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RESEARCH ARTICLE

Chemoselective Reduction of Organic Sulphoxides, Selenoxides and Telluoxides with Magnesium in Methanolic Media and its Mechanistic Insights

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Abstract: Mercuric chloride catalysed, a versatile, convenient and practical procedure has been developed for the reduction of chalcogenide monoxides, including sulphoxides, selenoxides and telluroxides into their respective chalcogenides. The reaction is performed using magnesium metal as the reducing agent in methanol at ambient temperature. The procedure demonstrates wide functional group tolerance and proceed under straight forward reaction conditions, making it well fitted for synthetic applications. Notably, sulphoxides required a relatively higher amount of magnesium compared to their selenium and tellurium congeners, indicating a clear difference in reactivity among the chalcogen series. The reactions proceed rapidly and proposed to be proceeding *via* Single Electron Transfer (SET) mechanism by magnesium. The methodology shows high chemselectivity, wide functional group tolerance under the reaction conditions and no instances of deselenization, detellurization, or desulphurization product were found, signifying the stability of chalcogenide products.

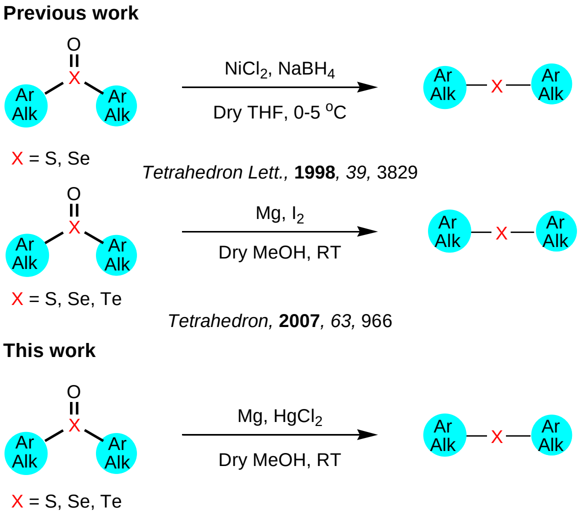
Keywords: *Chalcogenide,* chemselective reduction, *sulphoxides, selenoxides, telluoxides, single electron transfer (SET)*

1. Introduction

Over the past few decades, there has been huge increase in the number of practical synthetic processes involving metals, metal ions and organometallic compounds. Victor Grignard's discovery alkyl and aryl halides react with magnesium metal to produce homogeneous solutions marked the beginning of use of organometallic reagents in organic synthesis. Since then, these 'Grignard reagents', which are highly reactive carbon nucleophiles, have continued to be extremely helpful synthetic reagents.[1-5] Subsequently, magnesium has been reported to bring about a large number of organic transformations[6] predominantly in ethereal solvents. The use of magnesium in methanol as a synthetically useful reagent has shown tremendous potential recently.[7-19] Magnesium acts as a source of electrons both in ethereal solvents and methanol. Due to its low cost, ease of use, it is a preferred reagent over other reducing agents. Magnesium can be used in a variety of ways including the creation of organometallic complexes and the usage of fixed magnesium salts. Also, its reactions may be performed in protic as well as aprotic solvents. All its reactions require initiators like iodine and mercuric chloride. Mercuric chloride has been used in Julia olefination[11] and also as an initiator in magnesium catalysed reactions. Even though it has only been applied to a few reactions, more research can be done on it, particularly for reactions that call for extreme conditions.

Our research group has been investigating magnesium methanol's potential as a reductant and on study of chalcogenides over the past few decades.[20-28] We have recently reported the reduction of chalcogenide dioxide to chalcogenide using magnesium methanol using catalytic amount of mercuric chloride.[28] In continuation to our previous work, we extended our study with chalcogenide monoxide and Mg/ MeOH. Magnesium mediated reactions require an initiator, several initiators like $ZnCl_2$, $FeCl_3$, $CoCl_2$, I_2, $HgCl_2$, $BF_3.Et_2O$ were tried but completion of reaction could be achieved with $HgCl_2$ and Iodine only. Higher molar ratios of substrate to reagent were required when using iodine as initiator.[27] This led us to use mercuric chloride as initiator to further investigate the reaction and its mechanism.

