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Thermochemical properties of dibenzyloxy disulfides

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ABSTRACT

The in situ thermal behavior of dialkoxy disulfides (—OSSO—) has been previously explored, however their neat thermal stability has yet to be examined. We synthesized a library of ten dibenzyloxy disulfide derivatives with various *para*-substituents. Each derivative was analyzed by TGA and DSC to discern molecular fragmentation. A correlation of the pattern of fragmentation to Swain and Lupton's *R*-value was observed. Also, DSC analysis revealed that when the *para*-substituent was a phenyl (i.e. bis(p-phenylbenzyloxy) disulfide), and successive runs were performed, the thermogram showed the presence of the fragmentation, and upon ^{1}H NMR analysis its corresponding alcohol and aldehyde were observed.

1. Introduction

Although the dialkoxy disulfide was first synthesized by Lengfeld in 1895 [1], very little has been reported on this functionality until recently [2–11]. Whether the moiety existed as the linear arrangement (—OSSO—), or as its isomeric form, thionosulfite (—OS(=S)O—), was not confirmed until 1997 [3]; however, it has been reported to exist as the latter thionosulfite when in cyclic structures [4]. Due to the ability of a lone pair of electrons from one of the sulfurs to donate into the σ^* orbital of the adjacent S—O bond, the S—S bond is unusually short (ca. 1.95 Å) [5] and has an enhanced barrier of rotation of ca. 18 kcal/mol [6], compared to ~2.05 Å and ~8 kcal/mol for simple disulfides [12]. In addition, although thiosulphurous acid (HOSSOH) has been successfully synthesized at −70 °C, it readily decomposes at 20 °C to afford SO_2 [13], and no salts of this functionality have been reported.

The uniqueness of this functional group has been explored both photo- and thermolytically. Lunazzi and Placucci reported that under photolytic conditions, the alkoxy radicals could be formed from the dialkoxy disulfides, which were subsequently reacted with fullerenes [9]. In addition, Harpp and coworkers observed the conversion of bis-cubylmethyl-dialkoxy disulfide to bis-cubylmethyl sulfite with ambient light while open to the air [8]. Dibenzyloxy disulfides have also been shown to go through autocatalyzed decomposition while in solution and irradiated under UV-light in a manner depicted in Scheme 1 [14].

Thermally, benzylic [7] or cubylcarbinolic [8] dialkoxy disulfides have been heated in solution in the presence of dienes to afford cyclic di- and tetra-sulfides, in a pseudo-Diels-Alder reaction presumably via the liberation of S_2. Harpp had proposed [11], and we have further validated [15], a cage-based mechanism for this process (Scheme 2).

Although a number of studies have been conducted on the thermolytic behavior of dibenzyloxy disulfides in solution, no work has been reported on their thermal profile while neat. Herein, we report the synthesis of ten dibenzyloxy disulfides and their degradative properties when examined by thermal gravimetric analysis (TGA) as well as differential scanning calorimetry (DSC).

2. Experimental

2.1. Materials, preparation and characterization of compounds

All chemicals were reagent grade with the exception of 4-phenoxybenzyl alcohol which was synthesized according to published procedure [16]. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were recorded on a Varian instrument with solvent indicated with tetramethylsilane (TMS) as an internal standard and compared to authentic samples. Chemical shifts (δ) are given in ppm, coupling constants (J) in Hz, and spin multiplicities as s (singlet), d (doublet), ABq (AB quartet) and m (multiplet). Dibenzyloxy disulfides **8**–**10** are new chemical entities and full characterization

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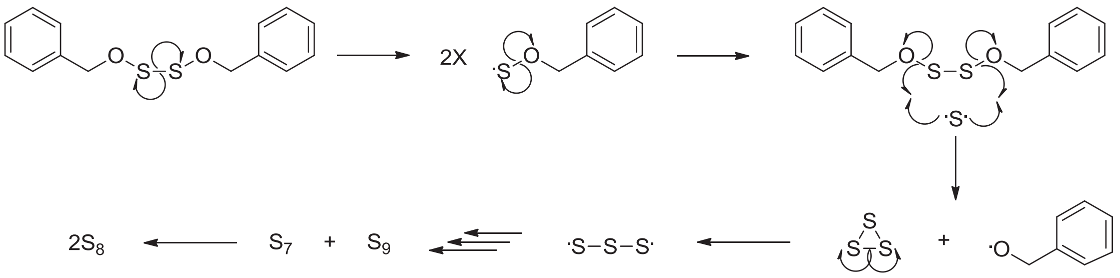


Scheme 1. Priefer's proposed mechanism of the photolytic, autocatalyzed decomposition of dibenzyloxy disulfides [13].

including HRMS recorded on a ThermoFinnigan MAT 95XL with ESI II source for electrospray were performed.

