Table I. Kinetic Data for the Rearrangement of *N*-Nitroso(2-methylamino)acetonitrile in Basic Methanol

t, °C	k, L mol^{-1} s^{-1}
1.0 ± 0.02	$(5.96 \pm 0.07) \times 10^{-3}$
11.0 ± 0.02	$(1.41 \pm 0.19) \times 10^{-2}$
23.0 ± 0.02	$(3.32 \pm 0.09) \times 10^{-2}$

$\Delta H^{\ddagger} = 12.0 \pm 0.34$ kcal mol^{-1} $\Delta S^{\ddagger} = -24.7$ eu

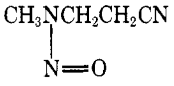
with those of α-isonitroso-*N*-methylaminoacetonitrile reported earlier.[2] A polarogram obtained after addition of the isolated crystals to the solution that yielded curve 6 (Figure 1) showed an increase in peak height without any shift in peak potential, strongly suggesting that the isolated product is the species giving rise to the peak at −1.74 V.

The kinetics of the reaction in eq 1 were determined by measuring the rate of decay of the peak current at −1.52 V, i.e., at the peak potential of species I. Kinetic studies were performed at 1.0, 11.0, and 23 °C.

The reaction was found to be second order (first order with respect to both I and base). The second-order rate constants and activation parameters were calculated in the usual fashion[6] and are given in Table I. The most significant value in the table is the large negative entropy of activation. A pathway that agrees with the kinetic observations is shown in Scheme I; a similar mechanism involving a three-membered ring was first suggested by Daeniker.[2]

The transformation of I to II is facilitated by the relatively high acidity of the methylene protons. The anion that forms is stabilized by the adjacent *N*-nitroso and nitrile groups. To check this conclusion, the next higher homologue of I, *N*-nitroso(3-methylamino)propionitrile (III), was examined polarographically under identical experimental conditions. Compound III gave single differential pulse polarographic peaks in *both* neutral and basic methanol solutions that remained unchanged over a period of 12 h.

The strong acidity of the methylene protons in I is further supported by NMR studies. At room temperature in D_2O, I incorporates two deuteriums instantaneously to yield I-D_2. No such incorporation occurs with III.



$$\underset{\underset{\displaystyle N{=}O}{|}}{CH_3N}CH_2CH_2CN$$

III

Experimental Section

α-Isonitroso-*N*-methylaminoacetonitrile (II) from *N*-Nitroso(2-methylamino)acetonitrile (I). Dry methanol (20 mL) was placed in a round-bottom flask fitted with a drying tube and a side arm for the passage of N_2 gas. Dry N_2 was bubbled through the solution for 10 min. A freshly cut piece of sodium metal (0.2 g) was then added and, after it had reacted, I (1.0 g) was added. The solution was allowed to remain at room temperature for 5 h in a nitrogen atmosphere and then evaporated to dryness under vacuum. The residue was dissolved in water (30.0 mL) and adjusted to pH 6.5 with hydrochloric acid. The solution was evaporated to dryness under vacuum and the residue was extracted with chloroform (300 mL). The chloroform solution was evaporated to dryness under vacuum and the residue was sublimed under vacuum yielding white needle-shaped crystals (0.5 g, 50%; mp 154–156 °C (lit.[2] mp 155–157 °C)): single spot on TLC; NMR (D_2O), $-CH_3$, δ 2.90 (s, 3 H); IR 2.93 (OH), 3.12 (NH), 4.42 (C≡N), 6.01 μm (C=N); UV$_{\max}$ in absolute ethanol 2550 Å. Anal. Calcd: C, 36.36; H, 5.05. Found: C, 36.39; H, 5.10.