2. Results and Discussion



Scheme 1. *Reduction of chalcogenide monoxides*

With this objective in mind, diphenyl sulphoxide (**1a**) was selected as a model substrate. To optimize the reaction

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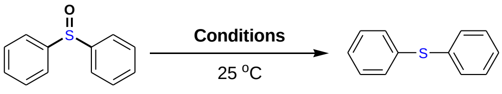
conditions, various substrate to magnesium molar ratios were tested using various initiators and solvents (Table 1). As a continuation of our previous work on the reduction of organic sulphones, selenones and tellurones,[28] the reaction progress was monitored by Thin Layer Chromatography (TLC), completion was determined by the complete consumption of the starting material.

Table 1. *Model reaction optimization*[a]



1a **2a**

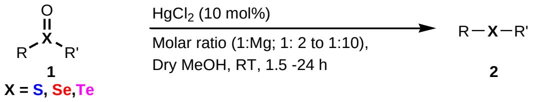
Entry	Catalyst (10 mol %)	Molar Ratio **1a**: Mg	Solvent	Time (h)	% Yield (**2a**)
1.	HgCl$_2$	1:1	MeOH	24	Traces
2.	HgCl$_2$	1:3	MeOH	24	Traces
3.	HgCl$_2$	1:4	MeOH	24	Traces
4.	**HgCl$_2$**	**1:5**	**MeOH**	**1.5**	**86**
5.	ZnCl$_2$	1:5	MeOH	24	Traces
6.	FeCl$_3$	1:5	MeOH	24	Traces
7.	CoCl$_2$	1:5	MeOH	24	Traces
8.	I$_2$	1:10	MeOH	3	76
9.	BF$_3$.Et$_2$O^c	1:5	MeOH	24	NR^b
10.	HgCl$_2$	1:5	THF	24	NR^b
11.	HgCl$_2$	1:5	EtOH^d	24	NR^b
12.	HgCl$_2$	1:5	MeOH^d	24	NR^b
13.	-^{e}	1:5	MeOH	24	NR^b

[a]Reaction condition: **1a** (0.49 mmol), HgCl$_2$ (10 mol%), anhydrous MeOH. [b]No reaction. [c]100 mol % of BF$_3$.Et$_2$O was used. [d]Regular laboratory alcohol was used without drying. [e]Reaction without any initiator.

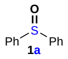
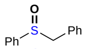
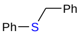
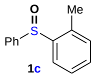
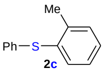
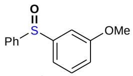
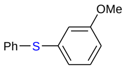
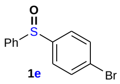
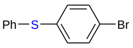
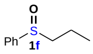
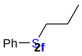
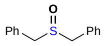
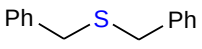
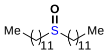
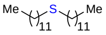
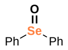
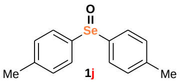
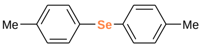
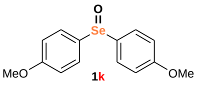
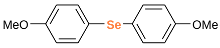
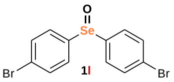
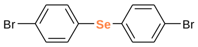
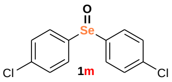
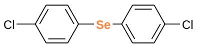
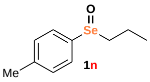
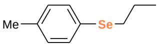
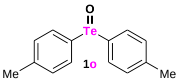
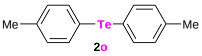
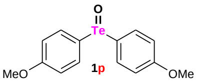
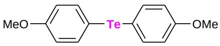
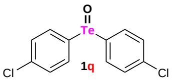
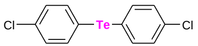
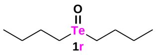
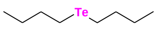
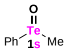
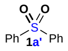
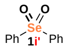
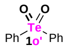
Firstly, to create the optimal molar ratios of substrate to Mg, conducted a reaction of diphenyl sulphoxide (**1a**) with Mg in 1:1 molar ratio. The reaction was incomplete and traces of corresponding to diphenylsulphide (**2a**) was observed under this condition (Table 1, Entry 1). Upon increasing the Mg concentration, **1a**:Mg molar ratios up to 1:4, this improved product **2a** formation slightly, but the reaction was incomplete even when continued for 24 h (Table 1, Entries 2 and 3). Notably, the product **2a** was exclusively formed with an isolated yield 86% when the molar ratio of **1a**:Mg was increased to 1:5 in just 1.5 hours (Table 1, Entry 4; details are in experimental section). A greater molar ratio of magnesium to substrate is typically required because of the competing reaction between magnesium and anhydrous methanol which occurs significantly faster, resulting in the production of magnesium methoxide whereas the product formation was not obtained while moist MeOH or EtOH (regular laboratory used alcohol, Table 1, Entries 11 and 12).

