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Generation of electrophilic phenyltellurium and phenylselenium species via photochemical electron transfer

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Abstract

Photochemical electron transfer (PET) reactions were carried out using PET sensitizer (TPT) and dichalcogenides (DPDT/DPDS). Sensitizer fluorescence quenching by dichalcogenides is found to be very efficient and it leads to the electron-transfer process. A phenylchalcogenide cation produced via dichalcogenide radical cation, was successfully utilized in cyclization reactions using variety of substrates. Thermal cyclization reaction supports the PET mechanism and also indicates the potentiality of the method in organic synthesis.

Keywords: Photochemical electron transfer; Thermal cyclization reaction; Dichalcogenides

1. Introduction

The fundamental principle in natural and artificial systems is the initial electron transfer from donor to acceptor followed by chemical transformation. Indeed Nature has mastered this basic principle in converting solar energy into a useful form of (photosynthesis) chemical energy. The same concept – photochemical electron transfer (PET) – has gained a lot of importance in the generation and study of radical ions in chemistry [1–7]. The reactive intermediates, radical ions, have received a great deal of attention during this decade [1–7]. The spectral, physical and mechanistic aspects of reactive intermediates generated via PET have increased its scope to extend and be applied to organic synthesis [8–13]. Similarly, spectroscopic studies indicated that the bond cleavage between Si–Si, Sn–Sn and Ge–Ge is feasible under PET conditions because of the low ionization energy associated within the cleavable bond [14–19]. Analogously, it is possible to study the bond cleavage in Te–Te and Se–Se compounds. We have prepared dichalcogenides, diphenyl ditelluride [20] (DPDT) and diphenyl diselenide [21,22] (DPDS) to study PET reactions. Indeed both compounds underwent PET cleavage leading to electrophilic tellurium and electrophilic selenium species. These reactive intermediates – electrophilic tellurium and selenium species – were thoroughly characterized by their chemical reactions and also indicated the possible application of this reaction in organic synthesis.

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2. Experimental

A Perkin–Elmer Lambda–2 UV–visible spectrometer was used to obtain the absorption spectra. A SPEX–Fluorolog 0.22 m fluorimeter was used for the fluorescence measurements. Mass spectra were recorded on a VG Micromass 7070H instrument. Proton nuclear resonance (H-NMR) spectra were recorded on FT–80 and or FT–200 MHz instruments in $CDCl_3$. HP-5880 Gas Chromatograph with FI Detector and with an integrator was used for GC analysis. Diphenylditelluride (DPDT) and diphenyldiselenide (DPDS) were prepared according to published procedures [20,21]. Triphenylpyrylium tetrafluoroborate was purchased from Aldrich and also prepared by adopting known procedures [23]. Other chemicals were purchased locally. All compounds were prepared according to published procedures and characterized by spectral means and procedures are given below.

2.1. Photolysis

A 450 W medium pressure mercury arc lamp was used for irradiations. $NaNO_2$ solution filter [24] was employed for >400 nm photolysis. Philips TL/03 lamps (20 W; 2 ft; ~400±20 nm) were also used for irradiations. A typical procedure involved irradiation of a 70 ml nitrogen-bubbled dry acetonitrile solution containing substrate (**1–5**, 2.5 mmole), triphenylpyrylium tetrafluoroborate (PET sensitizer, 500 mg) and diphenyldichalcogenide (DPDT or DPDS, 3.5 mmole) with pyrex filter using 450 W mercury

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arc lamp through $NaNO_2$ filter solution (>400 nm) or irradiated using Philips TL/03 lamps (~400 nm). Reaction was monitored by tlc or by GC. After 24 h of irradiation the reaction was stopped, the solvent was removed and the residue was chromatographed over silica gel to get pure cyclized products (**6–10**). The products were characterized by spectral means. The spectral data of all compounds are listed below. Stern–Volmer fluorescence-quenching studies were carried out using SPEX–Fluorimeter. A 450 W Xenon arc lamp was used for the purpose of excitation. Identical conditions were employed for all the fluorescence measurements. Slit widths were 2/2/2/2 mm, excitation wavelength was 400 nm. The fluorescence quenching of the PET sensitizer TPT (1.0×10^{-5} M) was observed at various quencher concentrations using DPDT or DPDS as quenchers. TPT fluorescence lifetime is taken as 2.1 ns [12] and quenching rates were calculated accordingly. All operations were at room temperature.

