DEOM, bp 92–96 °C (11 mm) [lit.[7] bp 106–108 °C (17 mm)]. The yield, based on diethyl malonate, is 180–205 g (41–47%).

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Registry No. 1, 60308-73-8; diethyl malonate, 105-53-3; diethyl oxomalonate, 609-09-6.

Thermal Decomposition of Benzyl α-Disulfone: The Question of Rate-Determining Homolysis of More Than One Bond in the Thermal Decomposition of an α-Disulfone

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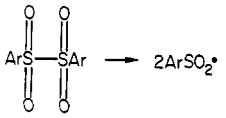
Aryl α-disulfones, $ArSO_2SO_2Ar$ (1), have been shown[1] to undergo thermal decomposition at elevated temperatures (145–165 °C) by a mechanism that involves rate-determining homolysis of the S–S bond (eq 1). The



$$\mathrm{ArS(O_2)\text{—}S(O_2)Ar} \longrightarrow 2\mathrm{ArSO_2}\cdot \qquad (1)$$

$ArSO_2\cdot$ radicals formed in eq 1 then react further in a variety of ways to yield the final decomposition products. Only a few lose sulfur dioxide, however, since only ~0.2 mol of SO_2 is produced per mole of 1 decomposing.

During a study of nucleophilic substitution of alkyl α-disulfones reported elsewhere[2] we had occasion to synthesize benzyl α-disulfone, $PhCH_2SO_2SO_2CH_2Ph$ (2). The present note describes a study of the thermal decomposition of 2 and presents evidence suggestive that the rate-determining step of this reaction may involve the homolysis of *two* bonds, rather than cleavage of only the S–S bond.

Thermal decomposition of 2 was achieved by heating a deaerated solution of the α-disulfone (0.04 M) in bromobenzene at 138 °C for 6 h. A slow stream of nitrogen was passed through the solution throughout the decomposition in order to sweep out sulfur dioxide as it was formed, and the large amount of sulfur dioxide liberated (1.6 mmol/mmol of 2) was determined by passing the exit gas stream through a trap containing standard iodine solution and measuring the amount of I_2 remaining at the end of the decomposition. Chromatography of the nonvolatile residue remaining after in vacuo removal of the bromobenzene solvent gave bibenzyl (0.41 mmol/mmol of 2) and dibenzyl sulfone (0.12 mmol/mmol of 2) as identifiable products.

The kinetics of the thermal decomposition of 2 in bromobenzene was followed by monitoring the rate of evolution of sulfur dioxide by using a technique (see Experimental Section) that has proved reliable in the past[3] for following the kinetics of decompositions where significant quantities of SO_2 are liberated. Plots of log $(1 - SO_2/SO_{2\infty})$ vs. time were nicely linear, showing that the thermal decomposition of 2 exhibits first-order kinetics. Their slope, the experimental first-order rate constant (k_1), was independent of initial concentration of 2. The results of the various kinetic runs are summarized in Table I. The dependence of k_1 on temperature indicates that $\Delta H^{\ddagger} = 37.8$ kcal/mol and $\Delta S^{\ddagger} = +17.3$ eu.

The clean first-order kinetics for the thermal decomposition of 2 is consistent with the rate-determining step of that decomposition being a unimolecular reaction of the α-disulfone. The large positive $\Delta S^{\ddagger}$ suggests this unimolecular reaction is a homolytic dissociation of 2. A heterolytic dissociation ($2 \rightarrow PhCH_2{}^{+}\ {}^{-}O_2SSO_2CH_2Ph$) would certainly not be expected to have a large positive $\Delta S^{\ddagger}$ in a nonpolar solvent like bromobenzene,[4] and neither would a concerted rearrangement. The formation of sizeable amounts of bibenzyl indicates that benzyl radicals are produced during the decomposition of 2, either directly or by loss of sulfur dioxide from $PhCH_2SO_2\cdot$. It thus appears that the thermal decomposition of 2, like that[1] of aryl α-disulfones (1), is a free-radical reaction involving rate-determining homolytic dissociation of the α-disulfone.

