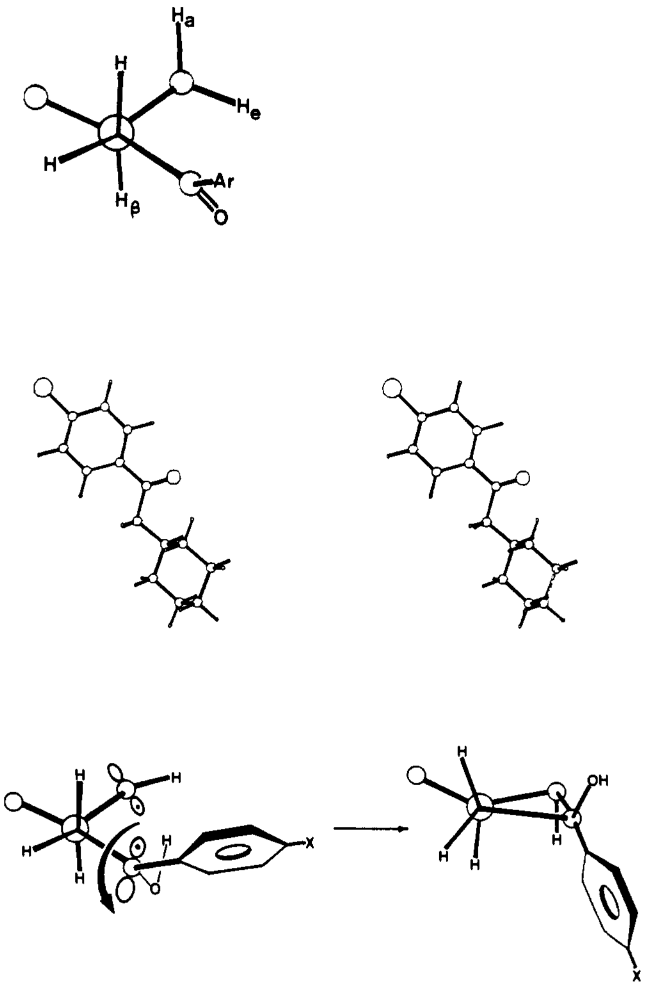

Figure 1. Newman projection down equatorial carbon–carbon bond of ketones **1a–c** (top), stereodiagram of ketone **1b** (middle), and motions involved in formation of *trans*-cyclobutanols **3a–c** (bottom).

ring. A Newman projection down the equatorial carbon–carbon bond is shown in Figure 1. Also shown in Figure 1 is a stereoview of ketone **1b**. This conformation clearly indicates that it is the equatorial hydrogen H_e that is abstracted in the solid state through a boatlike six-atom geometry. This conclusion is borne out quantitatively by the abstraction distances and angles in Table II. The carbonyl oxygen is much closer to H_e (2.6 Å) than to H_a (3.8 Å). This hydrogen abstraction distance of 2.6 Å is consistent with our suggested[8] upper limit of 2.72 Å (van der Waals radii sum) for this type of process.

Turning to the angle τ, the degree to which the hydrogen being abstracted lies outside the mean plane of the carbonyl group, Wagner has clearly pointed out that coplanar ($\tau = 0°$) hydrogen abstraction is not a strict requirement for the type II process.[1a] He has suggested a $\cos^2 \tau$ dependence for abstraction, which, in our case, would reduce the relative reactivity of H_e ($\tau \sim 45°$) by a factor of only two.

Table II also indicates that H_β is in a position favorable for abstraction through a five-membered transition state and in fact is more nearly coplanar with the abstracting oxygen n orbital ($\tau = 7–13°$) than is H_e; the tertiary nature of H_β should also facilitate its abstraction.[1] Nevertheless, no products corresponding to abstraction of H_β could be detected either in solution or the solid state. Reversible β-hydrogen abstraction is of course a possibility.

A final point of discussion concerns the difference in cyclization/cleavage ratios in the two media. It is widely accepted[1] that efficient cleavage requires a 1,4-biradical conformation in which both singly occupied p orbitals can overlap significantly with the central σ bond being broken; failing this, cyclization predominates. In the solid state, the 1,4-hydroxybiradical will have the same basic conformation as its ketonic precursor (Figure 1). We suggest that this is the predominant conformation in solution as well. This conclusion is based on the fact, noted previously, that the cyclization stereoselectivity in the solid state is identical with that observed in solution (Table I).