2.2. Representative synthesis of a dibenzyloxy disulfide

*Bis(4-nitrobenzyloxy) disulfide (**1**):* *p*-Nitrobenzyl alcohol (0.25 g, 1.63 mmol) was dissolved in anhydrous CH_2Cl_2 under N_2. Triethylamine (0.227 mL, 1.63 mmol) was added and the resulting solution was cooled to 0 °C. S_2Cl_2 (65.3 μL, 0.82 mmol) was added dropwise over 20 min. The solution was stirred at 0 °C for 2 h before being allowed to equilibrate to room temperature for 3 h. The reaction was quenched with dH_20, washed with 2 × 20 mL aliquots of brine. The aqueous phase was extracted with CH_2Cl_2 (2 × 10 mL), and the combined organic was dried over $MgSO_4$, filtered, and concentrated under reduced pressure. Column chromatography with a 2.5:1 ratio of hexanes:ethyl acetate afforded:

*Bis(p-nitrobenzyloxy) disulfide (**1**, 0.23 g, 93%)* as an off white solid mp. 102–104 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.89, 5.01 (ABq, *J* = 12.6 Hz, 4H), 7.49 (d *J* = 8.8 Hz, 4H), 8.22 (d, *J* = 8.8 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 75.1, 123.8, 128.7, 143.6, 147.9.

*Bis(4-phenylbenzyloxy) disulfide (**2**, 0.23 g, 90%)* as white solid. mp.105–107 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.85, 4.96 (ABq, *J* = 11.4 Hz, 4H), 7.35 (t, *J* = 7.6 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 4H), 7.59 (m, 12H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 76.5, 127.1, 127.3, 127.5, 128.8, 129.2, 135.5, 140.6, 141.4.

*Bis(4-chlorobenzyloxy) disulfide (**3**, 0.23 g, 92%)* as off white solid mp. 102–104 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.75, 4.86 (ABq, *J* = 11.4 Hz, 4H), 7.30 (d, *J* = 7.2 Hz, 4H), 7.33 (d, *J* = 7.2 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 75.8, 128.8, 130.0, 134.4, 134.9.

*Bis(4-methoxybenzyloxy) disulfide (**4**, 0.19 g, 76%)* as white solid. mp. 20–22 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 3.81 (s, 6H), 4.73, 4.84 (ABq, *J* = 11.0 Hz, 4H), 6.88 (d, *J* = 8.4 Hz, 4H), 7.24 (d, *J* = 8.4 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.3, 76.5, 113.9, 128.8, 130.5, 159.8.

*Bis(4-methylbenzyloxy) disulfide (**5**, 0.21 g, 84%)* as off white solid mp. 25–27 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.35 (s, 6H), 4.76, 4.87 (ABq, *J* = 11.2 Hz, 4H), 7.17 (d, *J* = 8.0 Hz, 4H), 7.24 (d, *J* = 8.0 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.3, 77.2, 128.8, 129.2, 133.6, 138.2.

*Bis(4-hydrogenbenzyloxy) disulfide (**6**, 0.20 g, 80%)* as off white solid mp. 43–45 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.80, 4.92 (ABq, *J* = 11.4 Hz, 4H), 7.17 (m, 10H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 76.8, 128.4, 128.5, 128.6, 136.6.

*Bis(4-tertbutylbenzyloxy) disulfide (**7**, 0.19 g, 72%)* as clear liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 1.32 (s, 18H), 4.77, 4.89 (ABq, *J* = 11.2 Hz, 4H), 7.29 (d, *J* = 8.0 Hz, 4H), 7.38 (d, *J* = 8.0 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 31.3, 34.6, 76.7, 125.5, 128.6, 133.6, 151.6.

*Bis(4-trifluorobenzyloxy) disulfide (**8**, 0.17 g, 68%)* as clear liquid. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.85, 4.96 (ABq, *J* = 12.0 Hz, 4H), 7.44 (d, *J* = 7.2 Hz, 4H) 7.62 (d, *J* = 7.2 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 75.7, 124.0 (*J* = 272.1 Hz), 125.5 (*J* = 3.8 Hz), 128.4, 130.6 (*J* = 32.2) 140.4. Exact mass calculated for $C_{16}H_{12}O_2F_6S_2$ 414.0177, found 414.01779.

