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SHORT COMMUNICATION

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Reaction of arylhydrazines with diaryl ditellurides in the air: Insight into bimolecular homolytic substitution on tellurium via Aryl–Te bond cleavage

Yuki Yamamoto | **Fumiya Sato** | **Shintaro Kodama** | **Akihiro Nomoto** | **Akiya Ogawa**

Department of Applied Chemistry, Graduate School of Engineering, Osaka Prefecture University, Sakai, Osaka, Japan

Correspondence
Akiya Ogawa and Shintaro Kodama, Department of Applied Chemistry, Graduate School of Engineering, Osaka Prefecture University, Sakai, Osaka, Japan.
Emails: ogawa@chem.osakafu-u.ac.jp and s-kodama@chem.osakafu-u.ac.jp

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Abstract

The reactivity of diaryl ditelluride and diaryl telluride toward aryl radicals was studied in detail. Diphenyl ditelluride underwent a bimolecular homolytic substitution (S_H2) reaction with a phenyl radical generated from phenylhydrazine in the air, to afford diphenyl telluride in excellent yield. Based on this diphenyl telluride synthesis, a one-pot synthesis of unsymmetrical diaryl tellurides was developed by the S_H2 reaction of in situ generated diphenyl telluride with arylhydrazines in the air. The selectivity of mono-/di-substitution and the reactivity of arylhydrazines depend on the nature of the substituents on the arylhydrazines, that is, electron-donating or -withdrawing group.

1 | INTRODUCTION

Organotellurium compounds have served as important synthetic intermediates,[1–9] and especially, diorganyl tellurides have been used as carbon nucleophile sources through a tellurium-lithium exchange reaction.[10–12] In addition, a relatively weaker carbon–tellurium bond (48 kcal/mol)[13] makes organotellurium compounds fascinating in radical chemistry.[14] For example, organotelluro groups can be easily reduced with tin hydrides to be replaced by hydrogen.[15] In radical chemistry, diorganyl ditellurides (R^1TeTeR^1) act as excellent carbon radical trapping agents (eq. 1).[16] Diorganyl tellurides (R^1TeR^2) have been used for a bimolecular homolytic substitution (S_H2) reaction with a carbon radical to generate a new carbon radical (eq. 2).[17–20]

$$R^{\cdot} + R^1TeTeR^1 \rightarrow R^1TeR + R^1Te^{\cdot} \tag{1}$$

$$R^{\cdot} + R^1TeR^2 \rightarrow R^1TeR + R^{2\cdot} \tag{2}$$

The S_H2 reaction at the tellurium atom has been applied to the synthetic reactions such as carbotelluration of alkynes,[21–23] group-transfer imidoylation,[24] and living radical polymerization.[25–28] These reactions are generally induced by a radical initiator such as AIBN and V-40, and bond cleavage of unsymmetrical tellurides, R^1TeR^2 (R^1 = aryl, R^2 = alkyl, acyl) occurs typically on an alkyl $C_{sp}{}^3$–Te bond or an acyl $C_{sp}{}^2$–Te bond rather than an aromatic $C_{sp}{}^2$–Te bond to generate new alkyl or acyl radicals. In contrast, the generation of aryl radicals from symmetrical and unsymmetrical diaryl tellurides has largely been unexplored.

Recently, we have developed two types of aryl radical generation methods. One is a photoinduced decomposition of triarylbismuthines[29] and the other is an air-induced decomposition of arylhydrazines.[30–34] In the latter method, arylhydrazine hydrochlorides can be employed as an aryl radical source, and by using this method, we successfully developed the arylation of aminoheterocycles and aromatic diamines,[30,31] the synthesis of unsymmetrical diaryl sulfides

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Dedicated to 82nd birthday of Professor Naomichi Furukawa.

and selenides,[32,33] and the aryl iodide synthesis by the trapping of aryl radicals with I_2.[34] On the basis of these studies, we have investigated the reaction of arylhydrazine hydrochlorides with diaryl ditellurides to synthesize symmetrical and unsymmetrical diaryl tellurides. Surprisingly, we have found that in situ generated diaryl tellurides (Ar^1TeAr^2) can also undergo an S_H2 reaction with aryl radicals (generated from arylhydrazines) at the tellurium atom to generate $Ar^1\cdot$ or $Ar^2\cdot$. Herein, we report an S_H2 reaction of diaryl ditellurides with arylhydrazines in the air to afford unsymmetrical or symmetrical diaryl tellurides.