Kinetics of I → II.[6] Methanol solutions of constant ionic strength containing tetrapentylammonium iodide as supporting electrolyte and base were prepared. A 10^{-2} M solution of I in methanol was used as stock solution. The cell was thermostated at 1, 11, and 23 °C using Forma Scientific Model 2095 refrigerated and heated bath and circulator. The decrease in peak current was monitored by recording successive polarograms at fixed time intervals. All the observations were made twice to check the reproducibility of the results. In all cases a plot of ln I_p vs. t was linear and independent of concentration of I, indicating the transformation to be first order with respect to I. The pseudo-first-order rate constants were calculated from the slope of ln I_p vs. t. Variation of the pseudo-first-order rate constants with concentration of base was linear at all temperatures, yielding a first-order reaction rate with respect to base. Second-order rate constants were calculated and are given in Table I. The activation parameters were calculated in the usual fashion.[6]

Spectra and Differential Pulse Polarography. NMR, UV, and IR spectra and differential pulse polarograms were obtained as previously described.[3]

Acknowledgments. This investigation was supported by DHEW Research Grants CA-18618 and 12227 from the National Cancer Institute and by the Samuel S. Fels Fund.

Registry No.—I, 3684-97-7; II, 66922-13-2; III, 60153-49-3.

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Stopped-Flow Study of Salt Effects on the Hydroxide and Borate Ion Catalyzed Hydrolysis of Covalent *p*-Tolylsulfonylmethyl Perchlorate in Aqueous Borax Buffer Solutions

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Received May 2, 1978

As part of a detailed study of environmental[1,2] and micellar effects[3] on the general base-catalyzed hydrolysis of covalent[4] arylsulfonylmethyl perchlorates, we have recently reported kinetic salt effects on the water-catalyzed process.[2] This pH-independent hydrolysis involves rate-determining proton transfer from the reactant to water via a dipolar transition state in which the negative charge at the α-sulfonyl carbon atom is highly dispersed.[1,5] The electrolyte effects were rationalized mainly by invoking the importance of electrostatic ion–water interactions in the joint cybotactic regions of the ions and the reactant and/or transition state. This theory emerged as a result of the observation that the salt effects were predominantly governed by the charge type and density of the distinct ions.[2] Electrolyte-induced changes in water structure were assigned a secondary role. As an extension of these studies, we now report the effects of some neutral electrolytes on the rates of hydroxide and borate ion catalyzed hydrolysis in aqueous borax buffer solutions of constant ionic strength (I = 1.0 M). The main objective of this work was to see

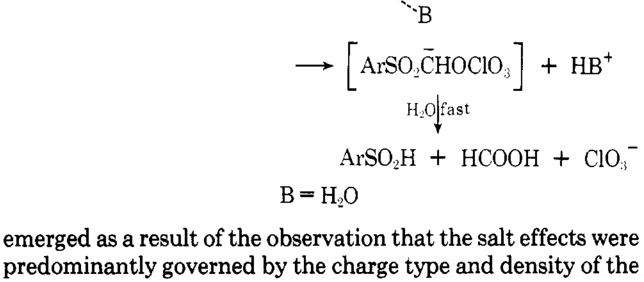

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Table I. Second-Order Rate Constants for the Hydroxide and Borate Ion Catalyzed Hydrolysis 1 and 1-d_2 in Aqueous Borax Buffer Solutions at 25 ± 0.05 °C

compd	conditions	pH (±0.02)	$k_{OH} \times 10^4$, $M^{-1} s^{-1}$	k_{borate}, $M^{-1} s^{-1}$	k^H_{OH}/k^D_{OH}	$k^H_{borate}/k^D_{borate}$
1	A[a]	9.20	9.21	13.0		
1-d_2	A[a]	9.20	1.51	1.9	6.1	6.8
1	B[b]	8.88	10.1	10.1		
1-d_2	B[b]	8.88	1.94	1.4	5.2	7.2

[a] 1×10^{-1}, 2×10^{-2}, 2.5×10^{-2}, and 4×10^{-2} M borax solutions. [b] As under *a*, but now at constant ionic strength (I = 1.0 M $NaClO_4$).