2.2. Synthesis of starting materials

2.2.1. 2–Allylphenol (**1**) preparation

Phenol was *O*-allylated using allylbromide and potassium carbonate in dry acetone to get phenylallyl ether in 85% yield [39]. The same phenylallyl ether was subjected to the Claisen rearrangement [39] to get *o*-allylphenol in about 70% yield. ^{1}H-NMR: δ 3.3 (d, J=1.25 Hz, 2H); 5.1(d, J=1.8 Hz, 2H); 5.95 (m, 1H); 6.8(m, 2H); 7.05 (d, J=1 Hz, 2H). Mass: 134 (80; M+), 115 (33.8), 105 (37), 91 (83.8), 77 (100).

2.2.2. 2-Allyl-4-methylphenol (**2**) preparation

The same procedure mentioned above was followed to synthesize 2-allyl-4-methylphenol by using *p*-cersol as a starting material [39]. ^{1}H-NMR: δ 2.25 (s, 3H); 3.3 (d, J=1.25 Hz, 2H); 5.2 (d, J=2 Hz, 2H); 6.0 (m, 1H); 6.8 (m, 4H). Mass: 148 (100; M+), 147, 133 (73), 121 (23), 105 (53), 91 (48), 77 (38).

2.2.3. 2-(3-Cyclohexenyl)phenol (**3**) preparation [40]

3-Bromocyclohexene reacted with phenol in dry acetone and in the presence of potassium carbonate to prepare phenyl-(3-cyclohexenyl) ether. Thus prepared, the phenyl-(3-cyclohexenyl) ether was subjected to the Claisen rearrangement to get **3** in good yields. ^{1}H-NMR: δ 1.45–2.2(m, 6H); 3.5 (m, 1H); 5.15 (s, 1H); 5.75 (m, 1H); 6.0 (m, 1H); 6.7 (m, 2H); 7.0 (m, 2H). Mass: 174 (100; M+), 160 (19.4), 146 (34), 131 (34), 120 (19), 115, 94 (49.2), 91 (16.4).

2.2.4. 2-Allylcyclohexanol (**4**) preparation

Cyclohexanone was treated with morpholine in benzene and refluxed to get a corresponding enamine [41]. The resulting enamine was treated with allylbromide to get 2-allylcyclohexanone. Thus prepared 2-allylcyclohexanone was reduced with sodiumborohydride to get **4** in about a 60% yield. ^{1}H-NMR: δ 0.8–2.6 (m, 11H); 5.0 (m, 2H); 5.6–5.9 (m, 1H). Mass: 140 (M+), 122 (33), 98 (34), 81 (100), 79 (30).

2.2.5. 4-Pentenoic acid (**5**) preparation

4-Bromo-1-butene reacted with Mg in dry ether and the corresponding Grignard compound was treated with solid carbon dioxide to get 4-pentenoic acid in a 75% yield. ^{1}H-NMR: δ 2.1–2.2 (m, 4H); 5.0–5.1 (m, 2H); 5.7–5.9 (m, 1H); 10.0 (broad, 1H). Mass: 100 (2.7; M+), 55 (62.5), 39 (100).

2.2.6. Spectral data of the products

Spectral data of product **6a**: ^{1}H-NMR: δ 2.95–3.2 (m, 2H); 3.3–3.5 (m, 2H); 5.0 (m, 1H); 6.8 (m, 2H); 7.1–7.4 (m, 5H); 7.8 (d, J=1.2 Hz, 2H). Mass: 340 (12; M+), 338, 207 (9), 133 (100), 105 (43), 77 (60).

Spectral data of product **6b**: ^{1}H-NMR : δ 2.9–3.1 (m, 2H); 3.3–3.4 (m, 2H); 4.8–5.0 (m, 1H); 6.6–6.8 (m, 2H); 7.0–7.1 (m, 2H); 7.2 (m, 3H); 7.5 (m, 2H); ^{13}C-NMR: δ 32.4, 35.3, 81.6, 109.5, 120.5, 124.9, 126, 127, 128.1, 129.2, 129.6, 133.1. Mass: 290 (32.8; M+), 288 (16.4), 158 (14.9), 133 (100), 119 (23), 105 (58.9), 91 (49.5), 77 (65.9).