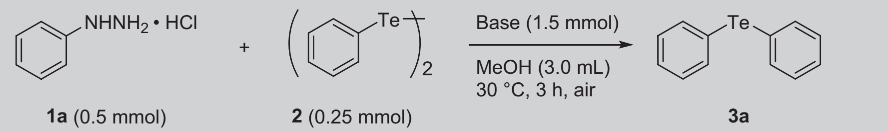
2 | RESULTS AND DISCUSSION

Initially, we examined the synthesis of symmetrical diaryl tellurides such as diphenyl telluride (**3a**) by the model reaction of phenylhydrazine hydrochloride (**1a**) with diphenyl ditelluride (**2**) in the presence of base in the air (Table 1). As **1a** decomposes in the presence of base in the air to generate a phenyl radical, the influence of base was firstly investigated. When an inorganic base was used for this reaction, **3a** was obtained in moderate yield (entries 1–3). $NaHCO_3$ as a weak base did not work well for this diphenyl telluride synthesis (entry 4). When NaOH as a strong base was used, **3a** was obtained in 82% yield (entry 5). Interestingly, when Et_3N was used, the desired reaction proceeded smoothly to afford **3a** in good yield (83%, entry 6). The use of a bulky base such as *N,N*-diisopropylethylamine (DIPEA) resulted in a similar yield of **3a** (entry 6 vs entry 7). Slightly decrease of the temperature (25°C) somewhat improved the yield of **3a** (87%, entry 8). This reaction did not proceed in the absence of a base or air (entries 9–10). When a slightly excess amount of **1a** (0.6 mmol) was used, **3a** was obtained in excellent yield (93% NMR yield; 84% isolated yield, entry 11).

Keeping the optimized reaction conditions in mind, we next investigated to synthesize unsymmetrical diaryl tellurides from 4-methoxyphenylhydrazine hydrochloride (**1b**) and diphenyl ditelluride (**2**) (Scheme 1). Fortunately, the desired unsymmetrical telluride **4a** was obtained as the major product in 42% yield, along with symmetrical tellurides **3b** (18%) and **3a** (8%). The formation of **3b** and **3a** strongly suggests that the bimolecular homolytic substitution (S_H2) reaction between the formed diaryl telluride (**4a**) and the aryl radical (p-MeO-$C_6H_4\bullet$) might take place competitively: p-MeO-$C_6H_4\bullet$ attacks **4a** to form **3b** and Ph•, which then attacks **2** to form **3a**.

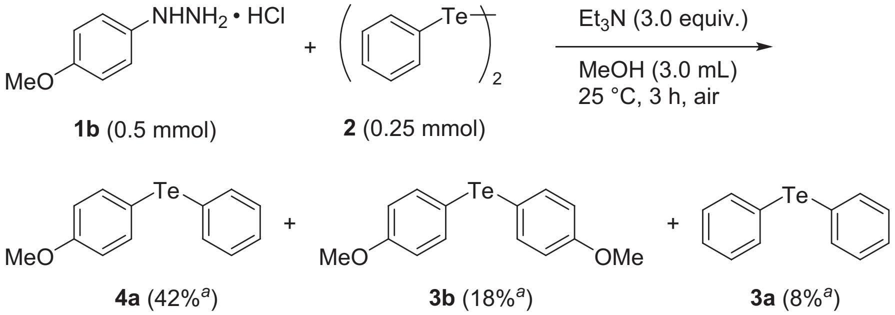
Table 2 represents the optimization of the synthesis of unsymmetrical diaryl tellurides. When the reaction was conducted in refluxing MeOH, both the yield and product selectivity of the desired product **4a** were improved (entries 1-3). The results encouraged us to examine the influence of solvents to this unsymmetrical diaryl telluride synthesis. A variety of alcohols were examined (entries 3–7), and the use of ^{i}PrOH led to the formation of unsymmetrical telluride **4a** in 44% yield (75% selectivity). When benzene and toluene as nonpolar solvents were used, **4a** was obtained in 35% and 23% yields, respectively (entries 8–9). The reaction