Table II. Kinetic Salt Effects on the Hydroxide and Borate Ion Catalyzed Hydrolysis of 1-d_2 at 25 ± 0.05 °C

electrolyte	molarity	$k_{obsd} \times 10^2$, $M^{-1} s^{-1}$, for $c_{borate} \times 10^2$ M[a]				$k_{OH} \times 10^{-4}$, $M^{-1} s^{-1}$	k_{borate}, $M^{-1} s^{-1}$
		1	2	2.5	4		
$NaClO_4$[b]	0.5	17.6	20.4	21.8	25.8	1.94	1.4
NaBr[a]	0.5	18.4	22.6	23.8	30.2	1.94	2.0
NaCl[b]	0.5	19.5	23.1	27.1	35.4[f]	1.98	2.1
CsBr[c]	0.5	28.6	34.5	35.7	49.0	2.00	3.4
Me_4NBr[d]	0.5	35.4	43.6	44.3	57.2	2.08	3.6
n-Bu_4NBr[e]	0.5	48.8	68.7	75.7	104	1.90	9.0

[a] Borax concentration. NaCl added until I = 0.5 M. [b] pH 8.88. [c] pH 9.01. [d] pH 9.14. [e] pH 9.22. [f] $c_{borax} = 5 \times 10^{-2}$ M.

whether the simple theory advanced for the neutral hydrolysis[2] could also provide a framework for understanding salt effects in these buffer systems.

Results and Discussion

It has been demonstrated that at low stoichiometric tetraborate concentrations (as employed in the present study) and at pH >2 dissociation into boric and (mono)borate ion is essentially complete:[6,7]

$$B_4O_7^{2-} + 7H_2O \leftrightarrows 2B(OH)_3 + 2B(OH)_4^-$$

Thus, pseudo-first-order rate constants (k_{obsd}) for hydrolysis of 1 and its dideuterated analogue (p-$CH_3C_6H_4SO_2CD_2OClO_3$ (1-d_2)) in borax buffers of pH ca. 9.0 will be composed of contributions due to hydroxide ion, borate ion, and water catalysis (eq 1) but the anion-induced deprotonation will dominate the "water" reaction[1,2] by many orders of magnitude (k_{obsd} (H_2O) = 6.05×10^{-4} s^{-1}, k_{borate} ca. 10 $M^{-1} s^{-1}$, k_{OH} ca. 10^5 $M^{-1} s^{-1}$ at 25 °C).

$$k_{obsd} = k_{OH}c_{OH} + k_{borate}c_{borate} + k_{H_2O}c_{H_2O} \qquad (1)$$

Second-order rate constants k_{borate} and k_{OH} for hydrolysis of 1 and 1-d_2 in borax buffers both at constant and at differing ionic strength (I) were obtained by the stopped-flow technique and are listed in Table I. The large primary kinetic deuterium isotope effects clearly substantiate rate-determining deprotonation of the substrates by the general base and definitely rule out a salt-induced mechanistic change toward an S_N2 type process. In view of the fast reaction of 1 with hydroxide ion, salt effects were largely determined for hydrolysis of 1-d_2. Results are displayed graphically in Figure 1. The k_{obsd} values pertain to buffer solutions of constant ionic strength 1 M in which the concentration of the neutral electrolyte under study (c_{salt}) was varied between 0 and 0.5 M at constant $I_{borate} + I_{NaCl}$ = 0.5 M (see Experimental Section). For the $NaClO_4$, NaBr, and CsBr solutions the pH (8.88) was constant within experimental error and identical to that in the absence of the salt. However, for the tetraalkylammonium salts there was a slight increase in pH (pH 8.8–9.2) with increasing c_{salt} which will be partly responsible for the rate acceleration observed for these electrolytes. Nevertheless, the *order* of the kinetic salt effect (n-Bu_4NBr > Me_4NBr > CsBr > NaBr > $NaClO_4$) is similar to that observed for the water-catalyzed reaction.[2] In order to separate salt effects operating on k_{OH} and k_{borate}, k_{obsd} values have been measured at constant c_{salt} = 0.5 M in the presence of varying concentrations of borax and NaCl (for these two salts the sum of their ionic strengths was maintained at 0.5 M leading to a total ionic strength of 1.0 M). Kinetic data are summarized in Table II and plotted in Figure 2. First of all, we note that in all moderately concentrated salt solutions there exists a linear dependence of rate on the borax concentration.[8] Second, the data show that there are only substantial kinetic salt effects on k_{borate} whereas k_{OH} is hardly affected by the nature of the electrolyte. The salt effects on k_{borate} follow the sequence $NaClO_4$ < NaBr < NaCl < CsBr ~ Me_4NBr < n-Bu_4 NBr. It appears that borate anion catalysis is accelerated by increasing charge density on the anion and by decreasing charge density on the cation. Therefore it is likely that the salt effects are not just determined by the availability of water molecules for hydration of the reactants and the transition state.[9] If structure 2 represents a likely model[10] for the transition state for proton transfer from covalent arylsulfonylmethyl perchlorates to the borate anion, it is reasonable to suppose that the hydrogen bond donor capabilities of the O–H_a and O–H_b bonds in 2 are enhanced