*Bis(4-cyanobenzyloxy) disulfide (**9**, 0.12 g, 67%)* as white-yellow solid mp. 27–29 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.84, 4.96 (ABq, *J* = 12.6 Hz, 4H), 7.44 (d, *J* = 7.6 Hz, 4H) 7.44 (d, *J* = 7.2 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 75.4, 112.0, 118.6, 128.6, 132.4, 141.6. Exact mass calculated for $C_{16}H_{12}O_2N_2S_2$ 328.0335, found 328.03448.

*Bis(4-phenoxybenzyloxy) disulfide (**10**, 0.65 g, 88%)* as off white solid mp. 24–26 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 4.77, 4.88 (ABq, *J* = 11.4 Hz, 4H), 6.98 (d, *J* = 8.4 Hz, 4H), 7.01 (d, *J* = 7.6 Hz, 4H), 7.13 (t, *J* = 7.2 Hz, 2H), 7.33 (m, 8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 76.3, 118.6, 119.1, 123.5, 129.8, 130.5, 131.3, 156.8, 157.7. Exact mass calculated for $C_{26}H_{22}O_4S_2$ 462.0954, found 462.09551.

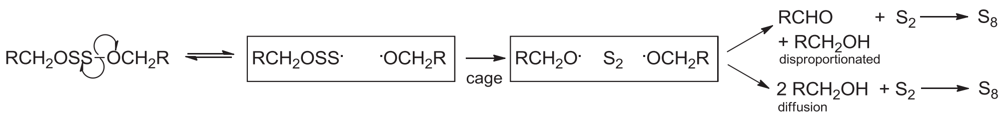
2.3. Measurements

Thermal properties were examined by thermogravimetric analysis, TGA, with TA instruments TGA Q50 System in a N_2 atmosphere and heated at a rate of 10 K/min. The differential scanning calorimetry, DSC, was run with a Perkin-Elmer Pyris 6 calorimeter in a N_2 atmosphere. The solid sample was placed in an aluminum capsule, heated and cooled at the rate of 5 K/min. All results were taken from the multiple heating and cooling runs, detecting the level of enthalpy change that is associated with a respective phase transition. The transition enthalpy, ΔH (kJ/mol), was determined from the peak area of the DSC thermogram. The transition entropy, ΔS (J/mol K), was calculated with the equation $\Delta S = \Delta H/T$, where T was the transition temperature corresponding to the DSC maximum. The thermodynamic data were mean values of several independent measurements carried out on different samples.

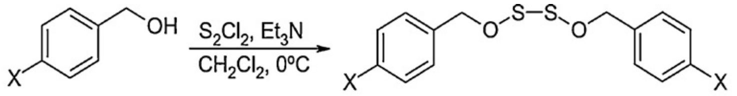
3. Results and discussion

3.1. Synthesis

Previous work on dibenzyloxy disulfide focused on seven *para*-substituted derivatives [14,15]. For this study we expanded this to ten total dibenzyloxy disulfides. Synthesis of the library was performed as previously described [14,15]. In short, two equivalents of a desired commercially available benzyl alcohol (with the



Scheme 2. Harpp's proposed cage mechanism of thermolytic decomposition of dialkoxy disulfides [11].



X= NO_2 (**1**), Ph (**2**), Cl (**3**), MeO (**4**), Me (**5**), H (**6**), *t*Bu (**7**), CN (**8**), CF_3 (**9**) PhO (**10**)

Scheme 3. Synthesis of dibenzyloxy disulfides.

exception of 4-phenoxybenzyl alcohol which was synthesized from its corresponding acid following published procedure [15]) with two equivalents of Et_3N and one equivalent of S_2Cl_2 were reacted in CH_2Cl_2 for 2 h at 0 °C than 3 h at room temp (Scheme 3). Compounds were purified via column chromatography and stored in a freezer until use. Even when stored cold, slight decomposition was occasionally observed, and these particular dibenzyloxy disulfide were re-purified prior to any thermal studies.

3.2. *Thermogravimetric analysis*

In an effort to understand the thermolytic character of dibenzyloxy disulfides with respect to the electron donating and withdrawing groups, we initially examined them using TGA. Several milligrams of each derivative were individually heated at 10 K/min to 1000 K or until a plateau in weight was reached and no further degradation was observed (Table 1). Of the ten dibenzyloxy disulfides that were evaluated, five (i.e. (**1**) NO_2, (**2**) Ph, (**6**) H, (**8**) CN, (**9**) CF_3) had a single slope degradation profile with some residual black charred material remaining on the pan. The remaining five had either a two-step (i.e. (**3**) Cl, (**5**) Me, (**7**) *t*Bu) or three-step (i.e. (**4**) OMe, (**10**) OPh) degradation profile.