TABLE 1 Optimization of reaction conditions for the synthesis of diphenyl telluride **(3a)**



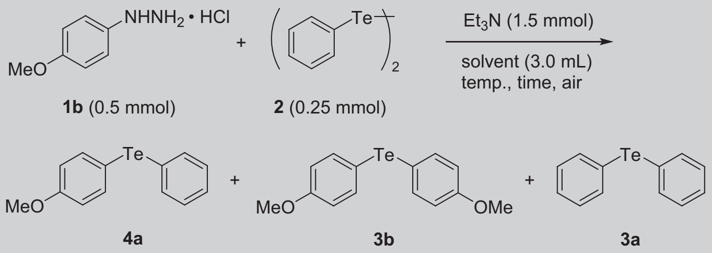
Entry	Base	Yield (%)[a]
1	$LiOH \cdot H_2O$	63
2	Cs_2CO_3	66
3	K_2CO_3	60
4	$NaHCO_3$	15
5	NaOH	82
6	Et_3N	83
7	DIPEA	85
8[b]	Et_3N	87
9[b]	None	N.D.
10[b,c]	Et_3N	3
11[b,d]	Et_3N	93 (84)

[a] The yield was calculated based on **2** and determined by ^{1}H NMR using 1,3,5-trioxane as an internal standard (isolated yield).
[b] At 25°C.
[c] Under argon atmosphere.
[d] Hydrazine hydrochloride **1a** (0.6 mmol) was used.



SCHEME 1 Reaction of 4-methoxyphenylhydrazine hydrochloride with diaryl ditelluride. ^{a}The yield was caluculated based on **2** and determined by ^{1}H NMR (1,3,5-trioxane, $CDCl_3$)

TABLE 2 Optimization of reaction conditions for the synthesis of unsummetrical diaryl tellurides