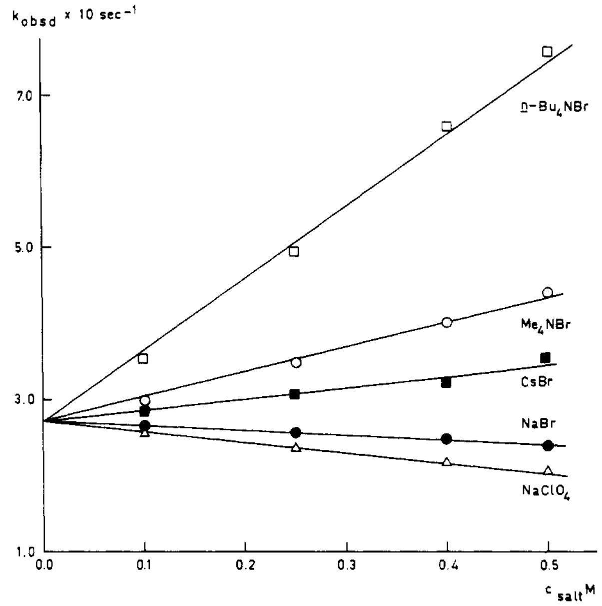


Figure 1. Plots of k_{obsd} vs. concentration of salt for hydrolysis of 1-d_2 in aqueous borax buffers of total ionic strength 1 M (see text).

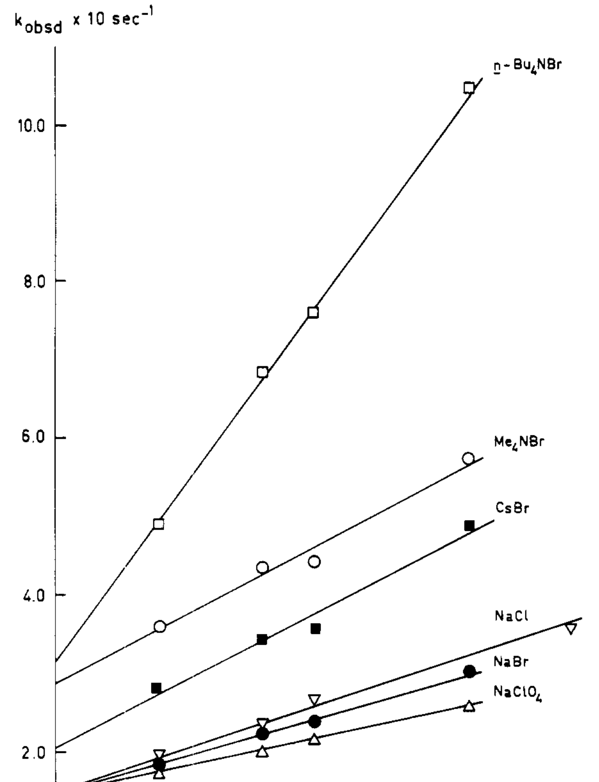
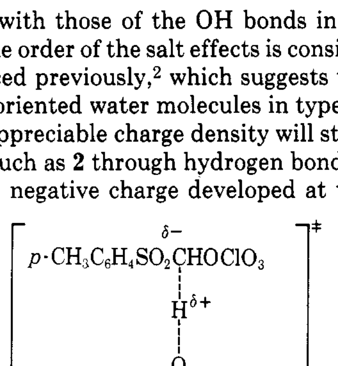


