Experimental Section

Rose Bengal (DyeTel) was converted to its benzyl ester as previously described.[3] It was purified by flash chromatography on a silica gel column with dimethoxyethane (DME) as the eluent. Solvents were spectroscopic grade (Aldrich). Absorption spectra were obtained on a Varian Cary 219 instrument.

Preparation of 1,10-Decanediylbis(Rose Bengal) (II). Rose Bengal, 4.07 g (4 mmol), and 1,10-dibromodecane, 0.3 g (1 mmol) were added to 100 mL of DMF and heated for 48 h at 70 °C. The DMF is removed in vacuo and the solid red product washed several times with water and several times with ether. The product after drying is passed through a flash column and eluted with a mixture of methylene chloride/methanol (8:1). There are four distinguishable colored fractions, the first fraction being the monoester and the third fraction the dimer. The dimer ($\delta_{CH_2O(O)CR}$ = 4.11 ppm) is distinguished from the monosubstituted ester (δ_{CH_2Br} = 3.38 ppm) by proton NMR.

Preparation of Polystyrene Containing Rose Bengal as the End Groups (III). In 100 mL of dry, freshly distilled THF, recrystallized naphthalene, 2.56 g (20 mmol), was dissolved under argon. To this solution was added sodium, 0.46 g (20 mmol), in small pieces. The mixture was stirred magnetically until all of the sodium dissolved, giving a deep green solution. This solution was filtered through sintered glass and stirred under argon as the stock initiator whose concentration was determined by titration with a standard acid. For polymerization an appropriate amount of the initiator was transferred into a 100-mL, three-necked flash fitted with a gas inlet and let out system and a septum through which extra THF was injected to make the solution volume about 50 mL. The filled flask was then cooled in an isopropyl alcohol–dry ice bath to about −78 °C and 6 mL of dry, distilled inhibitor-free styrene was injected. The solution turned red immediately and the polymerization was continued for 1 h. The living polymer chains (indicated because the color remained deep red) were then quenched with a large excess of dry, distilled 1,2-bromoethane until the solution became colorless. The polymer was subsequently precipitated with methanol and purified by repeated precipitation from benzene solution with methanol. The purified polymer was dried in vacuo at 45 °C.

Rose Bengal was attached to the polymer chains as follows: the polystyrene prepared above (2 g) was dissolved in dry DMF (50 mL) and Rose Bengal, 4 g (0.004 mol), added. The solution was magnetically stirred and heated for 24 h under nitrogen in the dark at 70 °C. The polymer was precipitated from the cooled solution with methanol and the Rose Bengal red polymer recrystallized by continual precipitation with methanol from a benzene solution.

Acknowledgment. We are grateful to Shu-Ren Wu for measuring the molecular weight, M_w, to David Luttrell for the synthesis of $RB(CH_2)_{10}RB$, and to the National Science Foundation (CHE B503551) for support of this work. Helpful discussions with Jerzy Paczkowski and Shwn-Mei Linden are also acknowledged.

Absolute Rate Constants for the Reaction of Triethylsilyl Radicals with Ring-Substituted Benzyl Chlorides[1]

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We have recently measured the absolute rate constants, k_1, for a large number of halogen atom abstraction reactions by the triethylsilyl radical.[3] Our results led us to propose that charge transfer in the transition state, as indicated in 1, was extensive for abstraction from alkyl

$$Et_3Si^\bullet + XR \rightarrow \underset{1}{[Et_3Si^+ \,{}^\bullet X \,:\bar{R}]} \rightarrow Et_3SiX + R^\bullet \quad (1)$$

iodides and bromides and from carbon tetrachloride. Although charge transfer appeared to be relatively unimportant in chlorine atom abstractions from simple alkyl chlorides,[3] it certainly would be expected to manifest itself if rate constants were measured for the reactions of the $Et_3Si^\bullet$ radical with a series of ring-substituted benzyl chlorides. The direction of charge separation shown above implies that electron-withdrawing substituents would increase the reaction rate. That is, a Hammett plot of log (k_2/M^{-1} s^{-1}) vs. substituent constant would be expected to have a positive slope, i.e., a positive ρ value.[4]