Comparison of the kinetic data for 2 in Table I with similar data[1] for the decomposition of 1 indicates that 2 decomposes about 50 times faster than 1 and that $\Delta H^{\ddagger}$ for 2 (37.8 kcal/mol) is 3 kcal/mol less than that for 1 (41 kcal/mol). The much faster rate, and lower $\Delta H^{\ddagger}$, for the thermal decomposition of 2 as compared to 1 brings to mind the rate behavior of the decompositions of *tert*-butyl peresters, RC(O)OOBu-*t*.[6] In the homolysis of such peresters those, like $PhCH_2C(O)OOBu$-*t*, where R, as R·, is a resonance-stabilized radical decompose by a mechanism (eq 2) where there is concerted cleavage of *both* the R–C(O) and O–O bonds in the rate-determining step, while those, like $CH_3C(O)OOBu$-*t* or PhC(O)OOBu-*t*, where R· would not be resonance-stabilized decompose considerably more slowly by a mechanism (eq 3) in which *only* the O–O bond is cleaved in the rate-determining step.[6]

$$\mathrm{PhCH_2CO_2OBu\text{-}}t \xrightarrow[\text{determining}]{\text{rate}} [\mathrm{PhCH_2}\cdots\mathrm{CO_2}\cdots\mathrm{OBu\text{-}}t] \rightarrow \mathrm{PhCH_2}\cdot + \mathrm{CO_2} + t\text{-}\mathrm{BuO}\cdot \qquad (2)$$

$$\mathrm{PhCO_2OBu\text{-}}t \xrightarrow[\text{determining}]{\text{rate}} [\mathrm{PhCO_2}\cdots\mathrm{OBu\text{-}}t] \rightarrow \mathrm{PhCO_2}\cdot + \cdot\mathrm{OBu\text{-}}t \qquad (3)$$

The concerted mechanism has a significantly lower $\Delta H^{\ddagger}$ than the mechanism where only the O–O bond is cleaved in the rate-determining step. However, $\Delta S^{\ddagger}$ for the concerted mechanism is less positive than $\Delta S^{\ddagger}$ for eq 3, and this partially offsets the rate acceleration provided by the lower $\Delta H^{\ddagger}$. Bartlett and Hiatt[6] suggested that the reason $\Delta S^{\ddagger}$ is less positive is because the concerted mechanism requires restrictions to rotation about certain bonds in the transition state that are not required when only the O–O bond is being cleaved in the rate-determining step. Pryor and Smith[7] have subsequently pointed out that, while the insights of Bartlett and Hiatt[6] are useful when treated as qualitative trends, one should not attempt to use relationships between $\Delta H^{\ddagger}$ and $\Delta S^{\ddagger}$ for perester and other peroxide homolyses to assign the *exact* number of bonds undergoing cleavage, or the number about which rotation is restricted, in the rate-determining transition state.

To gain further insight into whether concerted homolysis of more than one bond in the rate-determining step could

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[4] In 90% dioxane reactions of the type $RX \rightarrow R^{+}X^{-}$ generally have entropies of activation of the order of −20 eu;[5] $\Delta S^{\ddagger}$ for a process of this type should be *at least* this negative in bromobenzene.

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Table I. Kinetics of Thermal Decomposition of α-Disulfones in Bromobenzene

α-disulfone	temp, °C	$[(RSO_2)_2]_0$, M	$k_1 \times 10^4$, s^{-1}	$\Delta H^{\ddagger}$, kcal/mol	$\Delta S^{\ddagger}$, eu
$(PhCH_2SO_2)_2$, **2**	137.7	0.02	3.8		
		0.01	3.6	37.8	+17.3
	127.3	0.01	1.12 ± 0.01		
	116.7	0.01	0.28 ± 0.01		
$(PhCH_2CH_2CH_2SO_2)_2$, **3**	143.9	0.01	0.84 ± 0.02		
	133.8	0.01	0.22	42.3	+23.4
	133.5	0.01	0.21		
	127.1	0.01	0.090 ± 0.003		
$(CH_3SO_2)_2$	137.7	0.01	0.10		

be responsible for the faster rate of decomposition of **2** the kinetics of the thermal decompositions of 3-phenylpropyl α-disulfone, $(PhCH_2CH_2CH_2SO_2)_2$, **3**, and methyl α-disulfone were examined. In contrast to **2**, cleavage of a $C-SO_2$ bond in either of these α-disulfones will not yield a reasonance-stabilized radical. The decompositions of **3** and $MeSO_2SO_2Me$ lead to the liberation of only about half as much (~0.9 mmol/mmol of α-disulfone) sulfur dioxide as in the decomposition of **2**, but plots of log $(1 - SO_2/SO_{2\infty})$ vs. time are still nicely linear. Rate constants for the thermal decomposition of **3** at several temperatures are given in Table I; from them $\Delta H^{\ddagger} = 42.3$ kcal/mol and $\Delta S^{\ddagger} = +23.4$ eu for the decomposition of **3**. The rate of decomposition of methyl α-disulfone, obtained at one temperature only, is also given in Table I.