This biradical conformation is very poorly aligned for cleavage. In fact, as indicated by the crystal structure data, it is almost perfectly misaligned, with both singly occupied p orbitals oriented essentially at right angles to the central σ bond.[9] Thus extensive C(1)–C(2) and C(3)–C(4) bond rotations are required for cleavage. The latter motion is of course impeded by ring restraints, hence the preference for cyclization in solution. The reduced amount of cyclization in the solid state can then be ascribed not to enhanced cleavage in this medium but to crystal lattice restriction of cyclization. The product cyclobutanols **3** and **4** bear little structural resemblance to their biradical precursor, and their formation requires extensive molecular and atomic motion, most notably a large permanent displacement of the bulky aryl groups from their original position (Figure 1). This motion sweeps the aryl and hydroxyl groups through a large volume and would be expected to be topochemically disfavored in the solid state relative to the "less-motion" pathway required for cleavage.[10]

Extensions of these concepts to other type II systems and to other organized media are in progress.

Acknowledgment. Support by the Natural Sciences and Engineering Research Council of Canada is gratefully acknowledged.

Registry No. 1a, 79650-21-8; **1a** biradical, 87482-76-6; **1b**, 87482-75-5; **1b** biradical, 87482-77-7; **1c**, 58987-23-8; **1c** biradical, 87482-78-8; **3a**, 87482-79-9; **3b**, 87482-80-2; **3c**, 87482-81-3; **4a**, 87508-65-4; **4b**, 87508-66-5; **4c**, 87508-67-6.

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[9] The p orbital at C(4) was assumed to lie normal to the C(3)–C(4)–C(5) plane. The actual angles for compounds **1a**, **1b** and **1c** respectively were $\theta_1 = 100.7°$, 94.9°, and 96.7° and $\theta_4 = 86.6°$, 88.2°, and 88.6°.

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Kinetics of Free Radical Brominations. Effect of Ring Substitution on the Strength of Benzyl C–H Bonds

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Kistiakowsky and his students pioneered the use of kinetic measurements of gas-phase free radical brominations of hydrocarbons to establish bond dissociation energies (BDE).[1] The first reliable value for methane[2] was followed by measurements on ethane,[3] neopentane,[4] and toluene.[5] The toluene work has been criticized both for concluding that termination occurs by Br·

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[1] George B. Kistiakowsky, 1900–1982, in whose memory this work is dedicated.

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Table I. Kinetic Runs at 45 °C in CCl_4[a]

run	$[RH]_0$	$[Br_2]_0$	[AIBN]	chain length[b]	[HBr][c]	% reaction[d]	*n*, graphic	*n*, numeric
2	51.9	3.98	1.04	953	2.75	93	0.24	0.22
3	52.8	3.00	1.15	1337	0.96	99	0.30	0.22
6	51.9	3.26	5.59	571	1.45	99	0.21	0.24
7	54.5	2.53	3.41	771	1.14	98	0.29	0.35
8	53.5	7.11	1.18	3109	2.98	99	0.14	0.16
9	57.3	8.29	1.19	17811	4.03	99	0.21[e]	0.20

[a] Concentrations in millimoles per liter. [b] At maximum rate. [c] At time of maximum rate. [d] Conversion at end of recording of data. [e] From the intercept of run 9, $I = 1039\ M^{-1}$ min, a minimum value can be calculated for $k_3 = 1.5 \times 10^6\ M^{-1}\ s^{-1}$, using $fk_i = 3.07 \times 10^{-7}\ s^{-1}$ (Kurkov, V. P. Ph.D. Thesis, Columbia University, New York, NY, 1967, p 18) and $k_t = 1 \times 10^{10}\ M^{-1}\ s^{-1}$ (Strong, R. L. *J. Am. Chem. Soc.* 1965, *87*, 3563).

coupling and for yielding a BDE of 89.6 kcal/mol for benzyl C–H;[6,7] at present this value is still in doubt.[8] We have recommended a value near 88 kcal/mol,[9] and we have proposed that ring substitution affects the BDE.[10]

