

treatment with potassium *tert*-butoxide, probably¹² via the ylide **10**. The primary hydrocarbon products **5** and **11** cannot be isolated despite many variations in the reaction conditions. However, with added furan they are trapped as cycloadducts **12** (20%),^{13,14} **13** (13%),^{13,15} and **14** (7%)¹⁶ (Scheme I). The symmetry planes in **12** and **13** are evident from the appearance of only 10 resonances in their ¹³C NMR spectra^{14,15} and the orientation of the three-membered ring with respect to the oxygen bridge is confidently assigned from the deshielding influence the oxygen atom has upon the ¹H NMR shift of H12a in **12**. Thus **12** displays a one-proton doublet ($J = 4.6$ Hz) at 2.55 ppm for H12a, whereas the equivalent resonance in **13** (1.97 ppm, $J = 5.2$ Hz) is more shielded. It is interesting that the major isolable products emanate from **5**, formed by abstraction of the benzylic H9b of **10**. The appearance of **14**¹⁶ as a minor product reflects the loss of H1 from **10** to give **11**, which is known¹⁶ to add stereospecifically to furan to give **14**.

A complementary approach to **5**, free of competing elimination modes, employs retrodiene cleavage and parallels the successful syntheses of **1**^{3a} and **4**.^{7a} Thus vacuum pyrolysis of **16**,¹⁷ the dicyanoacetylene adduct^{7a} of **15**,¹⁸ followed by low-temperature manipulation of the product mixture, has provided a dibromodifluoromethane solution of **5**¹⁹ free of *o*-dicyanobenzene (**17**) (Scheme II). The NMR spectral data recorded at -60 °C are in full accord with **5**. The C1 methylene protons appear as a singlet (3.54 ppm) in the expected⁶ range for the cycloproparenes and the aromatic proton multiplets typify the phenanthrene system. The single high-field carbon resonance (23.6 ppm, C1) has the highest chemical shift (by 4 ppm) yet recorded for C1 of a C1-unsubstituted cycloproparene, but the magnitude of the C1-H coupling (170.9 Hz) is normal.⁶ Seven distinct aromatic carbon resonances are noted but the shielding of the ortho carbons C1b(9a) is reduced from the usual⁶ 14-16 ppm to ~6 ppm. Prepared in this way, **5** reacts with furan to give **12** (28%) and **13** (17%) and with α -pyrone to yield tetraene **15** (41%) (after loss of carbon dioxide below 0 °C) (Scheme II). On prolonged standing the solution of **5** decomposes even at -60 °C. However, **5** has been isolated as a moderately stable solid which decomposes slowly over a period of days at -78 °C.

Acknowledgment. Financial assistance from the New Zealand Universities Grants Committee (to B.R.D.) and an Alexander von Humboldt fellowship (to D.L.O.; 1984-1985) are acknowledged gratefully.

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(13) Compounds **12** and **13** may be named as *syn*- and *anti*-3,4:5,6-dibenzo-11-oxatetracyclo[6.2.1.1^{2,7}.0^{2,7}]dodeca-3,5,9-triene, respectively.

(14) **12**: mp 179.5-180.5 °C. Anal. (C₁₉H₁₄O) C, H. ¹H NMR δ 0.58 (d, $J = 4.6$ Hz, H12b), 2.55 (d, $J = 4.6$ Hz, H12a), 5.26 (s, H1,8), 6.34 (s, H9,10), 7.20-7.30 (m, H14,15,18,19), 7.45-7.60 (m, H13,20), 7.85-7.95 (m, H16,17); ¹³C NMR δ 25.6 (t, C12), 35.6 (d, C2,7), 79.4 (d, C1,8), 123.6, 126.7, 127.9, 128.9 (all d, C13-20), 130.5 (s, C3,6), 134.5 (s, C4,5), 139.4 (d, C9,10).

(15) **13**: mp 183-185 °C. Anal. (C₁₉H₁₄O) C, H. ¹H NMR δ 0.77 (d, $J = 5.2$ Hz, H12b), 1.97 (d, $J = 5.2$ Hz, H12a), 5.36 (s, H1,8), 6.45 (s, H9,10), 7.25-7.35 (m, H14,15,18,19), 7.50-7.60 (m, H13,20), 7.95-8.05 (m, H16,17); ¹³C NMR δ 31.9 (s, C2,7), 35.2 (t, C12), 84.8 (d, C1,8), 123.5, 126.6, 128.0, 129.0 (all d, C13-20), 130.5 (s, C3,6), 133.1 (d, C9,10), 134.8 (s, C4,5).

