

Novel thermal rearrangement reactions: rearrangement stereospecificity of complex $[\text{Fe}_2(\text{CO})_2(\mu\text{-CO})_2(\mu\text{-}t\text{-BuC}_5\text{H}_3\text{-Me}_2\text{GeGeMe}_2\text{C}_5\text{H}_3\text{Bu-}t)]$

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Received 4th January 1999, Accepted 3rd February 1999

Thermal treatment of 1,2-bis(3-*tert*-butylcyclopentadienyl)tetramethyldigermene with $[\text{Fe}(\text{CO})_5]$ in xylene gave the diiron complex $[\text{Fe}_2(\text{CO})_2(\mu\text{-CO})_2(\mu\text{-}3\text{-}t\text{-BuC}_5\text{H}_3\text{Me}_2\text{GeGeMe}_2\text{C}_5\text{H}_3\text{Bu-}t\text{-}3)]$ **1** existing as a mixture of *cis* and *trans* isomers (**1c** and **1t**) which were separated by preparative TLC. When the two isomers were heated in xylene the *cis* substrate (**1c**) rearranged to a *trans* product $[\text{Fe}_2(\text{CO})_4(\mu\text{-}3\text{-}t\text{-BuC}_5\text{H}_3\text{GeMe}_2)]$ **2t**, while the *trans* substrate (**1t**) afforded a *cis* product **2c**. This indicates that the rearrangement reaction is stereospecific, which is consistent with previous results. Molecular structures of **1c**, **1t**, **2t**, and **2c** were determined by X-ray diffraction.

Considerable attention has been focused on the synthesis and study of a variety of bridged binuclear metal–metal bonded transition-metal complexes.¹ These systems are suitable for studying interaction between two metal reaction sites in close proximity. In particular, bridged bis(cyclopentadienyl)tetracarbonyldiiron derivatives in which two cyclopentadienyls are linked together by certain alkyl or silyl groups have been receiving attention.² Compared to non-bridged analogues, the bridging linkage enables these complexes to exhibit characteristic structures and reactivity.³

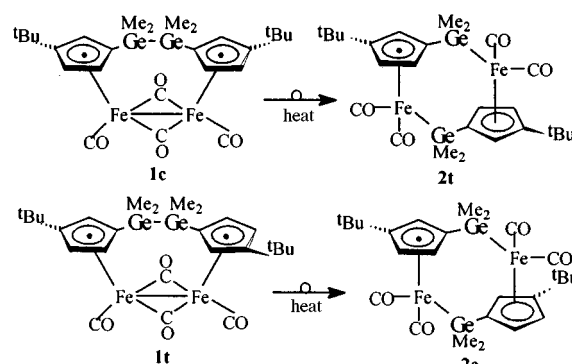
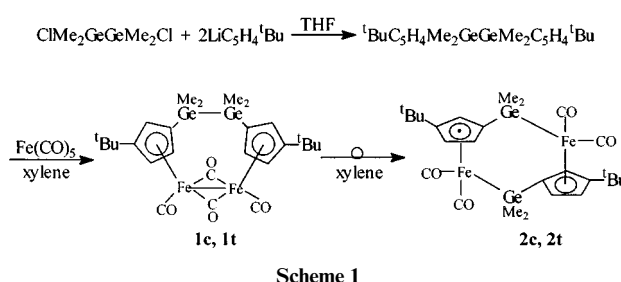
We reported a novel rearrangement reaction between Si–Si and Fe–Fe bonds in a binuclear iron complex $[\text{Fe}_2(\text{CO})_2(\mu\text{-CO})_2(\mu\text{-C}_5\text{H}_4\text{Me}_2\text{SiSiMe}_2\text{C}_5\text{H}_4)]$.⁴ The reaction involves formally the rupture of one Si–Si and one Fe–Fe bond and the formation of two Si–Fe bonds. Accordingly, it may be considered as a σ -bond metathesis between intramolecular Si–Si and Fe–Fe bonds. An alternative mechanism was subsequently proposed on the basis of detailed investigations of the rearrangement stereospecificity, reaction intermediate and cross-over reaction.⁵ The similarity between silicon and germanium prompted us to synthesize related germlyl-bridged analogues and further to examine the reaction stereospecificity.

Results and discussion

Stereospecificity of the reaction

1,2-Bis(3-*tert*-butylcyclopentadienyl)tetramethyldigermene, *t*-BuC₅H₄Me₂GeGeMe₂C₅H₄Bu-*t*, which was prepared from dichlorotetramethyldigermene ClMe₂GeGeMe₂Cl and *tert*-butylcyclopentadienyllithium, was treated with $[\text{Fe}(\text{CO})_5]$ in toluene–xylene (3 : 1) under reflux for 16 h. After work-up, preparative thin layer chromatography (TLC) afforded pure *trans* isomer **1t** (the two *tert*-butyl groups are *trans* to each other) and *cis* isomer **1c** (the two *tert*-butyl groups are oriented in the same direction) in yields of 14 and 10% respectively (Scheme 1).