Subsequently, using the same molar ratio (**1a**: Mg = 1:5) the reaction was screened with different initiators like ZnCl$_2$, FeCl$_3$, CoCl$_2$, and I$_2$. Reactions using ZnCl$_2$, FeCl$_3$, and CoCl$_2$ remained incomplete and only trace amounts of product **2a** was observed even with prolonged the reaction time (Table 1, Entries 5-7). But I$_2$ performed better, affording similar yield when used comparatively higher molar concentration (**1a**: Mg = 1:10) within 3 hours (Table 1, Entry 8). However, no product was observed while BF$_3$.Et$_2$O was used, even in stoichiometric amounts and prolong reaction time (Table 1, Entry 8). In absence of any initiator, reaction did not proceed at all and the starting material was recovered as such (Table 1, Entry 13). Among the tested conditions, the combination of anhydrous MeOH and mercuric chloride (10 mol %) seemed as the most effective condition (Table 1, Entry 4). This observation is aligned with the standard reduction potentials of the initiators: Mg^{2+} ions possess a higher positive reduction potential, indicating that activating magnesium with mercuric ions is significantly faster via *in situ* generation of a catalytic magnesium amalgam. This amalgamation significantly improving activity of magnesium. While iodine also triggers magnesium, its effect is comparatively milder, requiring higher

Table 2. *Substrate Scope*[a]



1 **2**

Entry	Substrate (S)	Moar ratio (S:Mg)	Time (h)	Product	Yield (%)^b
1.	 **1a**	1:5	6	Ph—S—Ph **2a**	**86**
2.	 **1b**	1:5	2.5	 **2b**	**78**
3.	 **1c**	1:5	6	 **2c**	**90**
4.	 **1d**	1:5	2	 **2d**	**83**
5.	 **1e**	1:5	1.5	 **2e**	**79**
6.	 **1f**	1:10	12	 **2f**	**84**
7.	 **1g**	1:10	24	 **2g**	**NR^c**
8.	 **1h**	1:10	24	 **2g**	**NR^c**
9.	 **1i**	1:2	1.5	Ph—Se-Ph **2i**	**76**
10.	 **1j**	1:2	1.5	 **2j**	**81**
11.	 **1k**	1:2	2	 **2k**	**85**
12.	 **1l**	1:2	3	 **2l**	**88**
13.	 **1m**	1:2	4	 **2m**	**83**
14.	 **1n**	1:2	6	 **2n**	**88**
15.	 **1o**	1:2	6	 **2o**	**76**
16.	 **1p**	1:2	6	 **2p**	**90**
17.	 **1q**	1:2	6	 **2q**	**86**
18.	 **1r**	1:2	5	 **2r**	**65**
19.	 **1s**	1:2	5	Ph—Te—Me **2s**	**71**
20.	**1a**	1:5^d	24	**2a**	**NR^c**
21.	**1a**	1:5^e	24	**2a**	**NR^c**
22.	**1a**	1:5^f	24	**2a**	**NR^c**
23.	 **1a'**	1:10	1.5	**2a**	**87**
24.	 **1i'**	1:4	1.5	Ph—Se-Ph **2i**	**91**
25.	 **1o'**	1:4	1.5	Ph—Te-Ph **2o**	**77**

[a]Reaction condition: **1** (0.49 mmol), HgCl$_2$ (10 mol%), Substrate (**1**): Mg molar ratio mention in table, anhydrous MeOH. [b]Isolated Yield. [c]No Reaction. Reaction preformed in [d]Mg(OMe)$_2$, [e]m-dinitrobenzene, [f]by bubbling oxygen.