Spectral data of product **7a**: ^{1}H-NMR: δ 2.2 (s, 3H); 2.8–3.0 (dd, J=0.235 Hz, 1.4 Hz, 1H); 3.0–3.2 (dd, J=0.235 Hz, 1.2 Hz, 1H); 3.2–3.4 (m, 2H); 4.85–5.0 (m, 1H); 6.5 (d, J=0.8 Hz, 1H); 6.8 (d, J=1.2 Hz, 2H); 7.1–7.3 (m, 3H); 7.8–7.9 (d, J=0.8 Hz, 2H). ^{13}C-NMR: δ 14.7, 20.8, 31, 36.8, 83.1, 96.1, 109, 125.4, 126.1, 127.1, 128.3, 129.1, 129.3, 138.6, 140.4, 157.2. Mass: 351 (46; M+), 207 (15), 205 (15), 147 (100), 146, 119 (40), 91 (30), 77 (40).

Spectral data of product **7b**: ^{1}H-NMR: δ 2.2 (s, 3H); 2.9–3.1 (m, 2H); 3.2–3.4 (m, 2H); 4.8–5.0 (m, 1H); 6.5 (d, J=0.8 Hz, 1H); 6.8–6.9 (m, 2H); 7.2–7.3 (m, 3H); 7.5–7.6 (m, 2H); ^{13}C-NMR: δ 20.6, 32.6, 33.4, 81.7, 108.8, 125.4, 127, 128.2, 129, 129.5, 132.8, 157. Mass: 304 (28.3; M+), 303 (10), 147 (100), 135 (16), 119 (16), 91 (16), 77 (11.6).

Spectral data of product **8a**: ^{1}H-NMR δ 1.4–2.2 (m, 6H); 3.4–3.5 (m, 1H); 3.5–3.7 (m, 1H); 4.8–4.9 (t, 1H); 6.7–6.9 (m, 2H); 7.0–7.1 (m, 2H); 7.15–7.4 (m, 3H); 7.8–7.9 (m, 2H). Mass: 378 (4.4; M+), 173 (34), 131 (100), 107 (35.8), 77 (52).

Spectral data of product **8b**: ^{1}H-NMR δ 1.2–2.2 (m, 6H); 3.35–3.6 (m, 2H); 4.6–4.8 (t, 1H); 6.7–6.8 (d, J=1.41 Hz, 1H); 7.1–7.4 (m, 6H); 7.5–7.6 (m, 2H). Mass: 330 (44; M+), 182 (6), 174 (100), 131 (77), 107 (40), 77 (44.7).

Spectral data of product **9a**: ^{1}H-NMR: δ 0.8–2.2 (m, 10H); 2.7 (m, 1H); 3.3 (m, 2H); 4.0 (m, 1H); 7.0–7.2 (m, 3H); 7.6–7.7 (m, 2H). Mass: 346 (21.6; M+), 343 (15), 206 (15), 121 (100), 95 (73.3), 77 (58.3).

Spectral data of product **9b**: ^{1}H-NMR δ 0.8–2.2 (m, 10H); 2.8–3.2 (m, 3H); 4.1–4.2 (m, 1H); 7.1 (m, 3H); 7.4 (m, 2H). Mass: 296 (28.3; M+), 157 (7.46), 125 (67.16), 107 (25.37), 95 (25), 81 (80), 77 (30).

Spectral data of product **10a**: ^{1}H-NMR δ 1.8–2.0 (m, 1H); 2.2–2.6 (m, 3H); 2.9–3.1 (m, 1H); 3.2–3.4 (m, 1H); 4.6–4.8 (m, 1H); 7.15–7.4 (m, 3H); 7.8–7.9 (m, 2H). Mass: 306 (12.5; M+), 207 (13.8), 203, 154 (34.72), 99 (6.9), 77 (100).

Spectral data of **10b**: ^{1}H-NMR δ 1.9–2.0 (m, 1H); 2.3–2.6 (m, 3H); 2.9–3.0 (m, 1H); 3.2–3.4 (m, 1H); 4.5–4.7 (m, 1H); 7.2 (m, 3H); 7.5 (m, 2H). Mass: 256 (88; M+), 171 (28.3), 149 (29.8), 99 (26.8), 85 (100), 77 (31.3).