(16) Compound **14** provides analytical and spectroscopic data in accord with those previously published: Coburn, T. T.; Jones, W. M. *J. Am. Chem. Soc.* **1974**, *96*, 5218-5227.

(17) Compound **16** may be named as 3,4:5,6-dibenzo-9,10-dicyano-tetracyclo[6.2.2.1^{2,7}.0^{2,7}]trideca-3,5,9,11-tetraene: 67%, mp 214-215 °C. Anal. (C₂₃H₁₄N₂) C, H, N. ¹H NMR δ 0.32 (d, $J = 5.7$ Hz, H13b), 1.99 (d, $J = 5.7$ Hz, H13a), 4.80 (t, $J = 3.9$ Hz, H1,8), 6.38 (t, $J = 3.9$ Hz, H11,12), 7.30-7.45 (m, H15,16,19,20), 7.70-7.80 (m, H14,21), 7.90-8.00 (m, H17,18); ¹³C NMR δ 26.6 (t, C13), 32.6 (s, C2,7), 47.0 (d, C1,8), 114.1 (s, CN), 123.3, 127.5, 128.1, 129.0, 131.1 (all d, C11,12,14-21), 127.0, 133.4, 137.4 (all s, C3-6,9,10).

(18) Compound **15** is available from triphenylene in seven steps: Tokita, S.; Schophoff, F.; Vogel, E. manuscript in preparation.

(19) 1H-Cyclopropa[1]phenanthrene (**5**): ¹H NMR δ 3.54 (s, H1), 7.68-7.80 (m, H3,4,7,8), 7.95-8.00 (m, H2,9), 8.75-8.85 (m, H5,6); ¹³C NMR δ 23.6 (t, $J_{CH} = 170.9$ Hz, C1), 124.1, 124.6 (both s, C1a,9b and C1b,9a), 125.3, 125.8, 127.4, 128.6 (all d, C2-9), 133.5 (s, C5a,5b).

Registry No. **5**, 278-91-1; **8**, 98420-55-4; **9**, 98420-57-6; **10**, 98420-58-7; **11**, 57741-39-6; **12**, 98420-59-8; **13**, 98523-81-0; **14**, 53137-63-6; **15**, 98420-60-1; **16**, 98420-61-2; **17**, 91-15-6; furan, 110-00-9; dicyanoacetylene, 1071-98-3; α -pyrone, 504-31-4.

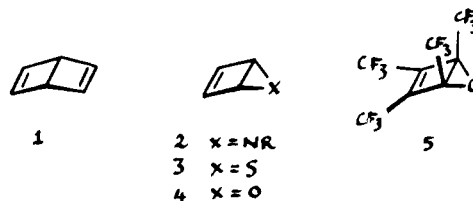
Dewar Furan: Its Generation and Trapping with Isobenzofuran

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It is now more than 20 years since van Tamelen and Pappas announced, in a landmark paper at that time, the preparation of the parent Dewar valence isomer **1** of benzene.² In contrast, the



heterocyclic Dewar valence isomers **2-4**³ remained unknown in unsubstituted form until 1985, when Strausz and his co-workers reported the trapping of Dewar thiophen (**3**), albeit in very low yield.⁴ Photoisomerization of the appropriate five-membered heterocycle has formed the major thrust for preparing such valence isomers and has been applied successfully to the generation of substituted Dewar thiophenes⁵ and Dewar pyrroles.⁶ However, irradiation of furans, while producing a variety of rearrangement products, offers no evidence for Dewar furans (5-oxabicyclo-[2.1.0]pent-2-enes).⁷ Indeed the few known examples,^{8,9} e.g., the perfluoromethyl derivative **5** reported by Lemal,⁸ have been prepared by indirect means.

Thus, in seeking a route to Dewar furan we designed the photochemical precursor **13** which is based on the established success of the 1,2-photoaromatization reaction to produce highly strained four-membered cyclic olefins.¹⁰ In this paper we discuss the results of this approach and announce the first example of the generation of Dewar furan **4** and its efficient trapping in adduct form with isobenzofuran (**15**).¹¹

The photosubstrate **13** was prepared as outlined in Scheme I.¹²

(1) Visiting Fellow ANU.