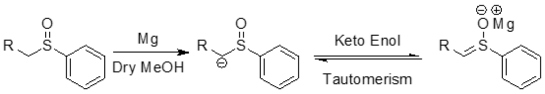
magnesium loading for parallel competence. In contrast, Zn^{2+} and Fe^{3+} with lower standard reduction potentials, are thermodynamically unqualified of oxidizing Mg and therefore fail to activate its surface effectively.

M^{n+}(M)	Zn^{2+} (Zn) <	Fe^{3+} (Fe) <	Co^{2+}(Co) <	I_2 (I^-) <	Hg^{2+} (Hg)
E° (in Volts)	-0.76	-0.36	-0.28	0.54	0.85

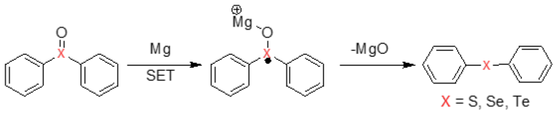
Encouraged by the efficiency of the model reaction, we proceed to examine the substrate scope under the optimized conditions (10 mol% $HgCl_2$; substrate: Mg molar Ratio = 1:5; MeOH; 25 °C). A variety of substituted aryl sulfoxides was tested revealing good to excellent yield (details in Table 2). The sulphoxides, namely, benzyl phenyl sulphoxide (**1b**), phenyl o-tolyl sulphoxide (**1c**), m-anisyl phenyl sulphoxide (**1d**), p-bromophenyl phenyl sulphoxide (**1e**) and phenyl n-propyl sulphoxide (**1f**) were deoxygenated within the range of 1.5-6.0 h giving very good yields of respective sulphides (**2b-f**) (Table 2, Entries 2-6). Phenyl n-propyl sulphoxide (**If**) required higher molar ratio (1:10) to undergo complete deoxygenation (Entry 5). Reaction of unactivated sulphoxide like dibenzyl sulphoxide (**1g**) and di(*n*-dodecyl)sulphoxide (**1h**) did not proceed at all and starting materials were recovered unchanged even after used of excess molar ratios of substrate to (Entries 7-8). This could be due to the formation of α-carbanions (the hydrogens are acidic) with magnesium methoxide, formed initially, which prevents further reaction (Scheme 2).

The sulphoxide with aryl groups directly bonded to sulphur might stabilized intermediate, but in dibenzyl sulphoxide lacks direct conjugation with the sulphur and the phenyl rings are one carbon away. Neither the benzyl group nor the dodecyl group activates the Sulphur towards nucleophilic or reductive attack. Moreover, the LUMO of sulphoxide in such cases relatively high and hence electron transfer is less favorable.

In view of the successful deoxygenation of various sulphoxides, we attempted to carry out this reduction for various selenoxide (Entries 9-14) and telluroxide (Entries 15-19) in different molar concentration under same reaction conditions (Table 2, Entries 9-19). Interestingly, di-*n*-butyltelluride (**1r**) responded the reduction. This might be Te=O bond is much weaker than S=O bond. The better *pπ-dπ* overlap in the case of sulphoxide took place and hence hard to break during reduction. Te is more electropositive and have and have realtively lower ionization energy than sulphur. The more polarizable making electron easier. The central atom Te is more reactive due to higher atomic size and have available



Scheme 2. *Possible reason for the unreactivity of unactivated sulphoxides*



Scheme 3. *Possible Mechanism of reduction of chalcogenide monoxides via SET mechanism*