The formation of two isomers **1c** and **1t** is very likely due to the group BuC₅H₄GeMe₂ being chiral and the ligand *t*-BuC₅H₄Me₂GeGeMe₂C₅H₄Bu-*t* is therefore a mixture of two different compounds, *meso* and *rac*. Study of the subsequent respective rearrangement reactions of **1c** and **1t** revealed a significant feature of the stereochemistry of the reaction. When the *cis* substrate **1c** was heated in refluxing xylene for 10 h only the *trans* product **2t** was formed (monitored by TLC) and obtained in 62% yield, while similar treatment of the *trans* substrate **1t**



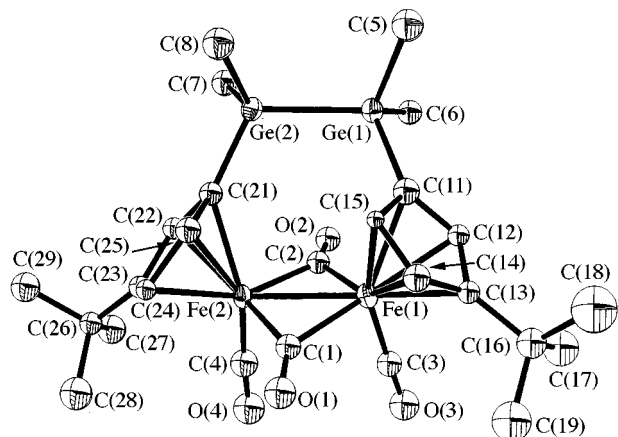
afforded only the *cis* product **2c** in 52% yield (Scheme 2). This strongly indicates that the rearrangement reaction is stereospecific, which is consistent with the result observed for the analogue $[\text{Fe}_2(\text{CO})_2(\mu\text{-CO})_2(\mu\text{-}3\text{-}t\text{-BuC}_5\text{H}_3\text{Me}_2\text{SiSiMe}_2\text{C}_5\text{H}_3\text{Bu-}t\text{-}3)]$.⁵ The observed products are also consistent with any mechanism in which the rings keep their same faces towards the iron atoms.

Complexes **1c** and **1t** are deep red crystals stable in the solid state, whereas **2c** and **2t** are yellow crystals rather stable in solution as well as in the solid state. The IR and ¹H NMR spectra of the respective *cis* and *trans* isomers of both reactants and products exhibited considerable differences. Both **2c** and **2t** showed two strong absorptions for terminal carbonyls at 1900–2000 cm⁻¹ in their IR spectra. However the bridging carbonyl groups of **1c** gave rise to two absorptions (1746.3, 1793.0 cm⁻¹), different from the one (1752.2 cm⁻¹) of **1t**. This is attributable to the difference of the molecular symmetry of the two isomers: **1t** has approximate C₂ symmetry, the two bridging carbonyl

Table 1 Selected bond lengths (Å) and angles (°) of complex **1c**

Fe(1)–Fe(2)	2.547(6)	Ge(1)–Ge(2)	2.410(5)
Ge(1)–C(11)	1.939(31)	Ge(2)–C(21)	1.883(27)
Fe(1)–C(11)	2.161(30)	Fe(2)–C(21)	2.139(25)
C(13)–C(16)	1.499(40)	C(23)–C(26)	1.495(38)
Ge(1)–PL(1) ^a	0.31	Ge(2)–PL(2)	0.37
Fe(1)–PL(1)	1.706	Fe(2)–PL(2)	1.721
Fe(2)–Fe(1)–C(11)	112.1(8)	Fe(1)–Fe(2)–C(21)	104.3(8)
Fe(1)–C(1)–Fe(2)	84.8(11)	Fe(1)–C(2)–Fe(2)	85.0(11)
Fe(2)–C(21)–Ge(2)	136.8(14)	Fe(1)–C(11)–Ge(1)	128.4(14)
Ge(2)–Ge(1)–C(11)	110.4(9)	Ge(1)–Ge(2)–C(21)	113.8(8)
PL(1)–PL(2)	90.8(16)		

^a PL means the plane of the Cp ring.