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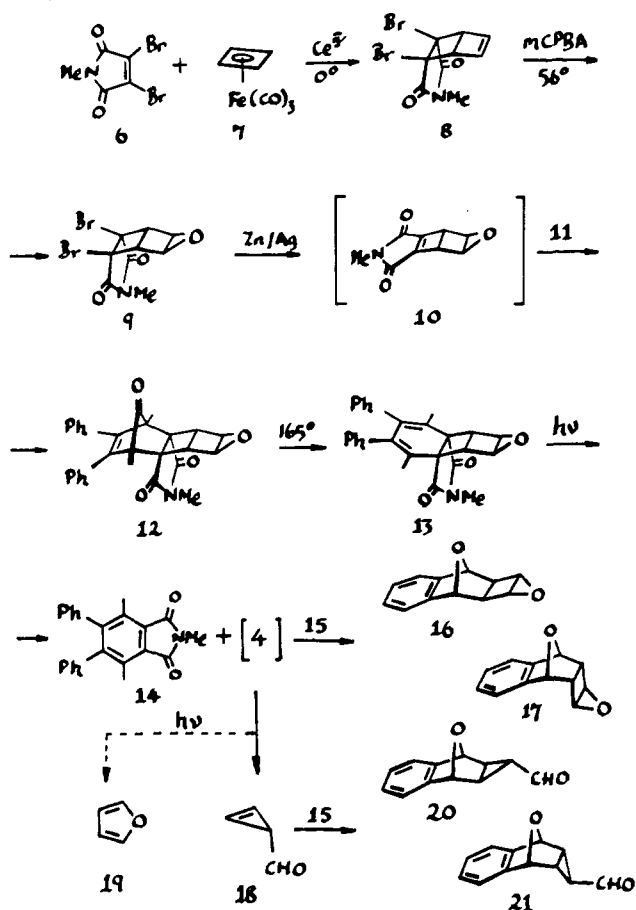
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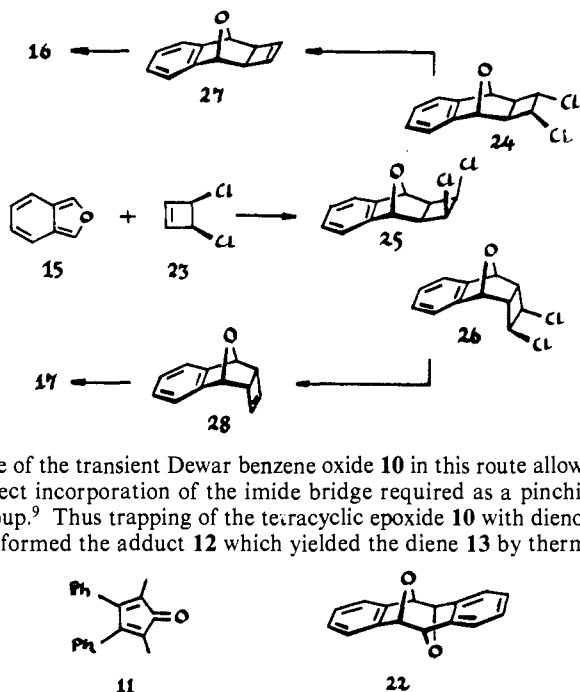
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Scheme I



Scheme II

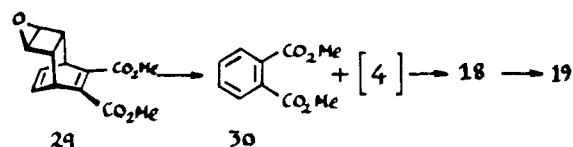


Use of the transient Dewar benzene oxide **10** in this route allowed direct incorporation of the imide bridge required as a pinching group.⁹ Thus trapping of the tetracyclic epoxide **10** with dienone **11** formed the adduct **12** which yielded the diene **13** by thermal

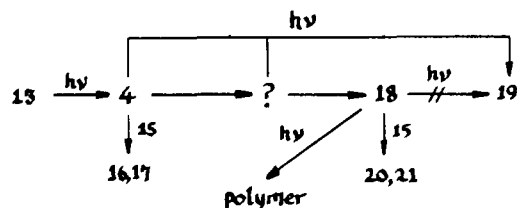
decarbonylation. Photoaromatization of the diene **13** took place rapidly (THF, 0 to -80°C , 254 nm) and the phthalimide **14** was isolated in quantitative yield. Furan (**19**) was the only other

(12) All new compounds gave satisfactory elemental analyses, ^1H NMR, and mass spectra. Melting points, $^\circ\text{C}$ (% yields): **8**, 158–159 (90); **9**, 177–178 (75); **12**, 220–221 (71); **13**, 209–210 (88); **14**, 217–218 (100); **16**, 123–124; **17**, 130–131; **20**, 80–82; **21**, 103–104.