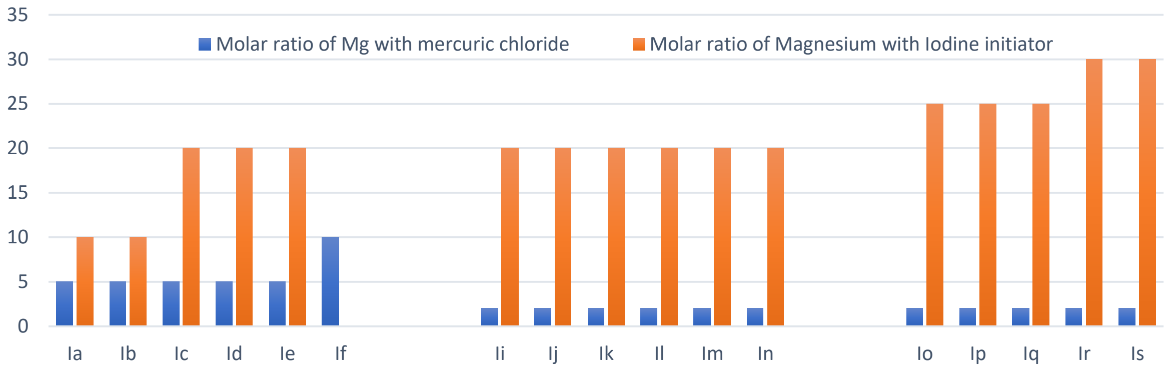


Figure 2. *Comparison of molar ratios Mg with mercuric chloride and iodine initiator*

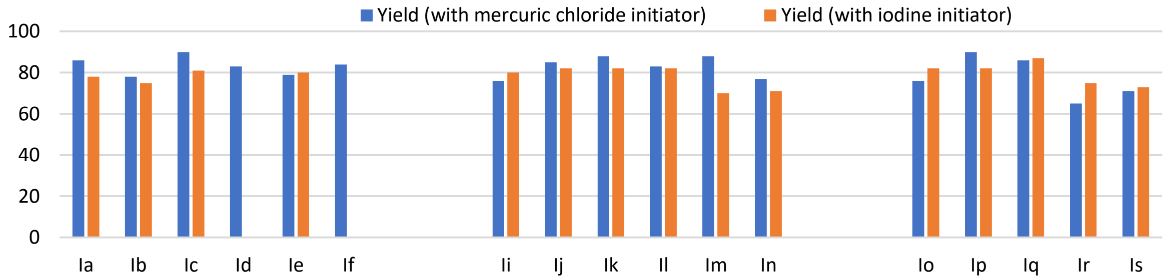


Figure 3. *Comparison of yields with different initiators*

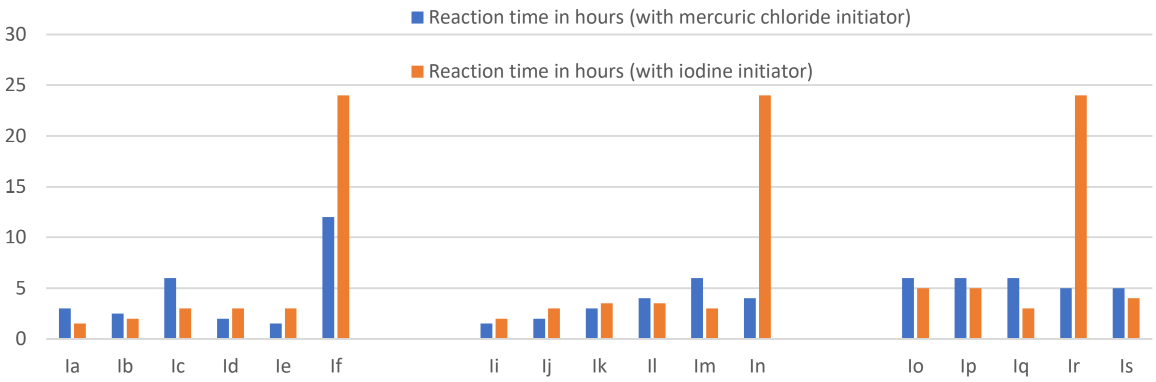


Figure 4. *Comparison of reaction time with mercuric chloride and iodine as initiator*

d orbitals. In all three different sulphoxides, selenoxides and telluroxide, no desulphurized, deselenized or detellurized products were produced in any of the cases and the reactions were chemoselective, other functional groups attached to the aryl unit like $–CH_3$, $–OCH_3$, –Cl and –Br remained unaffected under these conditions.

