Preparation and reactions of 4,4-dilithiodithienogermole

Jun Hyun Song, Taishi Nabeya, Yohei Adachi, Yousuke Ooyama, Joji Ohshita[*]

Department of Applied Chemistry, Graduate School of Engineering, Hiroshima University, Higashi-Hiroshima 739-8527, Japan

ABSTRACT

The reaction of 4,4-dichloro-2,6-di(n-butyl)dithienogermole (**DTGCl**) with lithium naphthalenide yielded a dark blue solution, which was treated with methyl iodide to provide a 4,4-dimethylated product in 67% isolated yield, indicating the formation of 4,4-dilithio-2,6-di(n-butyl)dithienogermole (**DTGLi**). Similar treatment of the dark blue solution containing **DTGLi** with trimethylsilyl fluoride and water gave the corresponding 4,4-disubstituted products. When trimethylsilyl chloride was used, however, no silylated products were obtained, likely due to steric hindrance. The reaction of **DTGLi** with **DTGCl** gave poly[2,6-di(n-butyl)dithienogermole-4,4-diyl], whose UV–vis absorption spectra revealed a more extended conjugation than that of a previously prepared product from the Wurtz-type coupling of **DTGCl** with sodium. Reinvestigation of the electronic states of poly(dithienogermole-4,4-diyl) derivatives on the basis of crystal orbital calculations was also accomplished.

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1. Introduction

There has been considerable interest in dithienosilole (DTS) and dithienogermole (DTG) derivatives because of the efficient conjugation in those systems [1], which arises from the interaction between the Si/Ge σ*-orbital and the bithiophene π*-orbital (σ*-π* conjugation) as well as the high planarity of the tricyclic systems [1,2]. The current focus is on their use as building units of π-conjugated oligomers and polymers that are applicable as electron-transporting layers and emissive materials of organic light-emitting diodes (OLEDs), semiconducting materials for organic thin film transistors (OTFTs) and polymer solar cells (PSCs) [3], and sensing materials [4]. However, the synthetic procedures for DTS and DTG have been limited. We recently demonstrated that the reactions of 3,3′-dilithio-2,2′-bithiophenes with tetrachlorogermane gave 4,4-dichlorodithienogermoles, as presented in Scheme 1, although our attempts to prepare similar DTS dichlorides were unsuccessful. The resulting DTG dichlorides were treated with several nucleophiles to provide 4,4-disubstituted DTGs as the first example of the direct derivatization of the bridging Ge atom of DTGs [5]. The DTG dichlorides underwent polymerization via Wurtz-type reactions with sodium to give poly(dithienogermole-4,4-diyl)s (Scheme 1) [6]. The polymers exhibited interesting electronic states, providing UV absorption bands that were likely due to σ-

conjugation within the polygermane chain and σ-π conjugation between the polygermane σ-orbital and the DTG π-orbital. The stacking of DTG π-systems that accumulated on the polygermane backbone was also speculated.

In the present study, we prepared 4,4-dilithiated DTG by treating DTG dichloride with lithium naphthalenide to explore further the scope of the synthetic utility of DTG dichloride. Although our attempts to isolate 4,4-dilithiated DTG failed, the reactions of thus-prepared 4,4-dilithiated DTG with electrophiles gave 4,4-disubstituted products, as expected. The reactions of 4,4-dilithiated DTG with DTG dichloride gave poly(dithienogermole-4,4-diyl), which has a more extended σ-conjugation than the product previously prepared by the Wurtz coupling of DTG dichloride with sodium [6]. The electronic states of poly(dithienogermole-4,4-diyl) were reinvestigated by crystal orbital calculations.

2. Results and discussion

2.1. Formation of 4,4-dilithiated DTG

The reaction of 4,4-dichloro-2,6-di(n-butyl)dithienogermole (**DTGCl**) with 5 equiv of lithium naphthalenide in THF at −78 °C yielded a dark blue solution. Treatment of the solution with an excess of methyl iodide at the same temperature immediately provided a light yellow cloudy mixture whose ^{1}H NMR spectrum indicated the formation of 4,4-dimethyl-2,6-di(n-butyl)dithieno-germole (**DTGMe**), although some unidentified signals were also

