] At 95 °C. [c] From ref 9. [d] At 75 °C. [e] The acetate product mixture was reduced with LAH, and the individual components were identified as the alcohols. [f] The solvolysis was done at 75 °C in the presence of 100% excess sodium acetate.

solvolysis of tertiary substrates proceed without significant solvent participation[13] (i.e., by a k_c process). It might be possible that the solvolysis of the secondary substrates **17–19** may be quite different mechanistically from that of the analogous tertiary derivatives in that the former proceeds by a k_s and the latter by a k_c process. If that were so, it is possible to explain not only the difference between the tertiary and secondary derivatives but also that between the 2-norbornyl and the other secondary derivatives, since the solvolysis of both *exo*- and *endo*-2-norbornyl tosylates does not involve nucleophilic solvent participation.[14,15] To strengthen this proposal, we examined the products of acetolysis of **17–19** and related derivatives (Table II). It can be seen that there is predominant inversion during the acetolysis of both the exo and endo isomers which is a good indication for solvent participation. In fact, solvent participation in the solvolysis of cyclopentyl tosylates is very large, except in limiting solvents such as trifluoroacetic acid and hexafluoroisopropyl alcohol. Unfortunately, the solvolyses of the secondary systems **17–19** were not examined in these limiting solvents. It is probable that such a study might resolve this question.

However, we thought that instead of attempting such indirect studies which often lead to nebulous conclusions, it is better to undertake a direct search for the long postulated nonclassical resonance energy in the transition states for the solvolyses or in the free 2-norbornyl cation. Definitive evidence for the presence or absence of such nonclassical resonance energy should provide a definitive answer to the question. That topic is the subject of the next, concluding paper of this series.

Experimental Section

Materials. The preparation of 5,6-*endo*-trimethylene-2-norbornanone and -9-norbornanone has been described earlier.[16] The lithium aluminum hydride (LAH) reduction of 5,6-*endo*-trimethylene-8-norbornanone in refluxing ethyl ether overnight afforded a mixture of the endo and exo alcohols, which, upon careful recrystallization from ether–pentane mixture, provided 99% (GC) pure 5,6-*endo*-trimethylene-8-*endo*-norbornanol in 80% yield (mp 159–160 °C). 5,6-*endo*-Trimethylene-8-*exo*-norbornanol was prepared as described in the literature.[17] LAH reduction of 5,6-*endo*-trimethylene-9-norbornanone in refluxing ether overnight afforded quantitatively a mixture containing 98% of 5,6-*endo*-trimethylene-9-*endo*-norbornanol. Recrystallization from pentane gave 99.3% pure (GC) 5,6-*endo*-trimethylene-9-*endo*-norbornanol (mp 55–56 °C). The corresponding exo alcohol was prepared by the reaction of the endo tosylate with tetrabutylammonium acetate, followed by the hydrolysis of the exo OAc by a procedure described in the literature.[18] The alcohols were converted to the corresponding tosylates by reaction with *p*-toluenesulfonyl chloride in the presence of pyridine. The melting points of the tosylates are as follows: **18A**, 40–41 °C [lit.[17] oil]; **18B**, 139–140 °C [lit.[17] 138.2–138.4 °C]; **19A**, 45–46 °C; **19B**, 85–86 °C. All of the compounds gave satisfactory C, H analyses.

Kinetics. The kinetics of acetolysis was followed by titrating the *p*-toluenesulfonic acid formed with an appropriate standard base.

Product Studies. The following general procedure was adopted. A solution (0.05 M) of the tosylate in acetic acid containing 0.10 M sodium acetate was kept at 75 °C in a sealed tube for 10 half-lives. After the solution was cooled, a 10.0-mL aliquot was added to 15 mL of ethyl ether containing a known amount of naphthalene as a GC standard, followed by neutralization with sodium hydroxide while cooling (20.0 mL of 30% solution). The aqueous phase was extracted with ether and combined with the ether layer. The ether solution, after being dried over anhydrous magnesium sulfate, was reduced with 0.5 g of LAH in 15 mL of ether to remove the acetate groups. After being worked up, the ether layer was examined by GC using a Perkin-Elmer 226 instrument equipped with a Carbowax 20M capillary column (150 ft × 0.01 in.). The data are reported in Table II.

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