No product was obtained when conducting reaction of diphenyl sulfoxide with pre-formed magnesium methoxide in methanol (Entry 20), this effectively ruled out the possibility of Meerwein-Ponndorf-Verley reduction type hydride transfer mechanism in the reduction process. To understand the mechanistic insight further, the standard reaction was carried out in presence of known radical quencher like m-dinitrobenzene and by bubbling oxygen gas through the reaction mixture. The product formation was completely stopped in both the cases, which suggested the reaction proceed through SET pathway. As, magnesium has been known as a single electron transfer reagent, here we suggest that the reduction proceeds via initial SET from **HOMO** of magnesium to the **LUMO** of the substrate R_2XO (X= S, Se, Te) triggered by the presence of mercuric ion (Scheme 3, Figure 1). This electron transfer leads to the formation of a radical anion intermediate, which undergoes an additional electron transfer followed by loss of magnesium dioxide.

Mercuric chloride is a better and a promising initiator when compared to previously reported work employing iodine initiator in terms of reaction time and molar ratio of substrate to magnesium (Figure 2, 3 & 4).[27] A considerable amount of product was obtained with some of the substrate with mercuric chloride, where iodine fails to produce any product even with higher molar ratios and after 24 h. Similar results have also been obtained with chalcogenide dioxides using magnesium methanol in catalytic amount of mercuric chloride (Entry 23-25).[28] Because of the broader range of application of mercuric chloride, we can argue that this piece of work is an improvisation of the previously published work.

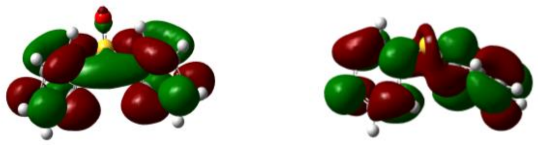

(A) **(B)**

Figure 1. *Graphical depiction of LUMO of diphenylsulphoxide (A), diphenylsulphide (B) using Gaussian 09 with B3LYP at 6-31G(d,p) basis SET.*[35]

Conclusion

In conclusion, magnesium in methanol when activated by catalytic amount of mercuric chloride provides a convenient method for the reduction of chalcogenide monoxides to the respective chalcogenides in moderate to good yields at ambient temperature. The methodology is also applicable for the reduction of chalcogenides dioxides using double the molar ratios of substrate to magnesium. Importantly, no evidence of desulphurization, deselenization or detellurization was observed in any case. Furthermore, the halogens attached to the aryl ring remained unaffected under these conditions. The mechanistic studies suggest that the reactions proceed via single electron transfer (SET) mechanism from HOMO of magnesium to LUMO of the chalcogenide oxides. The reaction methodology holds potential synthetic use in multistep organic reactions, owing to its facile, chemoselective and high yield. Although, efforts are presently ongoing to mitigate the limited reactivity of this methodology towards unactivated sulphoxide like dibenzyl sulphoxide and di(n-dodecyl)sulphoxide.

Experimental Section

General Information: The products were identified using lab technique like m.p., mixed m.p., and by other spectroscopic techniques like FTIR and NMR. Methanol used was dried by reported methods[29] and magnesium turnings were made silvery white by washing with hydrochloric acid (1%) followed by washing with water and acetone before use in any reaction. The computational calculations presented in this paper are carried out using Gaussian 09 package.[35]

Preparation of Chalcogenide Monoxides: Diphenyl sulphoxide was obtained commercially and used as such and other sulphoxides were prepared using literature procedure.[30] All selenoxide were prepared from selenide according to literature methods where initially selenides were brominated followed by alkaline hydrolysis to get the corresponding selenoxide.[31,32,33] Telluroxides were prepared by a similar route as reported literature.[34] The confirmation of chalcogenide monoxides were constant with literature reports.

General Procedure for Chemoselective Reduction of Chalcogenide Monoxides:

For sulphoxides: In 50 mL round bottomed flask fitted with an air condenser along with a calcium chloride guard tube, diphenyl sulphoxide (**1a**; 0.1 g, 0.49 mmol), anhydrous methanol (10 mL), Mg (0.0594 g, 2.47 mmol) and $HgCl_2$ (10 mol%) were taken and the reaction mixture was stirred vigorously on a magnetic stirrer at room temperature. After 15 min, the reaction mixture turned grey-black and turbid. The progress of the reaction was monitored by TLC (15 % ethyl acetate in petroleum ether). After complete disappeared of starting material on TLC, the reaction was quenched using minimum amount of saturated ammonium chloride solution until magnesium methoxide completely dissolves. It was then extracted with dichloromethane (3 x 10 mL) and washed with water. A tiny droplet of elemental mercury observed at the interface of aqueous and organic layer is separated and having been maintained in isolation. From the combined dried extract, the solvent was removed using standard rotavapour, a pale-yellow liquid obtained was vacuum dried and identified to be diphenyl sulphide (**2a**, 0.079 g, 86 %) by co-TLC with the authentic sample as recently reported. No further column purification was need. The product was confirmed by FT-IR and ^{1}H NMR which was superimposable and aligned with earlier reported data.