* Corresponding author.
  E-mail address: jo@hiroshima-u.ac.jp (J. Ohshita).
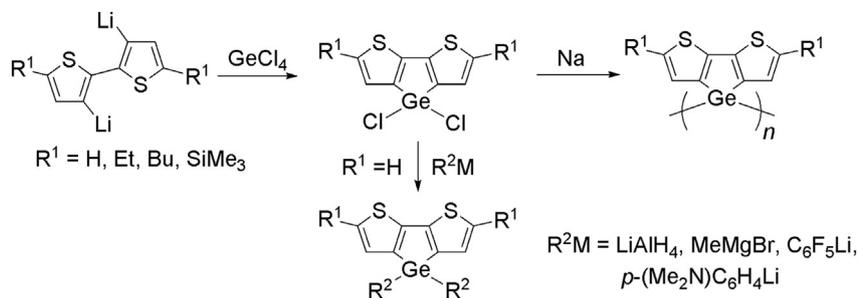
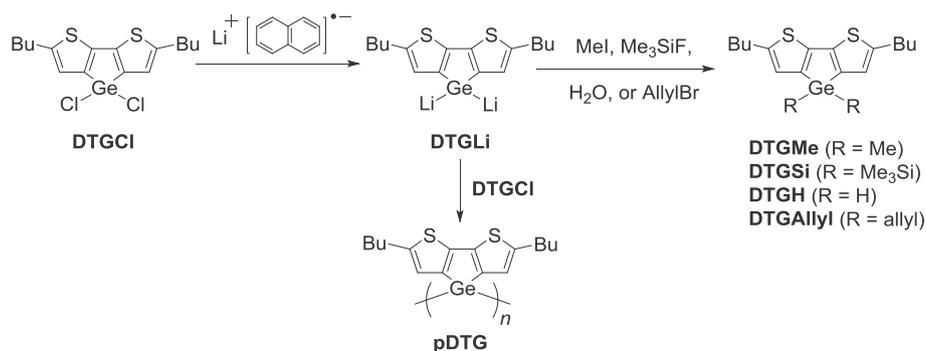**Scheme 1.** Preparation and reactions of DTG dichlorides.

observed. This clearly indicated the intermediary formation of 4,4-dilithio-2,6-di(n-butyl)dithienogermole (**DTGLi**). After the mixture was hydrolyzed with water and naphthalene was removed by sublimation, **DTGMe** was readily separated from the mixture by column chromatography in 67% yield, which was higher than the yields of substitution products from the reactions of DTG dichloride (R^1 = H in Scheme 1) with nucleophiles (R^2M) (22—51%). Similar treatment of the **DTGLi** solution with trimethylsilyl fluoride gave 4,4-bis(trimethylsilyl)-2,6-di(n-butyl)dithienogermole (**DTGSi**) in 26% isolated yield (Scheme 2). The low yield of **DTGSi** is due to its difficult isolation. In fact, the ^{1}H NMR spectrum of the reaction mixture revealed that **DTGSi** was formed in approximately 43% yield, together with 5,5′-di(n-butyl)-2,2′-bithiophene and 2,6-di(n-butyl)dithienogermole (**DTGH**) in approximately 10% yield each. With trimethylsilyl chloride, however, no substitution product was obtained, likely due to the steric hindrance of the chloride. In this reaction, a mixture containing 5,5′-dibutyl-2,2′-bithiophene was formed. Reactions of **DTGLi** with water and allyl bromide gave also substitution products, but only in low yields 7% NMR yield and 5% isolated yield for **DTGH** and **DTGAllyl**, respectively (Scheme 2). Unidentified signals were seen in the ^{1}H NMR spectrum of the reaction mixtures. Attempted crystallization of **DTGLi** by slow evaporation of the solvent failed.

DTGSi was expected to exhibit σ-π conjugation between DTG and Si-Ge-Si units, which would lead to an extended molecular conjugation compared to **DTGH** and **DTGMe**. Although these compounds showed nearly the same UV absorption spectral profiles (λ_{max} = 351 nm, ε = 13,000 L/(mol cm) for **DTGH**; λ_{max} = 350 nm, ε = 16,000 L/(mol cm) for **DTGMe**; λ_{max} = 345 nm, ε = 12,000 L/(mol cm) for **DTGSi**), as presented in Fig. 1, the absorption edge of **DTGSi** reached a lower energy than that of **DTGH**. The photoluminescence (PL) maximum of **DTGSi** appeared at 432 nm (Φ = 0.35), which was red-shifted by approximately 15 nm relative to that of **DTGH** (414 nm, Φ = 0.50) and **DTGMe** (415 nm, Φ = 0.50), again suggesting the extended conjugation of **DTGSi**.