Scheme III



Scheme IV



product detected (^1H NMR spectroscopy) in photolyses of the diene **13** conducted down to -80°C , in yields varying between 5% and 15%. However, when photolysis of the diene **13** was carried out in THF containing 20% w/v of isobenzofuran (**15**) a mixture of four $\text{C}_4\text{H}_4\text{O}$ /isobenzofuran cycloadducts was obtained, together with the phthalimide **14** and the head to head photodimer **22** of isobenzofuran.¹³ The adducts were separated by HPLC¹⁴ and shown to be the Dewar furan adducts **16** and **17** (ratio 3:2)¹⁵ and the cyclopropene carbaldehyde adducts **20** and **21** (ratio 3:1).¹⁶ The structure of these adducts was assigned by NMR and confirmed by separate synthesis (see Scheme II).¹⁷ The proportion of Dewar furan adducts **16** and **17** increased significantly as the temperature of the photolysis was lowered (from 20% at -5°C to 70% at -65°C) and a corresponding decrease in the adducts **20** and **21** was observed. These data indicate that aldehyde **18** is a secondary product, formed thermally from Dewar furan even at -65°C , and reflects the low activation energy attendant upon the conversion of Dewar furan (**4**) to the aldehyde **18**.

In seeking a way to produce an authentic sample of cyclopropenecarbaldehyde (**18**) we reasoned that thermal rearrangement of Dewar furan could be turned to our advantage (Scheme III). Thus flash vacuum pyrolysis (FVP) of the epoxide **29**¹⁸ at 510°C produced the phthalate **30** together with a 60:40 mixture of furan and the cyclopropenecarbaldehyde (**18**)¹⁹ via the presumed intermediacy of Dewar furan (**4**).

Irradiation of pure cyclopropenecarbaldehyde (**18**)²⁰ was carried out as for **13**, (see above) and, while rapid decomposition occurred, no simple photoproducts were observed. This accounts for the

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(14) The mixture was resolved into its components by HPLC on a Whatman Partisil M9 10/25 column using 10% ethyl acetate in hexane as eluent at 4 mL/min. Retention times: **22**, 13.3; **20**, 17.7; **17**, 18.7; **16**, 22.3 min.

(15) **16**: ^1H NMR (CDCl_3) δ 7.19 (m, 4 H), 5.34 (s, 2 H), 3.91 (d, $J = 2.2$ Hz, 2 H), 2.43 (d, $J = 2.2$ Hz, 2 H). **17**: ^1H NMR (C_6D_6) δ 6.85 (m, 4 H), 4.81 (m, 2 H), 2.81 (m, 2 H), 2.74 (s, 2 H).

(16) **20**: ^1H NMR (CDCl_3) δ 9.33 (d, $J = 4.4$ Hz, 1 H), 7.20 (m, 4 H), 5.18 (s, 2 H), 2.84 (dt, $J = 4.4, 2.7$ Hz, 1 H), 1.94 (d, $J = 2.7$ Hz, 2 H). **21**: ^1H NMR (CDCl_3) δ 9.13 (d, $J = 3.5$ Hz, 1 H), 7.18 (s, 4 H), 5.43 (dd, $J = 1.8, 0.5$ Hz, 2 H), 2.82 (m, 2 H), 0.99 (dt, $J = 3.5, 2.5$ Hz, 1 H).