We are investigating the liquid-phase kinetics of brominations of hydrocarbons, initiated by azobis(isobutyronitrile) (AIBN) (eq 1–6). The ratio $k_4/k_5 = n$ was determined at 45 °C by following

$$\mathrm{AIBN} \xrightarrow{fk_i} 2(\mathrm{CH_3})_2\mathrm{CCN} \tag{1}$$

$$(\mathrm{CH_3})_2\dot{\mathrm{C}}\mathrm{CN} + \mathrm{Br_2} \rightarrow (\mathrm{CH_3})_2\mathrm{CBrCN} + \mathrm{Br}\cdot \tag{2}$$

$$\mathrm{Br}\cdot + \mathrm{RH} \xrightarrow{k_3} \mathrm{HBr} + \mathrm{R}\cdot \tag{3}$$

$$\mathrm{R}\cdot + \mathrm{HBr} \xrightarrow{k_4} \mathrm{RH} + \mathrm{Br}\cdot \tag{4}$$

$$\mathrm{R}\cdot + \mathrm{Br_2} \xrightarrow{k_5} \mathrm{RBr} + \mathrm{Br}\cdot \tag{5}$$

$$2\mathrm{Br}\cdot \xrightarrow{k_t} \mathrm{Br_2} \tag{6}$$

spectrophotometrically (time scan) the absorbance of Br_2 in deaerated solutions of toluene, Br_2, and AIBN in CCl_4. If termination is by eq 6, the rate expression is eq 7, and a plot of 1/rate

$$\mathrm{d[RBr]/d}t = k_3k_5((fk_i[\mathrm{AIBN}])/k_t)^{1/2}([\mathrm{RH}][\mathrm{Br_2}])/(k_4[\mathrm{HBr}] + k_5[\mathrm{Br_2}]) \tag{7}$$

vs. $[HBr]/[Br_2]$ should be linear. This was found to be the case. The slope is $S = (k_4/(k_3k_5[\mathrm{RH}]))(k_t/(fk_i[\mathrm{AIBN}]))^{1/2}$, and the intercept is $I = (1/(k_3[\mathrm{RH}]))(k_t/(fk_i[\mathrm{AIBN}]))^{1/2}$. The ratio S/I is n. The spectrophotometer beam did not initiate the reaction, and the decomposition of AIBN did not exceed 0.5%. The early parts of spectral curves showed increasing rates, indicative of spurious inhibition. Data past the maximum rate was used. The variability of the chain length (Table I) indicates possible residual inhibition by O_2 or hydroperoxides, but n is fairly constant, 0.24 ± 0.04. Evidently, small residual inhibition would affect the exponent of $(k_t/fk_i[\mathrm{AIBN}])$, an effect eliminated in S/I. We take $n = 0.21$ as the most reliable since run 9 is the fastest. S and I were obtained also from the best numerical fit of the integrated rate expression to the data. Extrapolation of the gas-phase values to 45 °C gives $n = 0.205$.[5] The excellent agreement in n and in the mode of termination supports VanArtsdalen's BDE value near 89 kcal/mol[5] and is consistent with recent results of "thermal radical production".[11]

In comparing two hydrocarbons, RH and R'H, the difference in ΔH for reactions 3 and 3′ is Δ(BDE) for R–H and R′–H (eq 8).

$$\Delta H - \Delta H' = \{E_a(k_3) - E_a(k_4)\} - \{E_a(k_3') - E_a(k_4')\} \tag{8}$$

Table II. Relative Reactivities and Ratios $k(\mathrm{HBr})/k(\mathrm{Br_2})$[a]

RH	t°	n	n'	k_3/k_3'	apparent[b,g] rel reactivity
p-xylene[c]	45	0.49	0.21[d]	3.5 ± 0.3[e]	2.6 ± 0.2
m-chlorotoluene	45	0.075	0.21[d]	0.17 ± 0.01[e]	0.19 ± 0.01
p-xylene[c]	0	0.10	0.050[f]	4.2 ± 0.3[e]	3.7 ± 0.3
m-chlorotoluene	0	0.02	0.050[f]	0.15 ± 0.01[e]	0.20 ± 0.01

[a] R'H = toluene. Experiments were performed by light initiation with a 4-fold excess of hydrocarbons over bromine (no solvent), at least in triplicate, and were analyzed by NMR. [b] No HBr traps. [c] Per methyl. [d] From absolute rate measurements. [e] In the presence of 3-fold excess of 1,2-epoxybutane; NMR showed that the only product from epoxide was the HBr adduct. [f] Extrapolated with the temperature dependence of VanArtsdalen.[5] [g] See ref 24.