2.2. Preparation of poly(dithienogermole-4,4-diyl)

Poly[2,6-di(n-butyl)dithienogermole-4,4-diyl] (**pDTG**) was obtained from the reactions of **DTGLi** with **DTGCl** in the ratios of 1:1 and 1:2 in THF (Table 1). After treatment of a **DTGLi** solution with **DTGCl**, naphthalene was removed from the hydrolyzed reaction mixtures by sublimation under reduced pressure and the residue was directly analyzed by gel permeation chromatography (GPC) and UV—vis absorption, PL, and ^{1}H NMR measurements. The ^{1}H NMR spectra of the polymer samples resembled that of the product prepared by the sodium condensation of **DTGCl** (**pDTG4**) [6] and showed that the starting **DTGCl** was completely consumed. GPC analysis indicated a higher molecular weight for **pDTG2** prepared by the 1:2 reaction than **pDTG1** prepared by the 1:1 reaction. This is likely due to the formation of GeCl-terminated oligomers brought about by the use of an excess of **DTGCl**. In this reaction, an excess of lithium naphthalenide was used for the formation of **DTGLi**. These oligomers were subsequently linked therefore by further reduction by excess lithium naphthalenide or by hydrolysis/condensation forming Ge-O-Ge linkages during the workup process to increase chain length. In contrast, for **pDTG1**, lithium naphthalenide was used in large excess. The reaction of **DTGLi** with **DTGCl** would occur rapidly to give a polymer, but further reduction of the polygermane chain might have taken place to shorten the polymer chain. We also carried out the reaction of **DTGCl** with 2 equiv of lithium naphthalenide and resulting polymer **pDTG3** had a molecular weight that was higher than those of **pDTG1** and **pDTG2**.

As can be seen from Fig. 2, **pDTG1** and **pDTG2** have similar UV—vis absorption profiles: **pDTG1** has a pronounced band around 360 nm, which was assigned to be due to σ-π conjugation in the previous work [6], whereas **pDTG2** has only a shoulder at nearly the same energy. The spectrum of **pDTG3** exhibits ambiguous shoulders in this region although a maximum appears at 330 nm, indicating suppressed conjugation. The PL spectra of the polymers provided clear bands that were red-shifted in the order of

Scheme 2. Formation and reactions of 4,4-dilithio-2,6-di(n-butyl)dithienogermole.
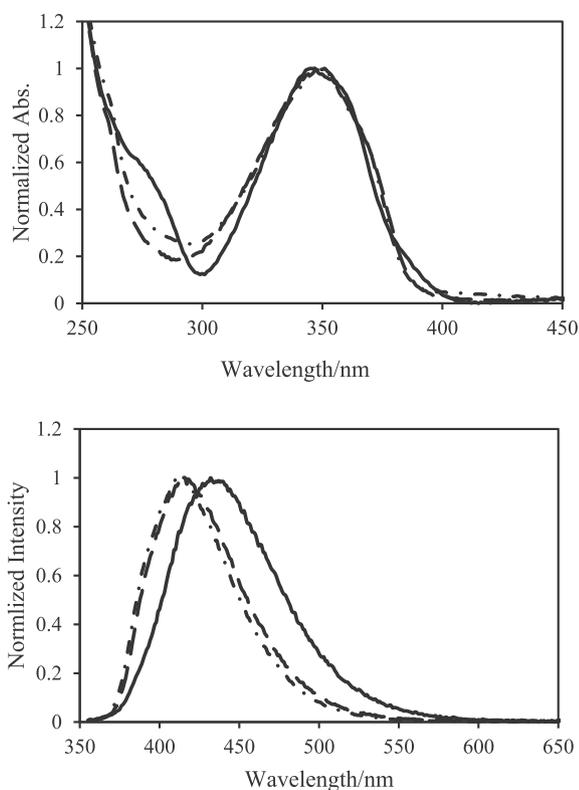
Fig. 1. UV–vis absorption (top) and PL (bottom) spectra of **DTGH** (- - -), **DTGMe** (– • –), and **DTGSi** (——) in THF, excited at 351, 350, and 345 nm, respectively.

Fig. 2. UV–vis absorption (top) and PL (bottom) spectra of **pDTG1** (——), **pDTG2** (- - -), and **pDTG3** (– • –).