**Fig. 1** Molecular structure of complex **1c**. Hydrogen atoms are omitted for clarity.

groups being in similar situations while **1c** has approximate C_s symmetry, the situation of the two bridging carbonyl groups being different. In the ^1H NMR spectrum the germanium methyls only exhibited a singlet (δ 0.45) for **1t**, but two singlets (δ 0.36 and 0.53) for **1c**. This is difficult to explain just from the differences in symmetry of the isomers. In solution there is probably coincidental overlap of the two resonances for **1t**. However for **2t** the germanium methyls exhibited two singlets, whereas there was only one singlet for **2c**, attributable to an accidental degeneracy. Their molecular structures were determined by single crystal X-ray diffraction.

Molecular structures of complexes **1c**, **1t**, **2c** and **2t**

The molecular structure of complex **1c** is presented in Fig. 1. The quality of this structure is poor due to poor crystal quality, but it serves to establish the connectivity. Table 1 provides selected bond distances and angles. The molecule of **1c** has mirror symmetry except for some twist about the Ge–Ge bond, with the two *tert*-butyl groups oriented in the same direction. The six-membered ring, Ge(1)–Ge(2)–C(21)–Fe(2)–Fe(1)–C(11) constituting the molecular framework takes a twist boat conformation. The Fe–Fe and Ge–Ge distances [2.547(6) Å, 2.410(5) Å] are close to those [2.544(3) Å, 2.408(2) Å] in the parent analogue [$\text{Fe}_2(\text{CO})_2(\mu\text{-CO})_2(\mu\text{-}t\text{-BuC}_5\text{H}_3\text{Me}_2\text{GeMe}_2\text{C}_5\text{H}_4)$] **3**.⁶ This indicates that the bulky *tert*-butyl substituents have little effect, which is different from the case of the silicon analogue [$\text{Fe}_2(\text{CO})_2(\mu\text{-CO})_2(\mu\text{-}t\text{-BuC}_5\text{H}_3\text{Me}_2\text{SiSiMe}_2\text{C}_5\text{H}_3\text{Bu-}t)$],⁵ possibly owing to the difference in Ge–Ge and Si–Si bond lengths. The dihedral angle between the cyclopentadienyl rings is 90.8°, similar to those in related analogues: *e.g.* 92.8° in *cis*- $[\text{Fe}_2(\text{CO})_4(\eta^5\text{-C}_5\text{H}_5)_2]$,⁷ 97.2° in $[\text{Fe}_2(\text{CO})_4(\eta^5\text{-}\eta^5\text{-C}_5\text{H}_4\text{SiMe}_2\text{-C}_5\text{H}_4)]$,² and 88.8° in $[\text{Fe}_2(\text{CO})_4\{\eta^5\text{-}\eta^5\text{-C}_5\text{H}_4\text{CH}(\text{NMe}_2)\text{-CH}(\text{NMe}_2)\text{C}_5\text{H}_4\}]$.⁸ Atoms Ge(1) and Ge(2) deviate from the linked cyclopentadienyl plane by 0.31 and 0.37 Å, respectively.

The molecular structure of complex **1t** is presented in Fig. 2.

Table 2 Selected bond lengths (Å) and angles (°) of complex **1t**

Fe(1)–Fe(2)	2.551(2)	Ge(1)–Ge(2)	2.416(2)
Ge(1)–C(7)	1.945(4)	Ge(2)–C(16)	1.953(4)
Fe(1)–C(7)	2.150(4)	Fe(2)–C(16)	2.158(4)
C(9)–C(10)	1.519(6)	C(18)–C(19)	1.529(6)
Ge(1)–PL(1)	0.13	Ge(2)–PL(2)	0.28
Fe(1)–PL(1)	1.753	Fe(2)–PL(2)	1.756
Fe(2)–Fe(1)–C(7)	111.9(1)	Fe(1)–Fe(2)–C(16)	106.2(1)
Fe(1)–C(1)–Fe(2)	83.1(2)	Fe(1)–C(2)–Fe(2)	82.7(2)
Fe(2)–C(16)–Ge(2)	133.7(2)	Fe(1)–C(7)–Ge(1)	128.5(2)
Ge(2)–Ge(1)–C(7)	109.8(1)	Ge(1)–Ge(2)–C(16)	115.4(1)
PL(1)–PL(2)	95.9		

Table 3 Selected bond lengths (Å) and angles (°) of complex **2t**

Fe–Ge	2.3741(8)	Fe–C(12)	2.103(5)
Ge–C(12')	1.970(5)	C(15)–C(16)	1.517(8)
Ge–Fe–C(12)	98.9(1)	Fe–Ge–C(12')	114.6(1)
Fe'–C(12')–Ge	131.8(2)		