Figure 2. Plots of k_{obsd} vs. borax concentration at total ionic strength 1 M for hydrolysis of 1-d_2 in the presence of 0.5 M $NaClO_4$, NaBr, NaCl, CsBr, Me_4NBr, and n-Bu_4NBr (see text).

as compared with those of the OH bonds in the $B(OH)_4^-$ anion. Now the order of the salt effects is consistent with the model advanced previously,[2] which suggests that polarized and strongly oriented water molecules in type I cospheres[11] of anions of appreciable charge density will stabilize[12] transition states such as **2** through hydrogen bonding to H_a and H_b. Since the negative charge developed at the α-sulfonyl carbon atom in **2** is strongly dispersed,[1] there will be no or only little transition state stabilization by interaction with water molecules of enhanced hydrogen bonding donor capability in the hydration sheaths surrounding cations of high charge density. This rationale then leads to the prediction, which is in agreement with experiment, that the greatest positive salt effects will be observed for electrolytes composed of cations of low charge density and hydration enthalpy ($Na^+ < Cs^+ < Me_4N^+ < n$-$Bu_4N^+$) and anions of high charge density and hydration enthalpy ($ClO_4^- < Br^- < Cl^-$). In conclusion, we note that the present results reveal a striking similarity between the order of kinetic salt effects on a molecule–ion reaction (i.e., 1-d_2 with borate ion) and on a molecule–molecule reaction (i.e., 1 with water). This is not a priori anticipated in terms of simple electrostatic theories for salt effects in aqueous media.[13] The present data and those obtained earlier[2] consistently suggest that kinetic salt effects on the hydrolysis of the arylsulfonylmethyl perchlorates at moderate or even higher electrolyte concentration predominantly reveal effects due to extensive hydration sphere overlap.[14,15] These effects appear to be determined by the strength of directional ion–water interactions and by the magnitude of charge separation and delocalization which accompanies the transfer of the reactants into the transition state.[15]



2

Experimental Section

Materials. p-Tolylsulfonylmethyl perchlorate (1) and its deuterated analogue (1-d_2) were prepared as described previously, taking into account the appropriate safety precautions.[2,5] The salts used in all experiments were of the highest quality available (usually from Merck or Fluka) and were used as such with the exception of n-Bu_4NBr, which was recrystallized twice from ethyl acetate and dried at 45 °C in vacuo over P_2O_5 for 20 h. The water was demineralized and distilled twice in an all all-quartz distillation unit.

Buffer solutions of low borax concentration (≤0.05 M) were of constant ionic strength, $I = 1.0$ M, and were made up by weight. These solutions were prepared as follows. Up to $I = 0.5$ M, NaCl was employed as the electrolyte. The final ionic strength was adjusted by adding calculated amounts of NaCl and the electrolyte under study. Thus, the salt effects reported in this paper pertain to variation between 0 and 0.5 M salt at a total ionic strength of 1.0 M.

Kinetic Measurements. The kinetic measurements were carried out using an Aminco-Morrow stopped-flow apparatus, connected to a data acquisition storage and retrieval system (DASAR). The two reagent solutions were injected in equal quantities. Temperature control was within ±0.1 °C. The change in absorbance at 230 nm was recorded on a W & W recorder (type 3012) to allow the calculation of the pseudo-first-order rate constants (k_{obsd}). These k_{obsd} values were reproducible to within 2%. The estimated accuracy of k_{OH} and k_{borate} listed in Table I is ±6 and ±10%, respectively. The pH measurements were carried out by means of a Findip pH meter, type 555A, using a glass and calomel electrode at 25 °C. The pH values are accurate to within ±0.02.