Table 1
Preparation of poly[2,6-di(n-butyl)dithienogermole-4,4-diyl].

polymer	conditions	M_n	M_w/M_n	PL λ_{max}
pDTG1	**DTGLi** + **DTGCl**	1600	2.0	460
pDTG2	**DTGLi** + 2 **DTGCl**	2600	2.6	455
pDTG3	**DTGCl** + 2 LiNapH[b]	5000	2.2	449
pDTG4[a]	**DTGCl** + Na	3200	2.5	442

[a] Reference 6. Reprecipitated from methanol.
[b] Lithium naphthalenide.

pDTG4 < **pDTG3** < **pDTG2** < **pDTG1**. The controlled polymerization using the reactions of **DTGLi** with 1 equiv of **DTGCl** seemed to enhance polymer conjugation. The PL bands were at lower energies than **DTGH** and **DTGSi**.

To know more about the electronic states of **pDTG**, we carried out crystal orbital calculations on model polymer **pDTG0** with hydrogens on the thiophene α-carbons in place of butyl groups, and the results are presented in Fig. 3. We employed dimeric bi(dithienogermole-4,4-diyl) as the repeating unit for the calculations. The nearly degenerate HOCO and HOCO-1 are based on the bithiophene π-orbital. Their energies are approximately 0.7 eV higher than the HOMO of a monomeric model (−5.5 eV) reported previously [6]. Originally, we speculated that the red-shifted absorption of **pDTG** relative to that of monomeric compounds would be ascribed to the σ-π conjugation between the DTG π-orbital and the polygermane σ-orbital [6]. However, as can be seen from Fig. 3, HOCO and HOCO-1 are little contributed from the polygermane σ-orbital. Therefore, it is likely that the intramolecular through-space π-π interaction between the DTG units that have accumulated on the polygermane chain is responsible for the redshift. In fact, we previously determined the fluorescence quantum yields of **pDTG** derivatives in THF to be Φ = 0.05 or less, much smaller than that of

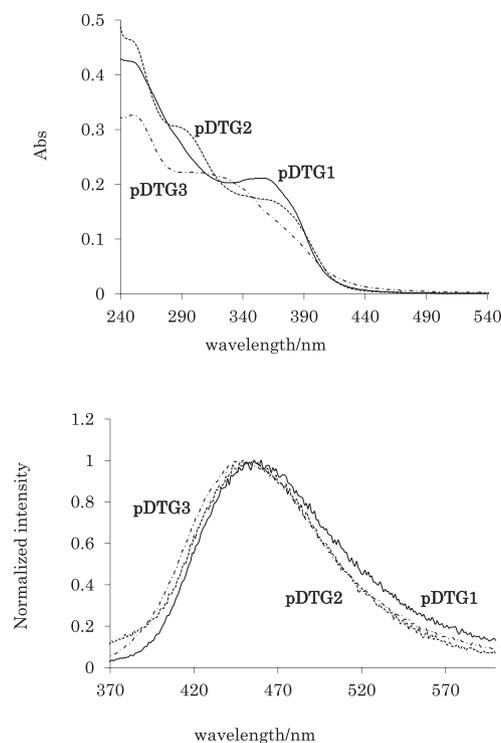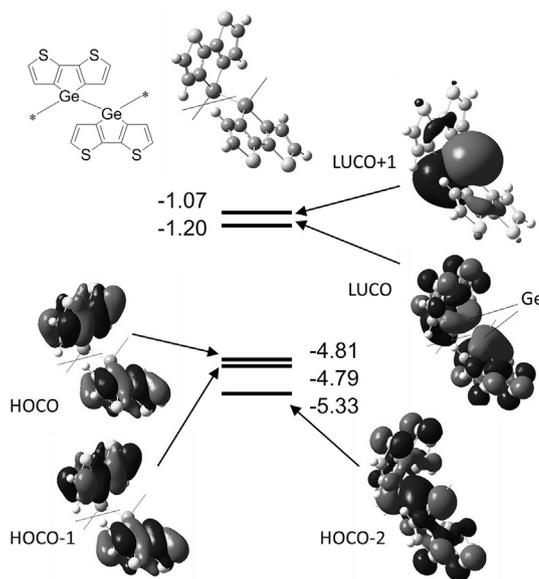**Fig. 3.** Optimized geometry of **pDTG0** with a translating vector for crystal structure formation and energy diagram and profiles of selected molecular orbitals.