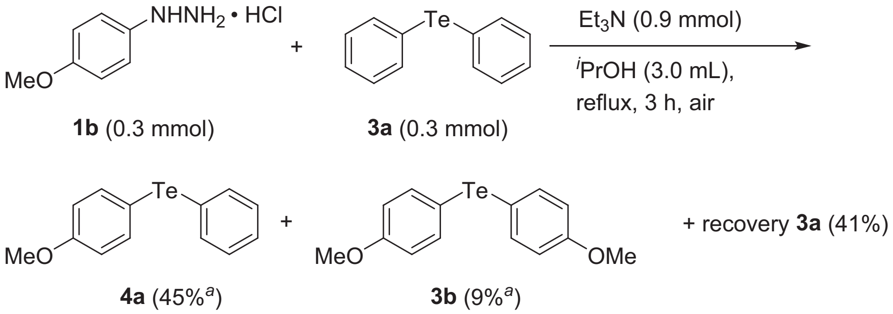


Entry	Solvent	Temp. (°C)	1b (mmol)	Time (h)	Yield (%)^a			Selectivity
					4a	3b	3a	4a (%)^b
1	MeOH	25 °C	0.5	3	42	18	8	64
2	MeOH	0	0.5	3	22	18	12	42
3	MeOH	reflux	0.5	3	52	19	7	67
4	EtOH	reflux	0.5	3	10	36	N.D.	22
5	^{i}PrOH	reflux	0.5	3	44	10	5	75
6	^{t}BuOH	reflux	0.5	3	23	6	4	74
7	^{n}BuOH	reflux	0.5	3	39	6	7	75
8	benzene	reflux	0.5	3	35	14	12	57
9	toluene	reflux	0.5	3	23	9	6	60
10	THF	reflux	0.5	3	14	3	N.D.	82
11	CH_3CN	reflux	0.5	3	50	16	16	61
12	AcOEt	reflux	0.5	3	10	2	N.D.	83
13	1,4-dioxane	reflux	0.5	3	34	10	8	65
14	^{i}PrOH	reflux	0.5	6	54	13	6	74
15	^{i}PrOH	reflux	0.5	24	50	15	15	63
16	^{i}PrOH	reflux	0.6	6	54	20	16	60
17	^{i}PrOH	reflux	0.8	6	60	33	22	52
18	^{i}PrOH	reflux	1.0	6	57	46	17	48

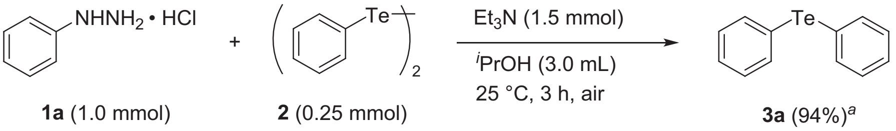
^{a}The yield was calculated based on **2** and determined by ^{1}H NMR (1,3,5-trioxane, $CDCl_3$).
^{b}Selectivity **4a** (%) = 100 × **4a / (4a + 3b + 3a).**

proceeded even when some aprotic solvents were used, but the yields and selectivities were lower than those in the case of ^{i}PrOH (entries 10–13). As to the reaction time, the yield and the selectivity of **4a** were improved by prolonging the reaction time for 6 h (entries 14 and 15 vs entry 5). Using excess amount of **1b** decreased the selectivity of **4a** (entries 16–18 vs entry 14).

In these reactions, symmetrical diaryl tellurides **3b** and **3a** were generally formed as by-products. This strongly suggests the S_H2 reaction also occurred at the tellurium of diaryl



SCHEME 2 Reaction of arylhydrazine hydrochloride with diaryl telluride. [a]The yield was calculated based on **3a** and determined by ^{1}H NMR (1,3,5-trioxane, $CDCl_3$)



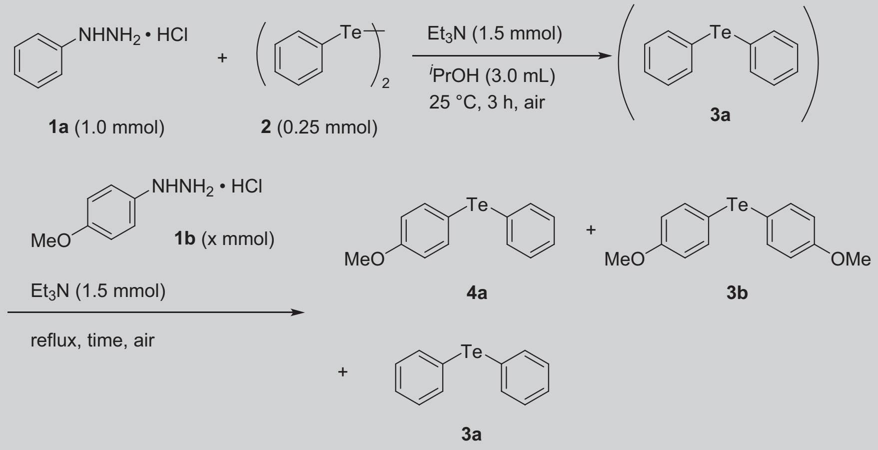
SCHEME 3 Synthesis of symmetical diaryl telluride **3a** in ^{i}PrOH. [a]The yield was calculated based on **2** and determined by ^{1}H NMR (1,3,5-trioxane, $CDCl_3$)

tellurides. Therefore, we next paid attention to the S_H2 reaction using diaryl telluride such as Ph_2Te as a carbon radical trapping reagent (Scheme 2).