(17) Reaction of **15** with *cis*-3,4-dichlorocyclobutene was effected in THF at reflux to produce a mixture of 1:1 adducts: **24**, mp 126–127 $^\circ\text{C}$; **25**, mp 176–177 $^\circ\text{C}$; **26**, 165–166 $^\circ\text{C}$. Dechlorination (Zn/EtOH, reflux, 12 h) yielded the *exo*-cyclobutene **27**, mp 73–74 $^\circ\text{C}$, from **24** and *endo* isomer **28**, mp 54–55 $^\circ\text{C}$, from **26**. Epoxidation (mCPBA) of **27** yielded epoxide **16** while **28** yielded epoxide **17**. The isomers **20, 21** were produced by reaction of **18** with **15** at -78°C in THF and were separated by centrifugal layer chromatography.

(18) Koz'min, A. S.; Yur'eva, N. M.; Kirin, V. N.; Bylina, G. S.; Luzikoo, Y. N.; Moiseenkov, A. M. *Zh. Org. Khim.* **1983**, *19*, 1543–1544.

(19) The ^1H NMR spectrum of **18** [$\delta(\text{CDCl}_3)$ 8.85 (d, $J = 7.1$ Hz, 1 H), 7.12 (d, $J = 1.3$ Hz, 2 H), 2.32 (dt, $J = 7.1, 1.3$ Hz, 1 H)] has previously been misreported (Tsuchiya, T.; Arai, H.; Igeta, H. *Tetrahedron* **1973**, *29*, 2747–2751).

(20) Mixtures of furan **19** and cyclopropenecarbaldehyde (**18**) were obtained upon the FVP of **29** which varied with the temperature (e.g., 420°C 10% conversion 100% **18**; 540°C 100% conversion 10% **18**, 90% **19**). Pure **18** was isolated by fractional distillation.

absence of cyclopropenecarbaldehyde (**18**) in the direct irradiation of the diene **13**. Additionally this experiment shows that cyclopropenecarbaldehyde (**18**) is not the precursor to furan (**19**) produced in the photolysis of **13**, although this is a well-documented photorearrangement of substituted acylcyclopropenes.²¹ As no furan is produced upon irradiation of the diene **13** in the presence of isobenzofuran, either Dewar furan (**4**) or some intermediate species involved in its thermal rearrangement to cyclopropenecarbaldehyde (**18**) must be photoreactive and act as the precursor to furan. On the basis of these results we can summarize the photolysis of the diene **13** as shown in Scheme IV, in which Dewar furan (**4**) plays a key role. Experiments on elucidating the nature of such intermediate(s) are under active investigation and will be reported in due course.

Registry No. **4**, 74496-19-8; **6**, 3005-27-4; **7**, 12078-17-0; **8**, 98652-86-9; **9**, 98652-87-0; **10**, 98652-88-1; **11**, 26307-17-5; **12**, 98652-89-2; **13**, 98652-90-5; **14**, 95641-37-5; **15**, 270-75-7; **16**, 98652-91-6; **17**, 98717-52-3; **18**, 36998-21-7; **20**, 98652-92-7; **21**, 98717-53-4; **23**, 2957-95-1; **24**, 98652-93-8; **25**, 98717-54-5; **26**, 98717-55-6; **27**, 98652-94-9; **28**, 98717-56-7; **29**, 84636-45-3.

(21) Inter alia: Padwa, A.; Akiba, M.; Chou, C. S.; Cohen, L. *J. Org. Chem.* **1982**, *47*, 183-191.

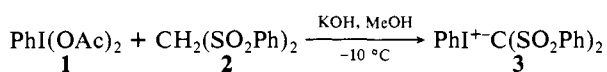
Phenylidonium Bis(phenylsulfonyl)methylide: A New Hypervalent Iodonium Ylide

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Compounds with a sulfonyl group in general¹ and *gem*-disulfones in particular² are increasingly attracting attention for their potential in organic synthesis. At the same time, iodonium ylides are being established as a class of ylides of synthetic and mechanistic interest.³ We report the synthesis and chemistry of a new type of iodonium ylide, the carbanion moiety of which is a β -disulfonyl group, i.e., phenylidonium bis(phenylsulfonyl)methylide (**3**). This compound, which could not be formed from the disulfone **2** and iodosylbenzene,⁴ has now been prepared from diacetoxyiodobenzene (**1**) and **2** in 90% yield by the general method of Schank and Lick.⁵



It is mentioned that the synthesis of the related ylides $\text{RC}_6\text{H}_4\text{I}^+\text{C}(\text{SO}_2\text{F})_2^-$ has been reported,⁶ without any reactions.