Comparison with the kinetic data for **2** shows that **3** decomposes 12 times and methyl α-disulfone decomposes 35 times *slower* than **2**. The $\Delta H^{\ddagger}$ for the decomposition of **3** is 4.5 kcal/mol greater than that for **2**, while $\Delta S^{\ddagger}$ for **3** is 6 eu more positive. The difference in $\Delta S^{\ddagger}$ causes the difference in the rates of decomposition of **2** and **3** to be 20 times smaller than would be the case if the relative rates were determined by $\Delta(\Delta H^{\ddagger})$ alone.

One sees that in the decomposition of α-disulfones RSO_2SO_2R a change from an R group $(PhCH_2CH_2CH_2)$ where R· is not a resonance-stabilized radical to one $(PhCH_2)$ where it is produces changes in rate, $\Delta H^{\ddagger}$, and $\Delta S^{\ddagger}$ which all parallel those seen in the perester decomposition on going from $CH_3C(O)OOBu$-*t* to $PhCH_2C(O)OOBu$-*t*. Although the decrease in $\Delta H^{\ddagger}$ on going to R = $PhCH_2$ in the α-disulfone decomposition is not as large as that in the perester decomposition,[8] it is still substantial.

While the rate, $\Delta H^{\ddagger}$, and $\Delta S^{\ddagger}$ for the decomposition of **2** as compared to **3** can hardly be said, given the caveat of Pryor and Smith,[7] to provide conclusive evidence for rate-determining homolysis of more than one bond in the decomposition of **2**, they are suggestive that the decomposition of **2** takes place by a mechanism (eq 4) in which both a $PhCH_2-SO_2$ and the S–S bond are cleaved in a concerted fashion in the rate-determining step, in contrast to the decomposition of **3** (eq 5), or **1** (eq 1), where only

$$\underset{\mathbf{2}}{(PhCH_2SO_2)_2} \xrightarrow[\text{determining}]{\text{rate}} [PhCH_2\cdots SO_2\cdots O_2SCH_2Ph] \rightarrow$$
$$PhCH_2\cdot + SO_2 + PhCH_2SO_2\cdot \quad (4)$$

$$\underset{\mathbf{3}}{[Ph(CH_2)_3SO_2]_2} \xrightarrow[\text{determining}]{\text{rate}}$$
$$[Ph(CH_2)_3SO_2\cdots O_2S(CH_2)_3Ph] \rightarrow 2Ph(CH_2)_3SO_2\cdot \quad (5)$$

the S–S bond undergoes homolysis in the rate-determining step. In order for the phenyl group to be able to stabilize mesomerically the developing $PhCH_2\cdot$ radical rotation about the $Ph-CH_2$ bond must be restricted in the transition state for eq 4; this could cause $\Delta S^{\ddagger}$ for **2** to be less positive than $\Delta S^{\ddagger}$ for **3**.

The formation of some dibenzyl sulfone in the decomposition of **2** shows that some combination of $PhCH_2\cdot$ and $PhCH_2SO_2\cdot$ radicals occurs. Given the relatively rapid rate expected for $PhCH_2SO_2\cdot \rightarrow PhCH_2\cdot + SO_2$, this sulfone is probably formed by cage recombination of initially formed $[PhCH_2\cdot \cdot O_2SCH_2Ph]$ radical pairs. Any $PhCH_2SO_2\cdot$ radicals that escape the initial solvent cage would almost certainly lose sulfur dioxide before encountering a benzyl radical in the bulk solution. The isolation of a product arising from a reaction involving $PhCH_2SO_2\cdot$ radicals shows that no more than two bonds in **2** undergo homolysis in the rate-determining step.