To clarify whether the bimolecular homolytic substitution (S_H2) reaction occurs on the tellurium of diaryl telluride, we carried out the reaction using equimolar amounts of 4-methoxyphenylhydrazine hydrochloride (**1b**) and diphenyl telluride (**3a**) in the presence of Et_3N in refluxing ^{i}PrOH in the air for 3 hours. As a result, unsymmetrical telluride **4a** was obtained in 45% yield along with 9% of **3b** and 41% of **3a** (recovered). The result clearly indicates that the S_H2 reaction on the tellurium of diphenyl telluride can occur successfully.

Thus, we next examined the synthesis of unsymmetrical diaryl telluride using diphenyl telluride (**3a**) generated in situ from diphenyl ditelluride (**2**). As indicated in Scheme 3, **3a** can be obtained in high yield by changing the solvent from MeOH (Table 1, entry 11) to ^{i}PrOH in the reaction of phenylhydrazine hydrochloride (**1a**) with **2**. Therefore, the

TABLE 3 Optimization of reaction conditions for the one-pot synthesis of unsymmetrical diaryl telluride



Entry	1b (mmol)	Time (h)	Yield (%)[a] 4a	3b	3a
1	0.5	6	41	5	56
2	0.6	6	60	12	47
3	1.0	1	58	16	38
4	1.0	2	72 (50[b])	26 (6[b])	30
5	1.0	4	71	34	21
6	1.0	24	62	32	23

[a]The yield was calculated based on **2** and determined by ^{1}H NMR (1,3,5-trioxane, $CDCl_3$).
[b]Isolated yield.

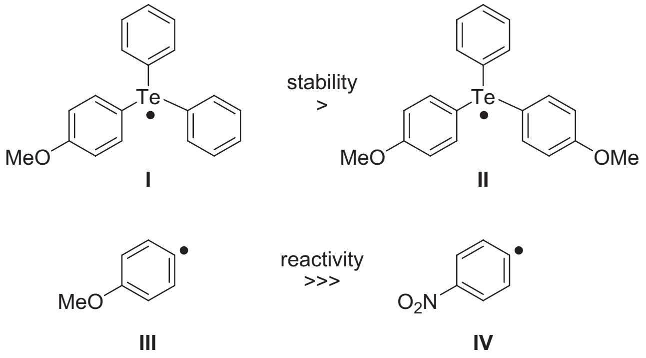


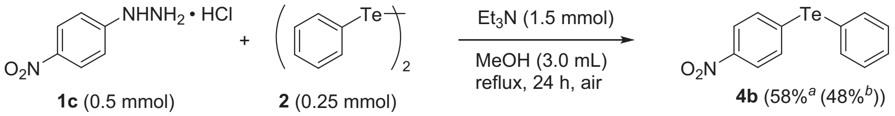
FIGURE 1 Reactivity of radical species and stability of S_H2 key species

unsymmetrical diaryl telluride synthesis might be simplified by a one-pot reaction in ^{i}PrOH via the generation of the symmetrical diphenyl telluride **3a**, and the subsequent reaction with arylhydrazine hydrochlorides (Table 3).