In an attempt to understand these observed degradation patterns we initially examined Swain and Lupton's *F*-value, which had been used to explain rates of decomposition in situ [15]. Unfortunately, no correlation was observed. However, utilization of Swain and Lupton's *R*-value [17] did prove successful (Table 2). It does appear that the more negative the Swain and Lupton's *R*-value is for a given substituent, the more likely it is to undergo a stepwise degradation. Between an *R*-value of −0.138 and −0.161 there is a two-step degradation process. Above −0.500, the degradation is three steps. On the other end, substituents that possess an *R*-value greater than −0.088 have a single step degradation profile.

It appears that substituents that have the ability to donate electron density into the system have a stabilizing effect. Since both the bis(methoxybenzyloxy) (**4**) and bis(phenoxybenzyloxy) (**10**) derivatives have a low onset degradation temperature of 383 and 398 K, respectively, the substituents are most likely stabilizing the daughter fragments rather than the starting dibenzyloxy disulfides. In addition, regardless of whether the degradation was 2- or 3-step, all five of these dibenzyloxy disulfides lost mass corresponding to sulfur between 475 and 525 K (Fig. 1). As a comparison, dibenzyl disulfide has previously been evaluated via TGA [18]. As with dibenzyloxy disulfides **6**, a one-step thermal decomposition was observed, with an onset, inflection point, and end point at 509, 544, and 570 K, respectively. The onset temperature is 111 K higher for the dibenzyl disulfide than compound **6**. However, the end point is only 80 K higher. Unfortunately, these two compounds cannot be perfectly compared since the reported dibenzyl disulfide was heated under air, whereas ours were under a nitrogen atmosphere.

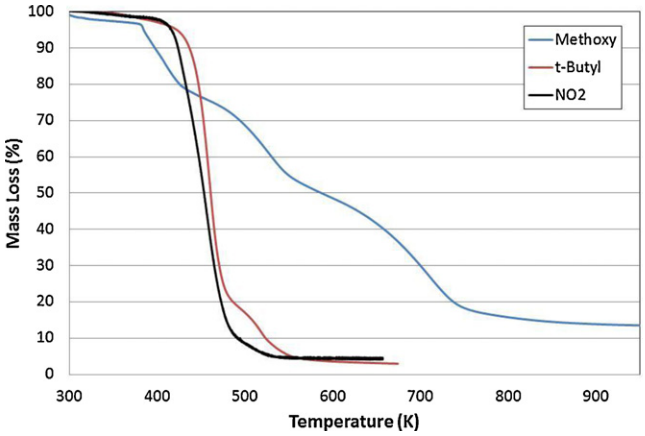


Fig. 1. Representative TGA curves for (**1**) NO_2, (**4**) MeO, and (**7**) *t*Bu.

3.3. *DSC analysis*

Only compounds **1**, **2**, **3**, and **6** were able to be run via DSC because from the synthesized library, they were the only solid compounds with melting points above room temperature. Compounds **1**, **3** and **6** each had endotherms at 367, 318, and 320 K, ΔH of 29.8, 38.1, and 19.5 kJ/mol, and ΔS of 81.2, 119.8, and 60.9 J/mol K, respectively. However, these three dialkoxy disulfides did not produce an exotherm upon cooling (Table 3). This is not surprising as these temperatures were near the onset temperature for degradation from the TGA results (Table 1). When the aluminum capsules were opened and NMR spectra were run on the material, no starting dialkoxy disulfides were detected. The temperature, enthalpy, and entropy of fusion of the dibenzyl disulfide analog of **6** has been previously studied [18]. The melting temperature of dibenzyl disulfide was 342 K, compared to 320 K for **6**. In addition, the ΔH and ΔS was reported to be 44.7 kJ/mol and 130.8 J/mol K for dibenzyl disulfide, which is significant elevated compared to compound **6**'s 19.5 kJ/mol and 60.9 J/mol K, respectively. These data suggests that the dialkoxy disulfide does not possess as strong intermolecular forces, when in solid state, as its corresponding disulfide.