If eq 4 is the correct explanation for the faster rate and lower $\Delta H^{\ddagger}$ for the decomposition of **2**, α-disulfones with an R group that yields a more resonance-stabilized radical than $PhCH_2\cdot$ should decompose even faster than **2** and with a lower $\Delta H^{\ddagger}$. We attempted to synthesize such an α-disulfone, $Ph_2CHSO_2SO_2CH_2Ph$, from the known[3a] thiosulfonate $Ph_2CHSO_2SCH_2Ph$, using the same type of oxidation procedure that is successful[2] in converting $PhCH_2SO_2SCH_2Ph$ to **2**. However, when applied to $Ph_2CHSO_2SCH_2Ph$ the oxidation failed to yield the desired α-disulfone; there was obtained instead a considerable amount of benzyl benzhydryl sulfone, $Ph_2CHSO_2CH_2Ph$, together with a number of unidentified products.

Experimental Section

Benzyl α-Disulfone (2). This compound and methyl α-disulfone were synthesized as described in an earlier paper.[2]

3-Phenylpropyl α-Disulfone (3). 1-Bromo-3-phenylpropane (20 g, 0.1 mol) was converted to the corresponding Grignard reagent, and the ether solution of the Grignard reagent was added to a solution of sulfur dioxide (30 mL, 0.69 mol) in 100 mL of ether at −78 °C. The resulting mixture was stirred for 30 min and then allowed to warm to room temperature. All volatile material was then removed by passing a stream of dry nitrogen through the mixture. This left a yellowish solid, the crude sulfinate salt $Ph(CH_2)_3SO_2MgBr$. This was immediately dissolved in 80 mL of aqueous ammonium chloride, and the resulting solution was then acidified to pH <1 by the addition of cold 50% sulfuric acid. The acidified solution was extracted twice with 150-mL portions of ether. The combined ether extracts were dried ($MgSO_4$) and the ether was removed under reduced pressure to give a residue of crude 3-phenylpropane-1-sulfinic acid as a slightly yellowish liquid. This was dissolved in a small amount of water and then neutralized by titration with standard 1 N sodium hydroxide. After titration the solution was extracted with ether to remove traces of organic impurities. The water was then removed, leaving sodium 3-phenylpropane-1-sulfinate (10.5 g, 51%) as a white solid. The procedure for sulfination of the Grignard reagent is that used successfully by Pinnick and Reynolds[11] to synthesize other alkanesulfinates.

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[8] The presently accepted values for $\Delta H^{\ddagger}$ for the decompositions of $CH_3C(O)OOBu$-*t*[9] (34.3 kcal/mol) and $PhCH_2C(O)OOBu$-*t*[9,10] (28.1 kcal/mol) are somewhat different than those used by Bartlett and Hiatt.[6] These values suggest that $\Delta(\Delta H^{\ddagger})$ on going to $PhCH_2C(O)OOBu$-*t* is −6 kcal/mol, not too much larger than the −4.5 kcal/mol for the change from **3** to **2** in the α-disulfone decomposition.

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Sodium 3-phenylpropane-1-sulfinate was oxidized to **2** with an acidic solution of cobaltic sulfate, using the general procedure for conversion of *n*-alkanesulfinates to α-disulfones developed by Denzer et al.[12] The crude product was recrystallized from benzene–ethanol, giving pure 3-phenylpropyl α-disulfone (19%): mp 114–116 °C; IR (KBr) 1113 and 1345 cm^{-1} (SO_2); NMR ($CDCl_3$) δ 2.31 (quintet, 2 H), 2.82 (t, 2 H), 3.42 (t, 2 H), 7.10–7.46 (m, 5 H). Anal. Calcd for $C_{18}H_{22}O_4S_2$: C, 58.99; H, 6.05; S, 17.50. Found: C, 59.10; H, 6.04; S, 17.56.