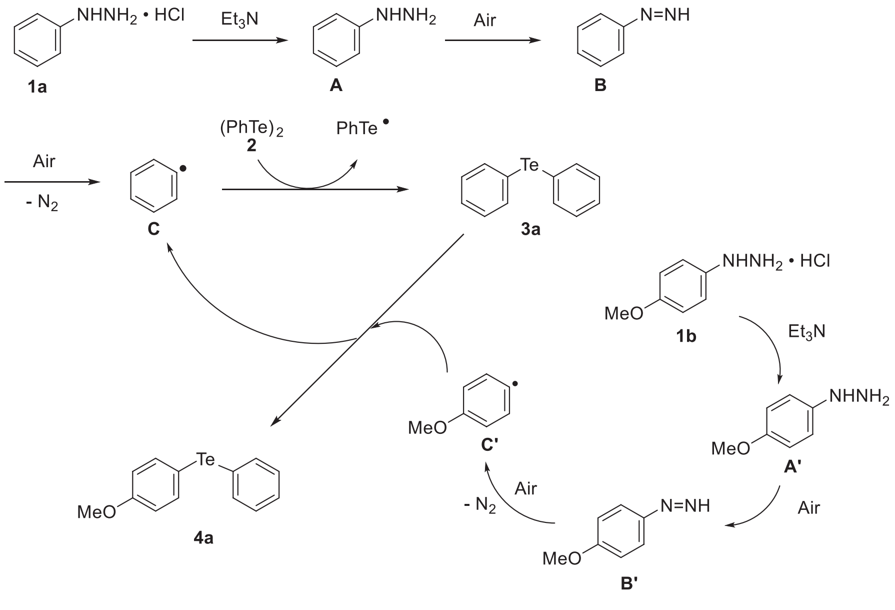
Thus, the one-pot synthesis of unsymmetrical diaryl telluride **4a** using 4-methoxyphenylhydrazine hydrochloride (**1b**) and diphenyl ditelluride (**2**) was investigated in detail. When the stoichiometric reaction of **1b** with in situ generated **3a** was carried out for 6 h, the desired unsymmetrical telluride **4a** was obtained in 41% yield along with **3b** (5%) (entry 1). Since 56% of **3a** was recovered in this reaction, we next examined the reaction using excess amounts of **1b** (entry 2). As a result, the yield of **4a** was improved (60%). When a similar reaction was conducted using further excess amounts of **1b** for 1 hour, a similar result was obtained (entry 3 vs entry 2). Prolonging the reaction time resulted in increase in both the yields of **4a** and **3b** (entries 4–5). However, the yield of **3b** was not further increased even prolonging the reaction time (24 hours) (entry 6). Comparing the methods using Ph_2Te (in situ generated) vs $(PhTe)_2$ (Table 3, entry 4 vs Table 2, entry 17), the one-pot reaction was more advantageous in terms of the higher yield of unsymmetrical diaryl telluride **4a**.

Keeping the diaryl telluride synthesis using phenylhydrazine bearing an electron-donating group such as *p*-MeO group in mind, we next examined a similar one-pot reaction of diphenyl telluride (**3a**) with arylhydrazine hydrochlorides bearing an electron-withdrawing group like *p*-NO_2 (**1c**). However, the S_H2 reaction of in situ generated **3a** with **1c** did not proceed, and only **3a** was obtained in 96% yield.

The selectivity of unsymmetrical/symmetrical diaryl tellurides and the influence of the substituents on the S_H2 reaction between **3a** and substituted phenyl radicals can be explained by the stability of radical species, as shown in Figure 1. In general, radical species are stabilized by an electron-withdrawing group and destabilized by an electron-donating one. In the case of *p*-MeO group, species **I** is more stable than species **II**; therefore, unsymmetrical diaryl telluride **4a** was obtained as the major product. In terms of the



SCHEME 4 Reaction of arylhydrazines bearing electron-withdrawing groups with diphenyl ditelluride. ^{a}The yield was calculated based on **2** and determined by ^1H NMR (1,3,5-trioxane, $CDCl_3$). ^{b}Isolated yield



SCHEME 5 A plausible reaction pathway for the synthesis of unsymmetrical diaryl tellurides

reactivity of radical species generated from arylhydrazine hydrochlorides, species **III** is much more reactive than species **IV**; therefore, the reaction proceeded smoothly with **1b** and did not proceed with **1c**.

Since the S_H2 reaction on the tellurium of diphenyl telluride did not proceed with **1c**, we next examined the synthesis of unsymmetrical diaryl telluride by the reaction of diphenyl ditelluride (**2**) with arylhydrazine hydrochloride bearing electron-withdrawing group like -NO_2 (**1c**). As we expected, the reaction of **1c** with **2** successfully afforded the unsymmetrical diaryl telluride **4b** in 48% isolated yield, and interestingly, no bis(*p*-nitrophenyl) telluride was obtained (Scheme 4).