2.2.7. Cyclization of compound **1** *using sodium persulfate [33]*

A 30 ml acetonitrile solution containing **1** (2.5 mmol; 0.337 g), sodium persulfate (2.38 g; 10 mmol) and diphenyldichalcogenide (DPDT/DPDS; 2.5 mmol) was heated to 60°C for 6 h. After cooling, the reaction mixture was taken into ether and washed with water, dried and evaporated. The residue was chromatographed over silica gel to get pure products **6a/6b**. The same procedure was adopted for other substrates also.

2.2.8. Cyclization of compound **1** *using sodiumnitrite and trifluoromethanesulfonicacid [32]*

A 30 ml acetonitrile and dichloromethane (15 ml each) solution containing $NaNO_2$ (0.131 g; 1.5 mmol) and trifluoromethanesulfonic acid (0.5 g; 3.0 mmol) was stirred at 0°C for 30 min and cooled to −10°C. DPDT or DPDS was added (3 mmol) at −10°C stirred for 20 min then cooled to −78° and added compound **1** (200 mg; 1.5 mmol), stirred to room temperature. The reaction mixture was taken into ether, washed with water, dried and evaporated. The resulting residue was chromatographed to get pure products **6a/6b**. The same procedure was adopted for other substrates also.

3. Results and discussion

DPDT [4], DPDS [21,22], the PET sensitizer triphenylpyrylium tetrafluoroborate [23] and all the starting materials (Chart) were synthesized (Section 2) accordingly. All the compounds were characterized by spectral data and are listed in Section 2. The PET sensitizer triphenylpyrylium tetrafluoroborate (TPT) is selected because of its advantages [12] over other PET sensitizers. PET reactions were carried out employing TPT as a sensitizer and using >400 nm light. The reaction mixture containing substrates (**1–5**), PET sensitizer TPT, dichalcogenide (DPDT/DPDS) regent in acetonitrile solvent (nitrogen bubbled) was irradiated using >400 nm (solution filter) [24]. The products were isolated by silica gel column chromatography and characterized by spectral means (Section 2). The results are presented in Table 1. The reaction is very smooth and control experiments revealed that light, PET sensitizer and reagents (DPDT/DPDS) are essential for cyclization reaction. The reaction is found to be very efficient when it is irradiated using >400 nm light, whereas when pyrex filter (>300 nm) was used, there was some precipitation of solid in irradiated solutions and this solid may be tellurium or selenium [25–27]. Therefore, through out all irradiations we used >400 nm wavelength light. We have carried out fluorescence quenching studies of TPT fluorescence using DPDT and DPDS as quenchers in acetonitrile solvent. Fluorescence of TPT was efficiently quenched by DPDT and DPDS indicating that their interaction with excited TPT is very efficient. Further Stern–Volmer plots constructed showed that the quenching rate is very high (>10^{10} M^{-1} s^{-1}). These studies indicate that the reagent (DPDT and DPDS) interacts efficiently with excited TPT and an electron transfer process is involved between them.

To explore the mechanism and compare the efficiency of PET-induced cyclization reaction, we carried out thermal cyclization reactions using super acid/$NaNO_2$ and persulfate reagents. The results of these studies are arranged in Table 2. Thermal cyclization affected with super acid/