Acknowledgment. We thank the Netherlands Foundation for Chemical Research (SON) for support in the purchase of the stopped-flow apparatus.

Registry No.—1, 14894-56,5; 1-d_2, 66922-37-0.

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(8) This linearity with buffer concentration in salt solutions of high ionic strength is by no means a general rule: E. S. Hand and W. P. Jencks, *J. Am. Chem. Soc.*, **97**, 6221 (1975).

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(10) In view of the rather high kinetic acidity of **1** and **1**-d_2, it is not a priori excluded that one or more intervening water molecules are present between the organic reactant and the anionic base in the transition state. This will not affect our rationalization for the order of the kinetic salt effects.

(11) For a detailed discussion of ion hydration, see: H. L. Friedman and C. V. Krishnan in "Water, Comprehensive Treatise", Vol. 3, F. Franks, Ed., Plenum Press, New York, N.Y., 1972, p 1.

(12) It is tentatively suggested that the insensitiviness of k_{OH} to electrolyte effect reflects the more favorable free energy of hydration of OH^- as compared with $B(OH)_4^-$ making it less susceptible to interaction with hydration spheres of other ions present in solution and to changes in charge distribution as a result of these interactions.

(13) E. S. Amis and J. F. Hinton, "Solvent Effects on Chemical Phenomena", Vol. 1, Academic Press, New York, N.Y., 1959, Chapter 5.

(14) In another type of approach M. C. R. Symons (*J. Chem. Res.*, in press) has recently rationalized the electrolyte effects on the water-induced deprotonation in terms of an equilibrium between completely hydrogen bonded

water molecules and water molecules containing "free" OH groups and "free" lone pairs. However, this theory cannot easily be applied to the anion induced process reported here.

(15) Kinetic salt effects have been earlier ascribed to water polarization as a result of specific interactions with cations or anions: (a) A. R. Olson and L. K. J. Tong, *J. Am. Chem. Soc.*, **66**, 1555 (1944); (b) D. B. Dennison, G. A. Gettys, D. G. Kubler, and D. Shepard, *J. Org. Chem.*, **41**, 2344 (1976).

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Regiospecific α-Tropolone Synthesis. A Selective Preparation of the Isomeric Thujaplicins

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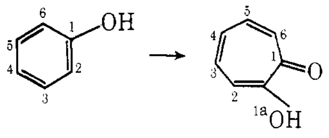
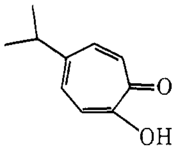
Received February 14, 1978

The seven-membered, aromatic α-tropolone ring **2** occurs naturally in three biosynthetically distinct classes:[1,2] in the essential oils of *Cupressae* (e.g., α-thujaplicin), in mold metabolites of the *Penicillium* family (e.g., stipitatonic acid) and in the *Colchicum* alkaloids (e.g., colchicine). The unique character of these seven-membered carbocycles has attracted considerable synthetic, biogenetic, and theoretical attention[3] since the structure elucidation of the first natural α-tropolone, stipitatonic acid by Dewar, in 1945. However, general synthetic entry into the α-tropolone system has been limited for the most part to the exhaustive oxidation of α-ketocycloheptanones[3] or the $[{}_{\pi}2_{s} + {}_{\pi}2_{a}]$ cycloaddition of dihaloketenes with cyclopentadienes followed by rearrangement.[3,4]

As a general approach to natural α-tropolone systems, we desired synthetic access to the α-tropolone ring via site-specific single-carbon expansion of the corresponding suitably substituted phenol (e.g., **1** → **2**). This approach allows the utilization of well-defined phenolic chemistry in establishing the requisite substitution pattern or functionality on the ultimately generated α-tropolone ring and minimizes subsequent chemical manipulation in the presence of the α-tropolone system. We wish to report the realization of this general synthetic objective as illustrated by regiospecific syntheses of the isomeric thujaplicins γ-**3** and β-**4**.