Table I. Kinetic Data for Chlorine Atom Abstraction from Substituted Benzyl Chlorides by the Triethylsilyl Radical

substituent(s)	$\Sigma\sigma^a$	$10^{-7}k_2$ (M^{-1} s^{-1})^b	$k_2^{subst}/k_2^{H\,c}$
3,5-$(CF_3)_2$	0.92	11.5 ± 1.6	7.54
3,5-Cl_2	0.72	9.3 ± 0.8	7.35
4-CN	0.70	8.3 ± 0.5	
3,4-Cl_2	0.61	4.8 ± 0.3	
3-CF_3	0.46	4.5 ± 0.1	3.57
3-F	0.34	4.2 ± 0.2	
3-Cl	0.37	3.7 ± 0.2	3.61
4-F	0.15	3.3 ± 0.2	
3-CH_3	−0.06	3.3 ± 0.2	
3-OCH_3	0.10	3.1 ± 0.2	
4-$C(CH_3)_3$	−0.15	2.4 ± 0.2	
4-CH_3	−0.14	2.2 ± 0.2	
none	0	1.9 ± 0.6^d	(1.0)

^{a} From ref 18. ^{b} Absolute rate constants measured at 24 ± 2 °C in EtSiH:$Me_3COOCMe_3$ solvent by using benzil as probe. Errors correspond to 95% confidence limits but include only random error. ^{c} Relative rate constants measured at 40 °C in Et_3SiH solvent. ^{d} Calculated at 24 °C from the Arrhenius parameters given in the supplementary material of ref 3.

$$Et_3Si^\bullet + XC_6H_4CH_2Cl \rightarrow Et_3SiCl + XC_6H_4CH_2^\bullet \quad (2)$$

A search of the literature uncovered only one, very limited, study of the relative reactivities of ring-substituted benzyl chlorides toward a silicon-centered radical.[5,6] The radical in question was $Me_3Si\dot{S}iMe_2$ and its relative reactivity at 60 °C toward benzyl chloride and the 4-CH_3, 3-CH_3, and 3-CF_3 substituted benzyl chlorides was measured[5] and apparently[13] gave a ρ value of +0.29. We therefore decided to measure absolute rate constants for

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(5) Sakurai, H.; Yamagata, M.; Murakami, M. *Abstracts, 23rd Annual Meeting of the Chemical Society of Japan, Tokyo* **1971**, *III*, 1260.

(6) There have been related studies on germanium-centered[7] and tin-centered[8-12] radicals.

(7) Sakurai, H.; Mochida, K. *J. Organomet. Chem.* **1972**, *42*, 339–343.

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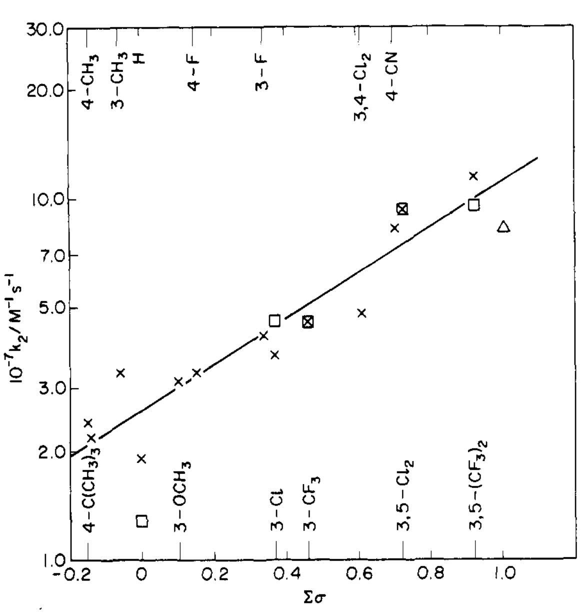

Figure 1. Hammett plot of k_2 vs. $\sum\sigma$ substituent constants. Absolute rate constants, X; relative rate constants normalized at $3\text{-}CF_3C_6H_4CH_2Cl$, □. The Δ corresponds to k_2 for $4\text{-}NCC_6H_4CH_2Cl$ plotted at the σ^- value for the 4-CN group.

reaction of $Et_3Si^\bullet$ with a number of meta-[14] and para-substituted benzyl chlorides at room temperature using our previously developed[3,15,16] laser flash photolytic technique. The general reliability of these k_2 values has been confirmed by the measurement of the relative reactivities at 40 °C of a few selected benzyl chlorides by competitive kinetics.