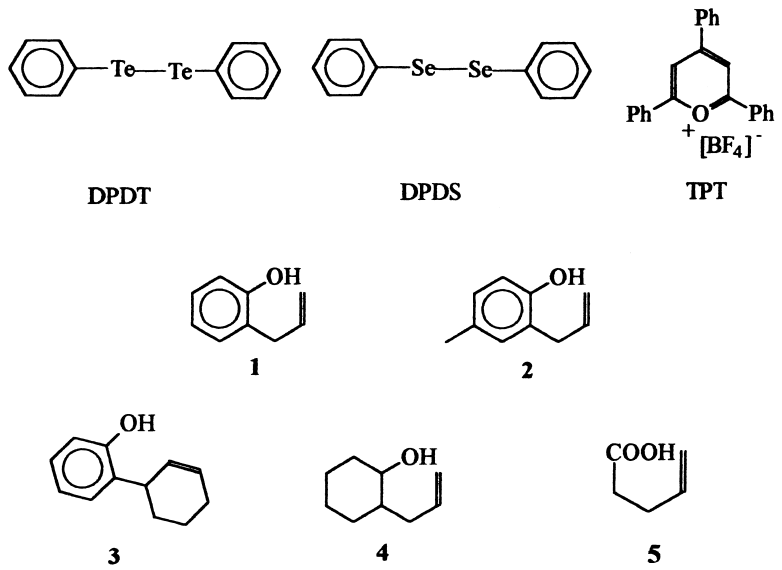
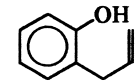
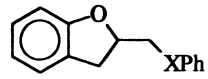
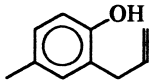
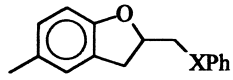
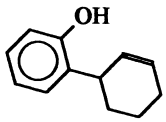
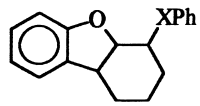
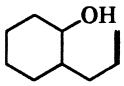
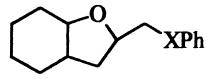
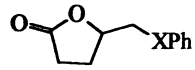


Chart.

Table 1
PET-induced cyclization using TPT as sensitizer and Ph–X–X–Ph as reagent

Substrate	Product
	
1	6a / 6b
	
2	7a / 7b
	
3	8a / 8b
	
4	9a / 9b
	
5	10a / 10b

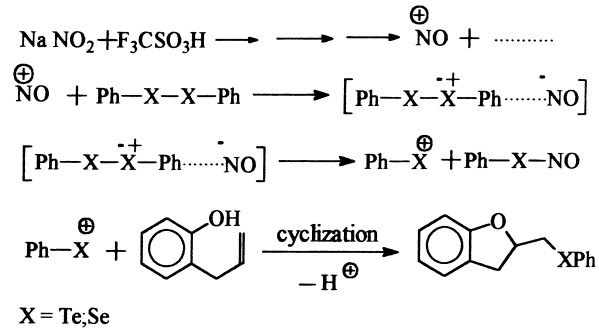
Ph–X–X–Ph is dichalcogenide (X=Te/Se); nitrogen bubbled acetonitrile solutions were irradiated using 450 W Hg lamp with solution filter (>400 nm); reaction was monitored by TLC and GC; a=telluride; b=selenide.

$NaNO_2$ system was conducted in acetonitrile/dichloromethane solvents under suitable conditions (Section 2). The reaction sequence [28–32] involved is shown in Scheme 1.

The reactive intermediate generated is the Ph–X^+ (phenyltellurium cation or phenylselenium cation) under super

Table 2
Products and yields of cyclization reaction effected by PET, super acid/$NaNO_2$ and persulfate procedures: A comparative study

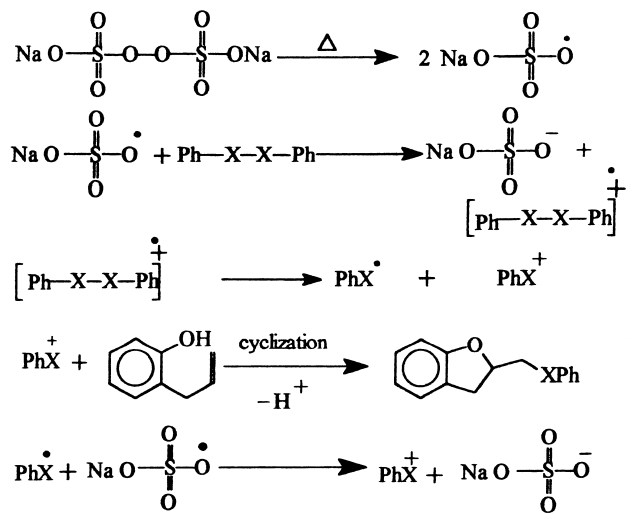
Product	PET method	Persulfate method	Super acid method
6a	66	64	80
6b	70	60	80
7a	73	63	84
7b	76	65	85
8a	65	59	76
8b	63	52	75
9a	59	48	68
9b	58	49	66
10a	59	48	72
10b	62	52	75