1 **2**



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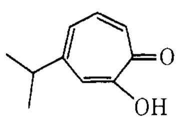

4

Our synthetic scheme called for the regiospecific establishment of a dihydroaromatic silyl ether. The recent development of lithium/ammonia reduction of O-silylated phenols affords excellent regiopredictability and facile synthetic entry into such systems.[7] For the synthesis of γ-thujaplicin **3** (Scheme I), dissolving metal reduction of triethylsilyl (4-isopropylphenyl) ether **5** afforded the dihydroaromatic silyl ether **6**. Subsequent sodium trichloroacetate mediated dichlorocyclopropanation and methanolic aqueous hydrochloric acid hydrolysis[9,10] afforded the stable bicyclic dichlorocyclopropanol **7**. The regiospecificity of dichlorocyclopropanation is well established to proceed via attack on the most nucleophilic olefin in cases not overshadowed by steric considerations. The stability of such unsaturated bicyclic α,α-dichlorocyclopropanols appears to be unique and **7** is thus a representative of a novel class of functionalized cyclopropane.[11,12] *syn*-Hydroxyl directed peracid epoxidation afforded the epoxide **8** which

Table I. Physical Data for Isolated Intermediates in γ-Thujaplicin Synthesis

	registry no.	bp, °C/mm or Mp, °C	IR, cm^{-1}	NMR (CDCl$_3$/Me$_4$Si), δ	MS (m/e) rel abundance, %
5	66967-06-4	155–165/0.1	1580 (w) 1250 (s) 740 (s)	0.48–1.05 (brm, 15 H) 1.13 (d, J = 6.5 Hz, 6 H) 2.75 (q, J = 6.5 Hz, 1 H) 7.75 (d, J = 10 Hz, 2 H) 8.05 (d, J = 10 Hz, 2 H)	250 (81) 235 (73) 195 (95) 121 (100)
6	66967-07-5	Kugelrohr 150/0.1	1610 (med) 1250 (stg)	1.05–0.50 (m, 21 H) 2.30 (b, qt, J = 7.0 Hz, 1 H) 2.65 (s, 2 H) 4.85 (s, 1 H) 5.35 (s, 2 H)	
7[a]	66967-08-6	white spindles 71.0–73.5	3425 (stg) 850 (stg)	0.97 (d, J = 7.0 Hz, 6 H) 1.82 (d, d, J = 7.5, 1.5 Hz, 1 H) 1.90–2.50 (m, 3 H) 2.64 (d, d, J = 6.5, 4.5 Hz, 2 H) 2.95 (brs, OH, 1 H) 5.30 (brs, 1 H)	220 (16) 185 (89) 167 (33) 97 (100
8[a,17]	66967-09-7	white blocks 75–77	3420 (stg) 1040 (stg) 830 (stg)	0.94 (d, d, J = 6.5 Hz, 6 H) 1.50 (qt, J = 6.5 Hz, 1 H) 1.62 (d, d, J = 9.0, 2.0 Hz, 1 H) 1.83 (d, d, J = 16.0, 2.0 Hz, 1 H) 2.31 (d, d, J = 16.0, 9.0 Hz, 1 H) 2.5 (d, J = 2.0 Hz, 2 H) 2.79 (t, J = 2.0 Hz, 1 H)	236 (4.2) 201 (65) 193 (96) 175 (88) 157 (100)
9[a]	66967-10-0	light oil	1622 (stg) 1599 (stg) 940 (stg)	1.20 (d, J = 8.0 Hz, 6 H) 2.80 (qt, J = 8.0 Hz, 1 H) 6.74 (d, J = 10.5 Hz, 1 H) 7.12 (s, 2 H) 7.65 (d, J = 10.5 Hz, 1 H)	182 (79) 139 (100) 109 (32) 103 (59)

[a] Satisfactory elemental analysis was obtained (C, H ±0.3%).

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