Absolute rate constants were measured at 24 ± 2 °C using equal volumes of Et_3SiH and di-*tert*-butyl peroxide as the solvent and benzil as the probe, as previously described.[3,15,16] These results are summarized in Table I. Relative rate constants were obtained by GC analyses following the thermally initiated radical reaction between triethylsilane (solvent) and equal volumes of two benzyl chlorides at 40 °C. These relative constants, $k_2^{\text{subst}}/k_2^{\text{H}}$ calculated from the loss of starting materials by using the method of Ingold and Shaw,[17] are also given in Table I.

A plot of log ($k_2/M^{-1}\ s^{-1}$) against the normal Hammett σ substituent constants[18] is shown in Figure 1. A reasonably good linear relation is obtained. As was expected, it has a positive slope: $\rho = +0.64$ (std dev = ±0.07, corr coeff 0.939). To our knowledge this is one of the largest positive ρ values to be reported for a free-radical reaction. It should be noted that while the LFP technique measures global rate constants that would include ring addition, this contribution is expected to be small. For example, for C_6H_5Cl, $k = 6.9 \times 10^5\ M^{-1}\ s^{-1}$,[3] significantly smaller than the rate constants in Table I.

The five compounds for which relative rates were measured are also shown in Figure 1. The relative rate constants have been normalized at the point for $3\text{-}CF_3C_6H_4CH_2Cl$. This compound was chosen because the $3\text{-}CF_3$ group has a σ value which puts it in the middle of the range of σ values for these five compounds (see Table I). That is, it has been assumed that $k_2^{3\text{-}CF_3}/k_2^{\text{H}} = 3.57$ corresponds to $k_2^{3\text{-}CF_3} = 4.5 \times 10^7\ M^{-1}\ s^{-1}$. The agreement between the relative and absolute rate data, ignoring the difference in temperatures, is quite satisfactory.[19]

The most extensive study of halogen abstraction from substituted benzyl halides by a group IV (4)[25] centered radical is that of Blackburn and Tanner.[11] These workers used competitive methods to measure relative rate constants at 90 °C in benzene for the reaction

$$n\text{-}Bu_3Sn^\bullet + XC_6H_4CH_2Y \rightarrow n\text{-}Bu_3SnY + XC_6H_4CH_2^\bullet \quad (3)$$

with Y = Cl, Br, and I. Positive ρ values were obtained in all cases. It was correctly claimed that Hammett plots of log ($k_3^{\text{subst}}/k_3^{\text{H}}$) gave quite good correlations with σ^- substituent constants[20] while only fair correlations were obtained with σ substituents constants.[21] However, in their study the range in σ_m values was rather limited[22] and it is the meta substituents that must be used to define ρ.[20] We have used double meta substitution to obtain $\Sigma\sigma_m$ values as high as 0.92 in order to define ρ as well as is possible. The substituents used by Blackburn and Tanner to distinguish between correlations with σ and with σ^- were 4-CN ($\sigma = 0.63$,[23] $\sigma^- = 1.00$) and $4\text{-}CO_2Et$ ($\sigma = 0.52$, $\sigma^- = 0.68$). We note that if we were to plot log ($k_2^{4\text{-CN}}/M^{-1}\ s^{-1}$) at its σ^-_p value of 1.0 (i.e., the Δ in Figure 1) we would obtain $\rho^- = +0.58$ (std dev = ±0.07, corr coeff 0.935). That is, the standard deviations and correlation coefficients are essentially identical for the correlation with σ^- and with σ. Our ρ value of +0.64 is substantially larger than Blackburn and Tanner's ρ of +0.40 for reaction of $n\text{-}Bu_3Sn^\bullet$ with the benzyl chlorides which would seem to indicate that there is a greater degree of charge separation in the transition state for reaction 2 than for reaction 3 (Y = Cl).[24] A notably better correlation with σ^- than with σ would, therefore, have been expected for reaction 2.

Experimental Section

General. All compounds used in this study were commercially available or could be readily prepared by standard procedures. The GC analyses were carried out on a Varian Vista 6000 chromatograph using a 50 m, 0.2 mm i.d. cross-linked methyl-silicone column. The laser flash photolysis (LFP) system and procedures were identical with those employed in earlier work.[3,15,16] The $Et_3Si^\bullet$ radicals were generated by LFP (337 nm) of $Et_3SiH{:}Me_3COOCMe_3$ (1.0:1.0, v/v). Benzil was used as a "probe" for $Et_3Si^\bullet$, the benzil/$Et_3Si^\bullet$ adduct being monitored at its absorption maximum (380 nm). Rate constants, k_2, were determined in the usual way by

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[14] We have concentrated more on meta substituents than para in order to avoid the potential radical stabilizing effects (via electron delocalization) of the latter.