Based on these mechanistic considerations, a possible pathway for this diaryl telluride synthesis is proposed, as shown in Scheme 5. Phenylhydrazine hydrochloride (**1a**) reacts with Et_3N to generate phenylhydrazine **A**, which is oxidized by air to convert to phenyl radical **C**. Trapping **C** by diphenyl ditelluride (**2**) affords diphenyl telluride (**3a**). 4-Methoxyphenyl hydrazine hydrochloride (**1b**) is converted into the corresponding aryl radical **C'** in the same way. Then, the S_H2 reaction between **C'** and **3a** occurs to afford the desired unsymmetrical telluride **4a** and phenyl radical **C**.

3 | CONCLUSION

In summary, the reaction of aryl radicals generated from arylhydrazines in the air, with diaryl ditelluride or diaryl telluride was investigated in detail. Symmetrical diphenyl telluride could be synthesized conveniently by the reaction of phenylhydrazine with diphenyl ditelluride. Based on this symmetrical diaryl telluride synthesis, the one-pot reaction of arylhydrazines with in situ generated diphenyl telluride in the air was examined. The selectivity of the reaction products (unsymmetrical/symmetrical diaryl tellurides) and the reactivity of the arylhydrazine hydrochlorides were found to be dependent on the stability of radical species. In the case of arylhydrazine hydrochloride bearing electron-donating group, unsymmetrical diaryl telluride was preferentially synthesized. Using arylhydrazine hydrochloride bearing electron-withdrawing group, the in situ generated aryl radical was not reactive enough to undergo the S_H2 reaction, whereas the aryl radical could be trapped by diphenyl ditelluride to afford an unsymmetrical diaryl telluride with no formation of any by-products such as symmetrical diaryl tellurides.

We are now investigating the effects of the substituents of diaryl tellurides on these reaction conditions in detail. We believe that our studies on the bimolecular homolytic substitution at the tellurium atom of diaryl tellurides lead to new approaches of aryl radical chemistry using diaryl tellurides as aryl sources.

4 | EXPERIMENTAL PROCEDURE

Unless otherwise stated, all starting materials and solvents were purchased from commercial sources and used without further purification. ^1H NMR spectra were recorded on JEOL JNM-ECS400 (400 MHz) FT NMR system or JEOL JNM-ECX400 (400 MHz) FT NMR system in $CDCl_3$ with Me_4Si as an internal standard. ^{13}C NMR spectra were recorded on JEOL JNM-ECX400 (100 MHz). ^{125}Te NMR spectra were recorded on JEOL JNM-ECX400 (126 MHz).

4.1 | Synthesis of diphenyl telluride (3a) (Table 1, entry 11)[35]

Phenylhydrazine hydrochloride (**1a**) (86.8 mg, 0.6 mmol), diphenyl ditelluride (**2**) (102.4 mg, 0.25 mmol), Et_3N (0.21 mL, 1.5 mmol), and MeOH (3.0 mL) were added to a round-bottomed flask, and the reaction mixture was stirred at 25 °C for 3 hours under air. After the reaction was complete, the resulting mixture was transferred into a round-bottom flask using MeOH (5 mL) and concentrated under reduced pressure. The residue was purified by column chromatography (eluent: hexane) to give diphenyl telluride (**3a**) (118.5 mg, 84% yield) as a pale-red oil; ^1H NMR (400 MHz, $CDCl_3$): δ 7.69 (d, $J = 7.8$ Hz, 4H), 7.27 (t, $J = 7.3$ Hz, 2H), 7.20 (t, $J = 7.6$ Hz, 4H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$): δ 138.1, 129.6, 128.0, 114.8; ^{125}Te NMR (126 MHz, $CDCl_3$): δ 691.3.