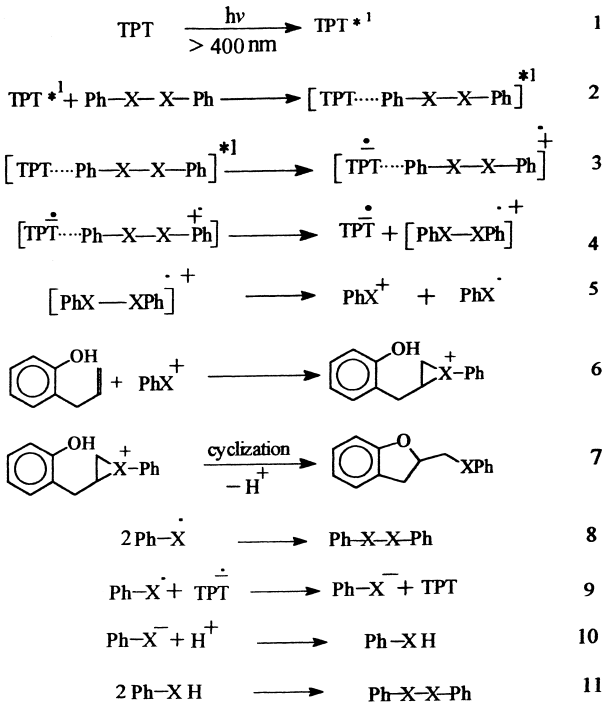
Scheme 1.

acid conditions, which is responsible for the cyclization reaction. This reaction sequence informs that a similar reactive intermediate may be involved under PET conditions. Another method of thermal reaction-induced cyclization using persulfate [33] was also conducted in acetonitrile solvent. The reaction mechanism involved is shown in Scheme 2. Heating the persulfate gives a radical species which efficiently interacts with dichalcogenide reagent (DPDT/DPDS) and generates a radical cation species of dichalcogenide involving electron transfer between dichalcogenide and sulfate radical. Thus the generated radical cation of dichalcogenide collapses to give a chalcogenide radical and cation. The chalcogenide cation is shown to induce cyclization reactions.

The mechanism of PET-induced cyclization is depicted in Scheme 3. The primary step is light absorption by the sensitizer TPT. The excited TPT^{*1} interacts with the dichalcogenide leading to complex formation. Control experiments indicated that light, sensitizer and reagent dichalcogenide are essential and this supports the two steps shown in Scheme 3 of the mechanism. Further, fluorescence-quenching studies indicate that dichalcogenide is involved in interacting efficiently with the TPT^{*1} excited



Scheme 2.



Scheme 3.

state. The fluorescence-quenching process will lead to complex formation (step 2) and the complex between excited TPT and dichalcogenide thus formed will be involved in the electron transfer process leading to dichalcogenide radical cation (steps 3 and 4). The radical cation of dichalcogenide thus formed dissociates to give phenylchalcogenide cation (step 5). The electron transfer process between excited TPT and dichalcogenide is feasible based on their reduction and oxidation potential [34–36]. The phenyl chalcogenide cation thus formed will react with substrate, allylphenol to give cyclized product, benzofuran derivative **6** (steps 6 and 7). The formation of benzofuran derivative is also consistent with the synthesis of phenylseleno-ether [37,38] via the involvement of phenylchalcogenide cation. The two thermal cyclization processes carried out (**1 and 2**) also indicate that PET reaction between TPT and dichalcogenide generates the same reactive intermediate, phenylchalcogenide cation via dichalcogenide cation radical. The other steps (steps 8–11) shown in Scheme 3 explain the formation of dichalcogenide. We have looked into the potentiality of this method suitable for organic synthesis by conducting thermal cyclization reactions (**1 and 2**) and comparing the yields obtained. Indeed, this PET cyclization procedure gives good yields (Table 2) that are comparable to the thermal methods. This method has an advantage compared to the method using the toxic phenylchalcono-halide procedure [27]. The synthesized phenylchalcogeno-ether has the advantage of further functional group modifications using chalcogenide moiety.

In conclusion it is shown that the PET process occurs between excited sensitizer (TPT) and dichalcogenide giving rise to phenyl chalcogenide cation via dichalcogenide cation radical and this phenylchalcogenide-induced cyclization in substrates present in the medium leads to cyclized phenylchalcogenide derivatives. The reaction is smooth and has synthetic potentiality.

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