Of the compounds that were run via DSC, only dialkoxy disulfide **2** gave both an endotherm upon heating and an exotherm upon cooling (Fig. 2). On the initial DSC cycle an endotherm at

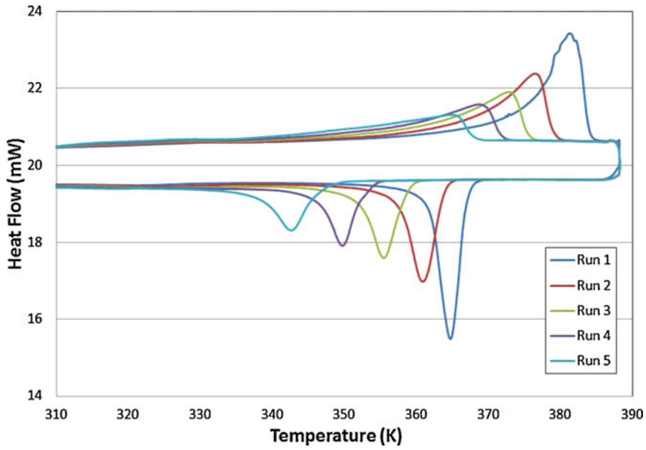


Fig. 2. DSC thermogram of bis(*p*-phenylbenzyloxy) disulfide (**2**).

Table 1
Temperature at onset, inflection and end for dibenzyloxy disulfides **1–10**.

Compound	Extrapolated onset temperature for degradation (K)	Temperature of inflection point (K)	Extrapolated end temperature for degradation (K)
Nitro (**1**)	410	448	533
Biphenyl (**2**)	403	438	467
Chloro (**3**)	418	447	464
	469	488	513
Methoxy (**4**)	383	408	425
	485	514	569
	633	675	789
Methyl (**5**)	403	426	454
	462	497	536
Hydrogen (**6**)	398	437	490
tert-butyl (**7**)	423	457	482
	486	507	540
Cyano (**8**)	380	441	532
TriFluoro (**9**)	393	441	484
Phenoxy (**10**)	398	479	570
	653	725	743
	755	823	883

Table 2
Swain and Lupton's *R*-values as comparison to # of degradation steps.

Compound #	Substituent	Swain and Lupton's *R*-value	# of degradation steps
10	OPh	−0.740	3
4	OMe	−0.500	3
3	Cl	−0.161	2
5	Me	−0.141	2
7	tBu	−0.138	2
2	Ph	−0.088	1
6	H	0	1
1	NO_2	0.155	1
8	CN	0.184	1
9	CF_3	0.186	1

Table 3
Transition temperatures, *T*, enthalpies, ΔH, and entropies, ΔS for dibenzyloxy disulfides **1**, **2**, **3** and **6**.

Compound	Heating			Cooling		
	T (K)	ΔH (kJ/mol)	ΔS (J/mol K)	*T* (K)	ΔH (kJ/mol)	ΔS (J/mol K)
Nitro (**1**)	367	29.8	81.2			
Biphenyl (**2**)	381	43.2	113.4	365	−29.7	−81.4
	377	31.6	83.8	360	−24.1	−66.9
	373	25.2	67.6	355	−19.0	−53.5
	369	19.6	53.1	349	−17.7	−50.7
	365	18.4	50.4	342	−17.4	−50.7
Chloro (**3**)	318	38.1	119.8			
Hydrogen (**6**)	320	19.5	60.9			

381 K (ΔH = 43.2 kJ/mol; ΔS = 113.4 J/mol K) and an exotherm at 365 K (ΔH = −29.7 kJ/mol; ΔS = −81.4 J/mol K) were observed. This heating/cooling was repeated four more times, and with each run there was a decrease in the temperature of the endotherm, ΔH, and ΔS upon heating. On the cooling runs the temperature of the exotherm decreased, while the ΔH and ΔS increased. The changes from run 1 to 5 are proposed to be due to the formation of a new product; hence introducing an impurity into the mix, which should lower and broaden the melting point. We have indeed observed this phenomenon before, albeit with a cubane based molecule [19]. However, as indicated above, this particular structural moiety is thermally sensitive and should fragment upon heating. Indeed, what was observed upon post-DSC NMR was starting alcohol and its corresponding aldehyde in an approximate 1:1 ratio.

4. Conclusion

The thermal behavior of a library of ten *para*-substituted dibenzyloxy disulfides was evaluated via TGA and DSC. A correlation to the number of degradation steps to Swain and Lupton's *R*-value was observed, whereby strongly electron-donating substituents stabilize daughter fragments. In addition, the DSC profile of bis(*p*-phenylbenzyloxy) disulfide (**2**) presented an unusual thermogram, where with each successive run the decomposition introduced an alcohol and aldehyde impurity at an approximate 1:1 ratio which caused a shift and broadening of both the endo and exotherms.

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