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[19] A plot of log ($k_2^{\text{subst}}/k_2^{\text{H}}$) vs. σ based on the relative rate data yields $\rho = +0.96$ (std dev = ±0.15, corr coeff 0.96).

[20] The σ^- substituent constants apply only to para substituents and express enhanced mesomeric interaction when the reaction center is directly conjugated with the substituent, σ^-_p being defined only for electron acceptors.[18] For donors and for all meta substituents normal σ values apply. Thus, it is the meta substituents that serve to determine ρ.[18]

[21] From ref 11. For Y = Cl: $\rho = 0.40$, $r = 0.92$ for σ; $\rho = 0.34$, $r = 0.97$ for σ^-. For Y = Br: $\rho = 0.22$, $r = 0.95$ for σ; $\rho = 0.17$, $r = 0.98$ for σ^-. For Y = I: $\rho = 1.05$, $r = 0.94$ for σ; $\rho = 0.81$, $r = 0.99$ for σ^-.

[22] From ref 11. For Y = Cl: σ_m up to 3-CN ($\sigma_m = 0.68$); for Y = Br: σ_m up to 3-Br ($\sigma_m = 0.39$); for Y = I: σ_m up to 3-Cl ($\sigma_m = 0.37$).

[23] The value used in ref 11.

[24] Temperature differences would not be expected to account for such large differences in ρ. Note also that our competitive experiments at 40 °C[19] gave a somewhat higher ρ value than the absolute rate measurements at ca. 24 °C.

[25] In this paper the periodic group notation in parentheses is in accord with recent actions by IUPAC and ACS nomenclature committees. A and B notation is eliminated because of wide confusion. Groups IA and IIA become groups 1 and 2. The d-transition elements comprise groups 3 through 12, and the p-block elements comprise groups 13 through 18. (Note that the former Roman number designation is preserved in the last digit of the new numbering: e.g., III → 3 and 13.)

monitoring the growth of the 380-nm absorption at various concentrations of added $XC_6H_4CH_2Cl$.

Relative Rate Constants. A mixture containing 30 μL (0.261 M) of $C_6H_5CH_2Cl$, 30 μL (0.237 M) of 3-$ClC_6H_4CH_2Cl$, and 0.019 g (0.109 M) of $Me_3CONNOCMe_3$ in 1.0 mL of Et_3SiH in a Pyrex tube was degassed, sealed under vacuum, and heated at 40 °C for 72 h (ca. 5 half-lives of the hyponitrite). The tube was opened, a known quantity of tetradecane was added as an internal standard, and the tube's contents were then analyzed by GC. Relative rate constants were calculated[17] from the quantities of the two reactants that were consumed. Similar competition experiments were carried out with 3-$ClC_6H_4CH_2Cl$ and 3-$CF_3C_6H_4CH_2Cl$, with 3-$ClC_6H_4CH_2Cl$ and 3,5-$(CF_3)_2C_6H_3CH_2Cl$, and with 3-$CF_3C_6H_4CH_2Cl$ and 3,5-$Cl_2C_6H_4CH_2Cl$.

Synthesis of the Enantiomers of *endo*-Brevicomin

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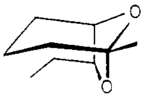
endo-Brevicomin (**1**) is a component of the volatiles of several economically important bark beetles in the genera *Dendroctonus* and *Dryocetes*.[1] In several species it is a minor component with undefined biological activity[1a,b] accompanying *exo*-brevicomin (**2**), which is often an aggregation pheromone.[2] Recently **1** has been reported to be an aggregation pheromone of *Dryocetes antographus*, a damaging pest of Norway spruce.[1c] Furthermore, (−)-**1** has been reported to be an antiaggregation pheromone for the southern pine beetle *Dendroctonus frontalis*, while (+)-**1** acts as to increase attractiveness of other known aggregation pheromones and kairomones (frontalin, *trans*-verbenol, and α-pinene) for this insect.[1e]



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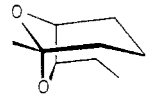
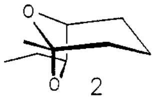