For selenoxides: In 50 mL round bottomed flask fitted with an air condenser along with a calcium chloride guard tube, diphenyl selenoxide (**1i**; 0.1 g, 0.40 mmol), anhydrous methanol (10 mL), Mg (0.02 g, 0.80 mmol) and HgCl$_2$ (10 mol %) was placed and the reaction mixture was stirred on a magnetic stirrer. The progress of the reaction was monitored by TLC (eluent 30% ethyl acetate in petroleum ether). The reaction was completed within 1.5 h and a new spot corresponding to diphenyl selenide by co-TLC with the authentic sample, was observed on TLC. Then the reaction mixture was quenched with minimum amount of saturated NH$_4$Cl solution until magnesium methoxide dissolved completely. The mixture was extracted with dichloromethane (3 x 10 mL) and washed water (2 x 10 mL). A tiny droplet of elemental mercury observed at the interface of aqueous and organic layer is separated and having been maintained in isolation. The combined extracts were dried over anhydrous Na$_2$SO$_4$, filtered through a cotton pad and concentrated under reduced pressure. After vacuum drying, a yellow liquid was obtained, which was identified to be diphenyl selenide (**2i**; 0.07 g, 76 %) by its superimposable IR spectra and NMR analyses with authentic sample.

For telluroxides: In 50 mL round bottomed flask fitted with an air condenser along with a calcium chloride guard tube, di(p-tolyl)telluroxide (**1o**; 0.24 g, 0.72 mmol), Mg (0.0360 g, 1.3 mmol) and 10 mL of dry methanol was taken and mounted over a magnetic stirrer. Then mercuric chloride (10 mol %) was added to the reaction mixture and was stirred vigorously. After 15 min, the solution turned yellow and then black. The progress of the reaction was monitored by TLC using 30% ethyl acetate in petroleum ether as eluent. The complete disappearance of the starting material was observed after 6 h and corresponding di(*p*-tolyl)telluride (**2o**; R$_f$ ~ 0.95) was observed on TLC and product was aligned in TLC with authentic sample. Then reaction was quenched with minimum amount of saturated NH$_4$Cl solution until the magnesium methoxide dissolved completely and was extracted with dichloromethane (3 x 10 mL) and washed with water (2 x 10 mL). A tiny droplet of elemental mercury observed at the interface of aqueous and organic layer is separated and having been maintained in isolation. The combined organic extracts were dried over anhydrous Na$_2$SO$_4$, filtered and the solvent was removed under reduced pressure to give a yellow solid. The solid thus obtained was identified to be di(p-tolyl)telluride (**2o**; 0.1824 g, 76 %) by m.p. 64°C (lit. m.p. 67°C),[25] mixed m.p., superimposable IR spectra and NMR analyses.

Computational details: All the computational calculations presented in this article are carried out using Gaussian 09 package.[35] Density functional theory (DFT) hybrid Functional B3LYP and 6-31G basis set with d and p polarization function is used for all calculations. The molecular orbital (HOMO/LUMO) isosurfaces are drawn using Gaussview software at an isovalue of 0.05.[36]

Supporting Information

Additional information on starting materials and products characterization are available in our earlier research article and in the references cited at appropriate places.[25,26] Details regarding theoretical calculations, predicted by Gaussian 09[35] with B3LYP/6-31G (d,p) level of theory is available Supporting Information at https://insuf.org/icms/icms.2025.02165SI.pdf

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Author Contribution Declaration

Kiran synthesized all the starting materials, performed all the reactions, data analysis, writing initial and final draft. Khurana conceptualized and designed the research, supervision.

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Data Availability Declaration

All the compounds are known and their data is available in our recent article and the references cited therein properly.

Declaration of competing interest

The author declares no known competing financial interests.

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