DTGH (Φ = 0.50 in THF), likely due to intramolecular aggregation of DTG units of **pDTG** [6]. LUCO shows σ*-π* conjugation characteristic to the DTG system. It is at approximately 0.2 eV lower energy than the LUMO of the DTG monomer [6]. This can be understood by enhanced σ*-π* conjugation using Ge-Ge σ*-orbital and/or the intramolecular through-space orbital interaction between the DTG units in LUCO, as in HOCO and HOCO-1. The HOCO-LUCO transition has a strong π-π* character with the energy of 3.61 eV (343 nm). On

the other hand, HOCO-2 and LUCO+1 are mainly located on the polygermane chain with a slight perturbation by the DTG π-orbital in HOCO-2. The energy gap of 4.26 eV (291 nm) between HOCO-2 and LUCO+1 is much larger than the HOCO-LUCO gap. This agrees with the previous assignment of the absorption bands of **pDTG**, that is, the bands around 250 nm and 380 nm are ascribable to σ-σ^* and π-π^* transitions [6], respectively, although the absolute values do not exactly match.

3. Conclusions

We prepared dilithio-DTG **DTGLi** in moderate yield through the reduction of dichloride **DTGCl** with lithium naphthalenide and explored its potential application as a new building unit of functional conjugated materials. **DTGLi** readily underwent nucleophilic substitution with organic electrophiles, providing new DTG derivatives. The reaction of **DTGLi** with **DTGCl** gave polygermane **pDTG** that showed a more extended conjugation than a previously prepared product from the Wurtz coupling of **DTGCl** with sodium [6].

There have been many reports concerning the synthesis and reactions of group 14 metallole dianions [7], including those of benzo-annulated siloles and germoles [8]. However, this is the first example of metallole dianions annulated with heteroaromatic systems. Further studies on dilithiated DTG are in progress with respect to the electronic states and reactivity.

4. Experimental

4.1. General

All reactions were carried out in dry argon. THF was distilled from calcium hydride and stored over activated molecular sieves (4 Å) until use. **DTGCl** was prepared as reported in the literature [5].

NMR spectra were recorded on a Varian System-500 spectrometer. EI-mass spectra were recorded on a Shimadzu QP−2020A spectrometer. APCI-MS with high resolution was performed on a Thermo Fisher Scientific LTQ Orbitrap XL spectrometer at N-BARD, Hiroshima University. UV−vis absorption and emission spectra were measured on Hitachi U-3210 and HORIBA FluoroMax-4 spectrophotometers, respectively. Polymer molecular weights were determined by GPC relative to polystyrene standards, using serially connected Shodex KF2001 and KF2002 columns and THF as the eluent.

4.2. Preparation and reactions of *DTGLi*

In a 50 mL Schlenk flask, naphthalene (1.93 g, 15.2 mmol) and lithium granules (0.105 g, 15.2 mmol) in 30 mL of THF were stirred at room temperature for 4 h. The resulting dark green solution was added dropwise to a solution of **DTGCl** (1.23 g, 2.92 mmol) in 20 mL of THF at −78 °C and the reaction mixture was stirred overnight at this temperature to form **DTGLi**. To this was slowly added trimethylsilyl fluoride (2.0 mL, 17 mmol) at −78 °C and the reaction mixture was stirred at room temperature for 4 h. The reaction mixture was poured into ice water and extracted with hexane. The extract was dried over anhydrous magnesium sulfate and the solvent was evaporated. The residue was analyzed by ^{1}H NMR measurement using naphthalene as the internal standard, and the formation of **DTGSi** in 43% yield was confirmed. After naphthalene was removed by vacuum sublimation at 80 °C, the residue was subjected to GPC to afford **DTGSi** (0.373 g, 26% yield) as a viscous colorless oil: ^{1}H NMR (δ in CDCl$_3$) 0.16 (s, 18H, SiMe$_3$), 0.94 (t 6H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 1.34−1.45 (m, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$), 1.62−1.72 (m, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$), 2.83 (t,4H, CCH$_2$CH$_2$CH$_2$CH$_3$,

J = 7.5 Hz), 6.68 (s, 2H, DTG). ^{13}C NMR (δ in CDCl$_3$) 0.11, 14.01, 22.34, 30.25, 34.19, 126.75, 143.00, 143.05, 144.82. ^{29}Si NMR (δ in CDCl$_3$) −5.61. HR-MS (APCI) *m/z* Calcd: 496.11630 [M]$^+$, Found: 496.11598.