$E_a(k_3) - E_a(k_3')$ can be obtained from the temperature dependence of k_3/k_3', the true relative reactivity in the absence of reversibility, i.e., when eq 4 (and 4′) is suppressed by the addition of epoxides that effectively trap HBr. The temperature dependence of n and n' gives $E_a(k_4) - E_a(k_5)$ and $E_a(k_4') - E_a(k_5')$. It is reasonable to assume that $E_a(k_5) \simeq E_a(k_5')$, since both reactions are exothermic by at least 10 kcal/mol. Consistent with this argument, carbon radicals have been assumed to react with Br_2 with $E_a \simeq 0$.[2–5] Relative reactivities were determined in the usual fashion[10] with a 3-fold excess of 1,2-epoxybutane. The rate expression for direct competition (no HBr traps), eq 9, has not been integrated heretofore.[12–14]

$$\mathrm{d[RBr]/d[R'Br]} = A\{((n'[\mathrm{HBr}])/[\mathrm{Br_2}]) + 1\}/\{((n[\mathrm{HBr}])/[\mathrm{Br_2}]) + 1\} \tag{9}$$

where $A = (k_3/k_3')([\mathrm{RH}]/[\mathrm{R'H}])$. For the common case of substantial excess RH and R′H over Br_2, the integrated form is eq 10. A may be calculated from the initial concentrations of

$$\frac{[\mathrm{RBr}]_f}{[\mathrm{RBr}]_f + [\mathrm{R'Br}]_f} = \frac{A(n'-1)}{A(n'-1) + (n-1)} + \frac{A(n-n')}{\{A(n'-1) + (n-1)\}^2} \ln \frac{An'+n}{A+1} \tag{10}$$

the hydrocarbons and k_3/k_3' as determined with epoxides or alternatively under "bromostasis" conditions;[15] the lhs of eq 10 is determined by complete reaction in the absence of HBr traps. If one of the n values is known the other is obtained by numerical methods. By such competitions with epoxides we obtained the results of Table II. Eq 10 allows determinations of n without recourse to isotopes[16] or large excesses of HBr or Br_2.[17] This method also provides an alternative to interrupting the reaction after a few percent conversion for estimating the effects of reversibility.[18,19] Table II yields $k_3(p\text{-CH}_3)/k_3(m\text{-Cl}) = 3.19$

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$\exp(1178/(RT))$, $n(p\text{-CH}_3) = 7555 \exp(-6092/(RT))$, and $n(m\text{-Cl}) = 228 \exp(-5067/(RT))$. The variation in the preexponential terms for n appears unexpected; VanArtsdalen found $n = 1016 \exp(-5381/(RT))$ for toluene. For the series p-CH$_3$, H, and m-Cl, we have the smooth trend of $\log (A_{\text{HBr}}/A_{\text{Br}_2}) = 3.87$, 3.01, and 2.36. All data of Table II give good Hammett plots vs. σ^+.

From eq 8, $\Delta H(m\text{-Cl}) - \Delta H(p\text{-CH}_3) = 1178 + 6092 - 5067 = 2.2$ kcal/mol. The benzyl C–H bond in m-chlorotoluene is stronger than that in p-xylene by 2.2 kcal/mol, and we estimate the uncertainty at ±0.6. This result is in agreement with Pryor's recent report of a difference of 1.9 kcal/mol.[20] Two independent methods lead to virtually the same result. The substitutent effect is in the direction predicted.[10] As with phenols,[21] Hammett correlations for benzyl hydrogen abstraction by radicals should not be interpreted solely on the basis of "polar effects" in the transition state.[10,22,23]

Registry No. p-Xylene, 106-42-3; m-chlorotoluene, 108-41-8; hydrogen bromide, 10035-10-6; bromine, 7726-95-6.