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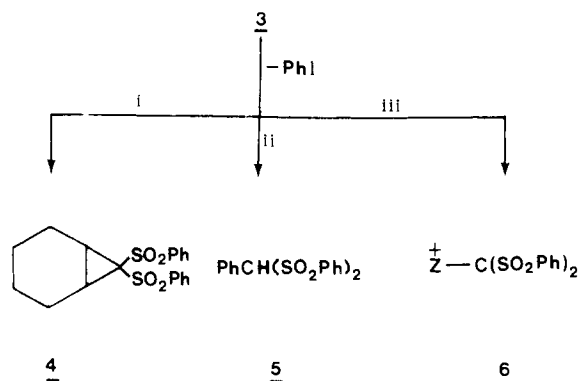
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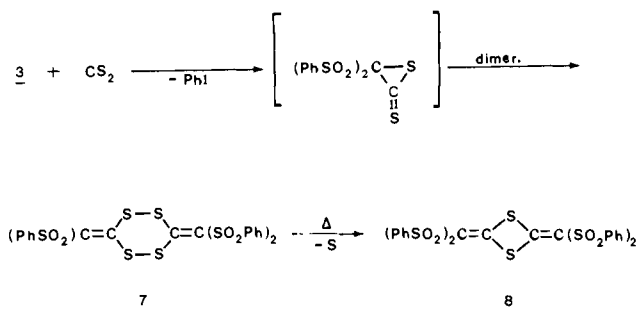
(5) Schank, K.; Lick, C. *Synthesis* **1983**, 392.

Scheme I^a



^a Reactants, conditions, and products (with % yields). (i) cyclohexene, MeCN-*hν*, 4 h, **4** (31%); (ii) PhH, *hν*, 6 h, **5** (65%); (iii) Z = pyridine, reflux, Cu(acac)₂, 4 h, **6a** (75%); Z = Ph₃P, CHCl₃, reflux, Cu(acac)₂, 3 h, **6b** (92%); Z = (Me₂N)₂CS, CHCl₃, reflux, Cu(acac)₂, 7 h, **6c** (78%); Z = Me₂S, *hν*, 2.5 h, **6d** (84%); Z = PhSMe, reflux, Cu(acac)₂, 1 h, **6e** (29%).

Scheme II



The stability of **3** is limited, and it decomposes on attempted recrystallization. However, it can be stored for at least 2 weeks at -10 °C without change. Its spectral characteristics are in agreement with the proposed structure.⁷ Ylide **3** enters into several types of reactions, three of which are shown in Scheme I and involve transfer of the bis(phenylsulfonyl)methylene moiety to several nucleophiles to give *gem*-disulfones which are otherwise not easily accessible. Thus, cycloaddition occurs with cyclohexene to yield the bicyclic disulfone **4**; C-H bond insertion is observed with benzene to give the phenylated disulfone **5**, and transylidation occurs with various S, N, and P nucleophiles which are converted into their ylides **6**. With few exceptions yields are good. These reactions have been carried out under two sets of conditions, either photolytically⁸ or thermally in the presence of catalytic amounts of Cu(acac)₂, and optimal conditions are shown in Scheme I. It is noted that ylides **6b**,⁹ **6c**,¹⁰ and **6d**¹¹ have been prepared previously in low yields.

When **3** was heated in carbon disulfide under reflux precipitation of the tetrathianic tetrasulfone **7** occurred in 30% yield. The formation of **7** probably proceeds as suggested for an analogous reaction of diphenyldiazomethane with CS₂.¹² Heating **7** in diglyme with Cu powder leads to partial desulfurization yielding the dithietanic tetrasulfone **8** (Scheme II).

The dihalobis(phenylsulfonyl)methanes **9a-c** have been obtained from **3** and *N*-halosuccinimides without any added catalyst. The

(6) Maletina, I. I.; Mironova, A. A.; Savina, T. I. *Yagupolskii, Y. L. Zh. Org. Khim.* **1979**, *15*, 2416.

(7) All new compounds have elemental analysis and spectral data (IR, ¹H NMR, MS) consistent with the assigned structures.

(8) A Philips 400-W low-pressure mercury lamp has been used. Irradiation was performed in Pyrex tubes under N₂ (λ_{max} (**3**) 277 nm, in MeOH).

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(12) Schonberg, A.; Frese, E.; Brosowski, K. H. *Chem. Ber.* **1962**, *95*, 3077.