4.2 | Reaction of 4-methoxyphenylhydrazine hydrochloride (1b) with diphenyl telluride (3a) (Scheme 2)

4-Methoxyphenylhydrazine hydrochloride (**1b**) (52.4 mg, 0.3 mmol), diphenyl telluride (**3a**) (84.5 mg, 0.3 mmol), Et_3N (0.13 mL, 0.9 mmol), and ^{i}PrOH (3.0 mL) were added to a round-bottomed flask, and the reaction mixture was refluxed for 3 h under air. After the reaction was complete, the resulting mixture was transferred into a round-bottom flask using MeOH (5 mL) and concentrated under reduced pressure. The yield of the products was confirmed by ^1H NMR spectroscopy (internal standard: 1,3,5-trioxane, $CDCl_3$).

4.3 | One-pot synthesis of unsymmetrical diaryl tellurides (Table 3, entry 4)[35]

Phenylhydrazine hydrochloride (**1a**) (144.6 mg, 1.0 mmol), diphenyl ditelluride (**2**) (102.4 mg, 0.25 mmol), Et_3N (0.21 mL, 1.5 mmol), and ^{i}PrOH (3.0 mL) were added to a round-bottomed flask, and the reaction mixture was stirred at 25 °C for 3 hours under air. After the reaction, 4-methoxyphenylhydrazine hydrochloride (**1b**) (174.6 mg, 1.0 mmol) and Et_3N (0.21 mL, 1.5 mmol) were added to the reaction mixture and refluxed for 2 hours under air. After the reaction was complete, the resulting mixture was transferred into a round-bottom flask using MeOH (5 mL) and concentrated under reduced pressure. The residue was purified by preparative TLC on silica gel (eluent: $CHCl_3$/hexane) to give 4-methoxyphenyl phenyl telluride (**4a**) (77.3 mg, 50% yield) as a pale-red oil, and bis(4-methoxyphenyl) telluride (**3b**) (11.0 mg, 6% yield) as a pale-red oil, respectively.

4a; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 (d, $J = 9.2$ Hz, 2H), 7.56 (d, $J = 6.9$ Hz, 2H), 7.14-7.21 (m, 3H), 6.79 (d, $J = 8.7$ Hz, 2H), 3.79 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 160.1, 141.3, 136.5, 129.5, 127.4, 116.0, 115.7, 103.3, 55.27; ^{125}Te NMR (126 MHz, $CDCl_3$): δ 667.0.

3b; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.62 (d, $J = 8.7$ Hz, 4H), 6.76 (d, $J = 8.7$ Hz, 4H), 3.78 (s, 6H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 160.0, 139.8, 115.5, 104.7, 55.3; ^{125}Te NMR (126 MHz, $CDCl_3$): δ 652.0.

4.4 | Reaction of arylhydrazines bearing electron-withdrawing groups with diphenyl ditelluride (Scheme 4)[36]

4-Nitrophenylhydrazine hydrochloride (**1c**) (94.8 mg, 0.5 mmol), diphenyl ditelluride (**2**) (102.4 mg, 0.25 mmol), Et_3N (0.21 mL, 1.5 mmol), and MeOH (3.0 mL) were added to a round-bottomed flask, and the reaction mixture was refluxed for 24 hours under air. After the reaction was complete, the resulting mixture was transferred into a round-bottom flask using MeOH (5 mL) and concentrated under reduced pressure. The residue was purified by preparative TLC on silica gel (eluent: $CHCl_3$/hexane) to give 4-nitrophenyl phenyl telluride (**4b**) (77.5 mg, 48% yield) as a yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, $J = 8.7$ Hz, 2H), 7.86 (d, $J = 6.9$ Hz, 2H), 7.58 (d, $J = 8.7$ Hz, 2H), 7.44 (t, $J = 7.6$ Hz, 1H), 7.33 (t, $J = 7.6$ Hz, 2H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 147.3, 140.4, 135.4, 130.3, 129.5, 128.0, 123.8, 112.7; ^{125}Te NMR (126 MHz, $CDCl_3$): δ 727.0.

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ORCID

Shintaro Kodama https://orcid.org/0000-0003-4190-9539

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