Supplementary Material Available: The derivation of eq 10; for run 9, the spectrophotometer chart, the FORTRAN program for the numerical method and output, and the plot of (1/rate) vs. [HBr]/[Br$_2$] (7 pages). Ordering information is given on any current masthead page.

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Regioselectivity in the Addition of Carbanions to (1,4-Dimethoxynaphthalene)tricarbonylchromium. A New Entry into Anthracyclinone Synthesis

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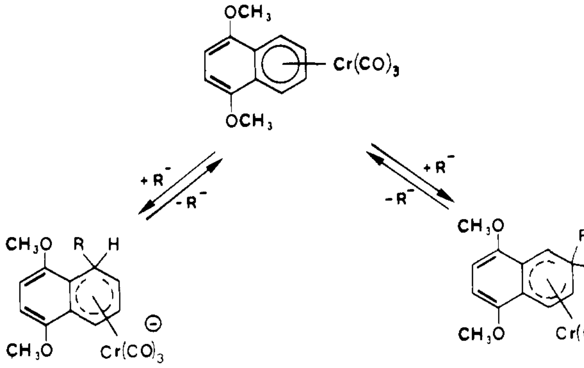
One of the fascinating aspects of the reaction of carbon nucleophiles with substituted arenes coordinated to Cr(CO)$_3$ is the high regioselectivity of the addition often encountered.[1-7] Theoretical analysis[4,8,9] as well as carefully designed experiments[4,6] have demonstrated the interplay of electronic and steric factors[10] in this reaction. Naphthalenetricarbonylchromium (**1**) exhibits a particularly high α regioselectivity (>98%) with a range of nucleophiles of different size and reactivity.[2,11] It has been pointed out[8] that conformational effects of the Cr(CO)$_3$ group cannot account for the observed regioselectivity (the Cr(CO)$_3$ adopts a staggered conformation[12]); however, a correlation with the LUMO coefficient of the free arene has been suggested.[2,13]

In this communication we consider the question of regioselectivity in (1,4-dimethoxynaphthalene)tricarbonylchromium (**2**).[15,16] We expected that superposed on the factors governing regioselectivity in **1** steric and electronic effects due to the OMe groups may become apparent in the reactions of **2**. The data listed in Table I suggest that these effects do, in fact, dominate regiochemistry under conditions of kinetic control.

In a typical reaction, complex **2** (1.5 mmol) was added as a solid to a solution (10 mL of THF, −78 °C) of the carbanion (1.05 equiv) prepared according to standard procedures. After dropwise addition of hexamethylphosphoric triamide (HMPT, 4 mL)[17] the mixture was stirred for the time and at the temperature indicated in Table I. The solution was then recooled to −78 °C, treated with iodine (9 mmol in 10 mL of THF), and left to warm up overnight. Isolation by conventional procedures yielded the substituted naphthalenes.

Steric effects are evident under conditions favoring kinetic control of the addition. The small nucleophile 2-lithioacetonitrile yields predominantly the α substitution product **3** (entries 1 and 2) whereas the sterically more demanding 2-lithio-2-methylpropionitrile gives under analogous conditions predominantly β addition (entries 3–5). With the latter, conversion is virtually complete after 1 h at −50 °C. Consequently, the different product distribution observed on increasing reaction time and/or temperature (entries 4–6) is indicative of a rearrangement to the thermodynamically favored α addition intermediate. Presumably this transfer occurs via dissociation of the carbanion as depicted in eq 1. On the basis of the observed slow exchange of (benzene)tricarbonylchromium between carbanions, reversibility of nucleophilic attack on Cr(CO)$_3$-coordinated arenes has been



(1)

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[16] Substituents on naphthalene have a strong directing effect on the coordination behavior of the arene. Thus, 1,4-dimethoxynaphthalene reacts with Cr(CO)$_6$ regiospecifically to yield the complex with the Cr(CO)$_3$ group coordinated to the nonsubstituted ring.

[17] Complex **2** forms a suspension in the reaction mixture, on addition of HMPT at −60 °C; a homogeneous solution is formed after 30 min.

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