Procedure for Kinetic Runs. The apparatus used for the kinetic runs was of a design previously described[3a] and shown to be effective for following the progress of reactions that evolve substantial amounts of sulfur dioxide. The desired amounts of α-disulfone and purified bromobenzene were placed in the reaction flask and deaerated by passing a slow stream of dry nitrogen through the solution for 1 h at room temperature, after which the reaction vessel was placed in a constant-temperature bath to initiate decomposition. A stream of nitrogen was passed through the solution during the decomposition to sweep out sulfur dioxide as it was formed. The nitrogen stream was subsequently passed through a trap containing standard iodine solution where the sulfur dioxide was absorbed, and the rate of evolution of sulfur dioxide was followed in the manner outlined by Kice, Parham, and Simons.[3a]

Products of Decomposition of 2. Benzyl α-disulfone (0.62 g, 2 mmol) was decomposed in 50 mL of bromobenzene at 138 °C, using the same apparatus and means of estimating the amount of sulfur dioxide produced (1.6 mmol/mmol of **2**) as in the kinetic runs. After decomposition was complete the bromobenzene was removed under reduced pressure at 90 °C. The residue was chromatographed on silica gel, using successively hexane, various hexane–benzene mixtures, benzene, benzene–ether, ether, ether–methanol, and finally methanol as eluents. Elution with hexane afforded a white solid which was recrystallized from ethanol, giving 0.150 g (0.82 mmol) of bibenzyl, mp 50–53 °C (lit.[13] mp 52–53 °C), identical in all respects with a known sample. Elution with 1:1 benzene–ether gave a fraction, also recrystallized from ethanol, that was dibenzyl sulfone (0.06 g, 0.24 mmol), mp 146–148 °C (lit.[14] mp 151–152 °C), identical with an authentic sample. There was also a small fraction eluted with methanol that was not identified, although the presence of strong absorption in the infrared at 1180–1220 and 1050 cm^{-1} suggested that it was either the hydrate of a sulfonic acid or a sulfonate salt.

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Registry No. **2**, 76625-87-1; **3**, 77357-66-5; 1-bromo-3-phenylpropane, 637-59-2; 3-phenylpropylsulfinyloxymagnesium bromide, 77357-67-6; 3-phenylpropane-1-sulfinic acid, 70385-57-8; sodium 3-phenylpropanol-1-sulfinate, 77357-68-7; sulfur dioxide, 7446-09-5; $(CH_3SO_2)_2$, 10383-49-0.

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Communications

Reductive Cyclization of Keto Acids to Polycyclic Aromatic Hydrocarbons by Hydroiodic Acid–Red Phosphorus

Summary: Hydroiodic acid–red phosphorus in acetic acid causes ring closure of *o*-naphthoylbenzoic acids and of *o*-naphthoylnaphthoic acids to yield benz[*a*]anthracene and dibenzanthracenes.

Sir: Although hydroiodic acid–red phosphorus (HI/P) was introduced as a reagent into organic chemistry over a century ago[1] it has been only sporadically used in the past. Examples of its use are hydrogenations,[1] deoxygenations of alcohols,[2] ketones,[3] keto acids,[3] and quinones,[4] cleavage of phenol ethers,[5] and reductive cleavage of lactones.[6]

A recent report[4] on the reductive cyclization of 2-(9-phenanthroyl)benzoic acid in the presence of HI/P to dibenz[*a*,*c*]anthracene ascribes the ease of cyclization in this special case to the relatively high olefinic character of the phenanthrene 9,10 bond.

We have found that reductive cyclization with HI/P can quite generally be applied to *o*-naphthoylbenzoic acids and to *o*-naphthoylnaphthoic acids. The use of HI/P therefore provides a simple high-yield route to benz[*a*]anthracene, dibenz[*a*,*h*]anthracene, and dibenz[*a*,*j*]anthracene as well as to their hydroxylated derivatives (Table I).

The keto acids **1–6** were prepared in 70–80% yield by the addition of the Grignard reagent from a suitable aromatic bromo compound to an aromatic 1,2-dicarboxylic acid anhydride as described by LaBudde and Heidelberger,[14] modified according to Braun.[15]

The classic way to transform these keto acids to the corresponding polycyclic aromatic hydrocarbons consists of Friedel–Crafts cyclization with sulfuric acid or polyphosphoric acid to the quinones and reduction with aluminum tricyclohexoxide[16] or zinc/pyridine/acetic acid[17] to polycyclic aromatic hydrocarbons. Both steps, especially the cyclization of **4–6**, afford low yields.[14]

Reductive cyclization with HI/P is therefore a very convenient method to overcome the disadvantages of the classic synthesis of dibenzanthracenes.

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