Quenching **DTGLi** with methyl iodide (5.0 eq for **DTGCl**), water (excess), and allyl bromide (5.5 eq for **DTGCl**) was carried out similarly to that above. Data for **DTGMe**: ^{1}H NMR (δ in CDCl$_3$) 0.57 (s, 6H, CH$_3$Ge), 0.95 (t, 6H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.2 Hz), 1.42 (br sextet, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.6 Hz), 1.47−1.68 (br quintet, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.6 Hz), 2.83 (t, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.6 Hz), 6.72 (s, 2H, DTG). ^{13}C NMR (δ in CDCl$_3$) −2.46, 13.87, 22.75, 29.99, 34.01, 128.15, 141.73, 144.52, 146.04. HR-MS (APCI) *m/z* Calcd: 380.06822 [M]$^+$, Found: 380.06807. **DTGH** was analyzed as the reaction mixture and the data were consistent with those obtained by the reduction of **DTGCl** (*vide infra*). Data for **DTGAllyl**: ^{1}H NMR (δ in CDCl$_3$) 0.96 (t, 6H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 1.42 (sext, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 1.68 (quint, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 2.11 (d, 4H, GeCH$_2$CH=CH$_2$, J = 8.0 Hz), 2.83 (t, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 4.94 (ddt, 1H, GeCH$_2$CH=CH$_2$, J = 10.0, 1.8, 0.9 Hz), 5.00 (dq, 1H, GeCH$_2$CH=CH$_2$, J = 7.0, 1.8 Hz), 5.86−5.97 (m, 1H, GeCH$_2$CH=CH$_2$), 6.69 (s, 2H, DTG). ^{13}C NMR (δ in CDCl$_3$) 13.99, 20.02, 22.35, 30.12, 34.13, 114.45, 126.66, 134.34, 139.33, 145.04, 146.20. HR-MS (APCI) Calcd: [M]$^+$: 432.09952, Found: 432.09972.

4.3. Reaction of *DTGLi* and *DTGCl*

To a mixture of **DTGLi** prepared from 1.26 g (3.00 mmol) of **DTGCl**, 0.108 g (15.6 mmol) of lithium, and 1.99 g (15.6 mmol) of naphthalene in THF was added a solution of **DTGCl** (1.26 g, 3.00 mmol or 2.52 g, 6.00 mmol) in THF (20 mL) at −78 °C. After the mixture was stirred for 24 h at room temperature, the brown mixture was filtrated and the filtrated was concentrated to a half volume under reduced pressure. The concentrated solution was added to 200 mL of methanol and the mixture was stirred for 1 h then the resulting solid was collected by filtration. The solid was reprecipitated from THF to provide 1.23 g (26%) or 2.41 g (36%) of **pDTG1 or pDTG2, respectively**. Polymer **pDTG** gave nearly identical NMR spectra regardless of the reaction conditions: ^{1}H NMR (δ in CDCl$_3$) 0.75−1.09 (br, 6H, CCH$_2$CH$_2$CH$_2$CH$_3$), 1.13−1.44 (br, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$), 1.44−1.73 (br, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$), 2.35−2.96 (br, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$), 5.80−7.10 (br, 2H, DTG).

4.4. Reduction of *DTGCl* for preparation of an authentic sample of *DTGH*

To a solution of 0.799 g (1.90 mmol) of **DTGCl** in 100 mL of THF was added 3.80 mL (3.80 mmol) of a 1.0 M lithium aluminum hydride solution in THF at 0 °C. After stirring the reaction mixture for 60 min at room temperature, it was hydrolyzed by adding 20 mL of a saturated aqueous solution of ammonium chloride. The organic layer was separated and the aqueous layer was extracted with chloroform. The organic layer and the extracts were combined and dried over anhydrous magnesium sulfate. After evaporation, the residue was purified by GPC to afford **DTGH** as a viscous orange oil (0.316 g, 45% yield): ^{1}H NMR (δ in CDCl$_3$) 0.94 (t, 6H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 1.41 (sext, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 1.67 (quint, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 2.83 (t, 4H, CCH$_2$CH$_2$CH$_2$CH$_3$, J = 7.5 Hz), 4.97 (s, 2H, GeH$_2$), 6.78 (s, 2H, DTG). ^{13}C NMR (δ in CDCl$_3$) 13.95, 22.26, 30.10, 34.19, 126.85, 134.85, 146.05, 146.71. IR 2070 cm^{-1} (Ge-H). HR-MS (APCI) *m/z* Calcd: [M]$^+$: 352.03692, Found: 352.03687.

4.5. Computation

Crystal orbital calculations were performed on a Gaussian 09 program by using the periodic boundary conditions formalism (PBC) at the PBC/B3LYP/6-31G(d) level. For the calculations, a dimer was used to define the unit cell.

Acknowledgement

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jorganchem.2019.01.009.

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