(−) 1



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Because of the significant damage to pine caused by the southern pine beetle[3] in the southwestern U.S. and the potential for development of integrated pest management programs based on semiochemical technology,[4] we sought a synthesis of (+)- and (−)-**1** that would be ammenable to the production of the (gram) quantities required for field evaluation of alternative strategies. Although many syntheses of (±)-**1** have been reported,[5] only four syntheses of (+)- and (−)-**1** are available. One synthesis involves threo-selective addition of Grignard reagents to chiral β-alkoxy aldehydes. This synthesis yields mixtures of **1** and **2** that must be separated by preparative gas chromatography.[6]

Two recent syntheses of (+)- and (−)-**1** utilized Sharpless kinetic resolution of allylic alcohols. One involves a six-step (29% overall, 78.5% ee) route commencing with kinetic enantio- (90:10) and threo- (93:3) selective epoxidation of divinylcarbinol[7] while the second route proceeded in five steps (7% overall, 96–97% ee) from (±)-1-penten-3-ol. The higher chiral purity in the second route was achieved through crystallization of a key intermediate.[1c]

Most recently[8] the enantiomers of **1** have been prepared in high (>99% ee) enantiomeric purity and good chemical yield (60% over three steps) by a route used earlier[5i] that involves stereoselective cyclization of the ketal formed by reaction of 4-(phenylsulfonyl)-2-butanone and *erythro*-1-bromopentane-2,3-diol. The latter intermediate was available in enantiomerically pure form from degradation of α-D-(+)-glucose over L-(+)-arabinose by multistep procedures.

The present route to (+)- and (−)-**1** is based on our earlier synthesis (+)- and (−)-**2**[9] and does not involve any chromatographic separation. It involves seven steps and proceeds in an overall yield of 20% (Scheme I).

The synthesis commenced with protection of 5-bromo-2-pentanone[5a] (**3**) with 2,2-dimethyl-1,3-propanediol to give **4**. The bromo ketal was chain-extended by reaction with the dianion of propargyl alcohol.[9] Lithium metal reduction to the desired *E* allylic alcohol, **5**, was conducted without

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(1) *endo*-Brevicomin. (a) Inactive component of frass of western pine beetle, *Dendroctonus brevicomis*. Silverstein, R. M.; Brownlee, R. G.; Bellas, T. E.; Wood, D. L.; Browne, L. E. *Science (Washington, D.C.)* **1968**, *159*, 889–891. (b) Minor component of volatiles of mountain pine beetle, *Dendroctonus ponderosae*, and western balsam bark beetle, *Dryocetes confusus*. Schurig, V.; Weber, R.; Nicholson, G. J.; Oehlschlager, A. C.; Pierce, H. D., Jr.; Pierce, A. M.; Borden, J. H.; Ryker, L. C. *Naturwissenshaften* **1983**, *70*, 92–93. (c) Aggregation pheromone of *Dryocetes antographus*. Mori, K.; Sew, Y.-B. *Tetrahedron* **1985**, *41*, 3429–3431. (d) (±)-Antiaggregation pheromone for southern pine beetle, *Dendroctonus frontalis*. Vité, J. P.; Renwick, J. A. A. *Naturwissenschaften* **1971**, *7*, 578–582. (e) (−)-Antiaggregation pheromone and (+) aggregation pheromone for *D. frontalis*. Vité, J. P.; Billings, R. F.; Ware, C. W.; Mori, K. *Naturwissenschaften* **1985**, *72*, 99–100.

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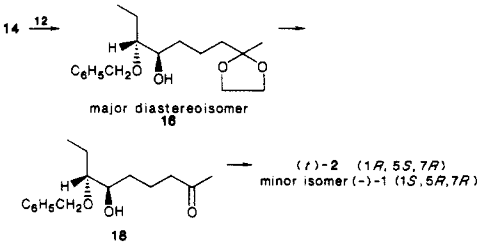
(6) Bernardi, R.; Fugani, C.; and Grasselli, P. *Tetrahedron Lett.* **1981**, *22*, 4021–4024. This paper erroneously illustrates erythro selectivity in addition of $\mathrm{ClMgCH_2CH_2(\overline{COCH_2CH_2O})CH_3}$ (**12**) to an enantiomer of α-(benzyloxy)butyraldehyde while reporting the reaction is threo-selective. Structures **16** and **18** in this paper should be



Furthermore, as pointed out by a referee the recent statement made in the article by Yusufolgu et al. "According to our results, in the former synthesis (Bernardi, et al.) the sign of the optical rotation disagrees with the configuration of 5 (*endo*-brevicomins)". This statement is not correct. Bernardi et al. have numbered the usual C_1 as C_7 and the usual C_7 as C_1. That is the cause of confusion.

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