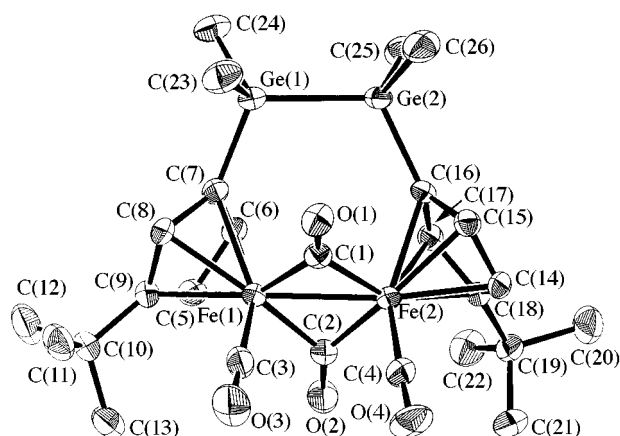
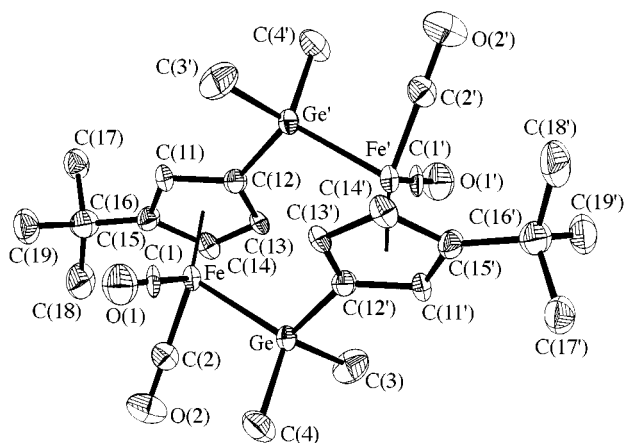
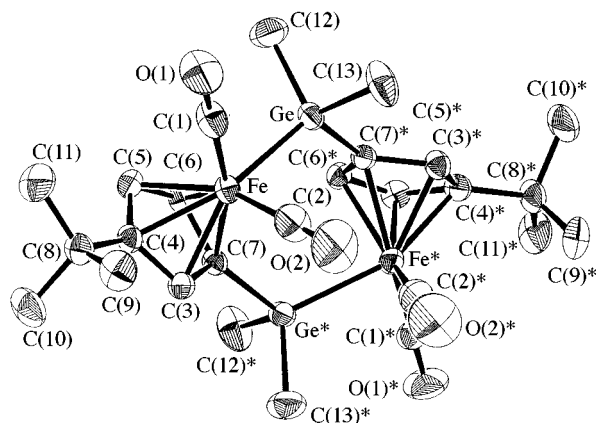
**Fig. 2** Molecular structure of complex **1t**. Hydrogen atoms are omitted for clarity.

Table 2 provides selected bond distances and angles. The molecule of **1t** is similar to that of **1c**. It is different in that the substituents on the rings are *trans* to each other. Like **3c**, the six-membered ring also adopts a twist boat conformation. The dihedral angle between the cyclopentadienyl rings is 95.9°. Atoms Ge(1) and Ge(2) deviate from the linked cyclopentadienyl plane by 0.127 and 0.280 Å, respectively, which are much smaller than in **1c** owing to the reduced steric effect. The remaining molecular parameters are almost equivalent to those in **1c**.

The molecular structure of complex **2t** is illustrated in Fig. 3. Selected bond distances and angles are listed in Table 3. Molecules of **2t** consist of two $[\text{Me}_2\text{Ge}(\eta^5\text{-}t\text{-BuC}_5\text{H}_3)\text{Fe}(\text{CO})_2]$ moieties linked to each other by two Ge–Fe bonds. Like many analogues,^{4,5,9} **2t** has C_i symmetry, and the six-membered ring Fe–Ge–C(12')–Fe'–Ge'–C(12) constituting its molecular framework adopts a stable chair conformation. Pannell and co-workers⁹ recently reported an analogous tin complex unexpectedly obtained in a base-induced migration reaction, which has a structure similar to that of **2t** and the corresponding six-membered ring of which also takes a chair conformation. A noteworthy fact revealed so far is that whereas the six-membered rings in related Fe–Fe complexes, for both silicon and germanium analogues, may be in twisted boat or boat conformation, the corresponding rings in the rearrangement products always take standard chair conformations.^{4–6} This is a noticeable structural characteristic for the products. It appears that such chair conformations, with high symmetry, are prefer-

Table 4 Selected bond lengths (Å) and angles (°) of complex **2c**

Fe–Ge	2.3760(6)	Fe–C(7)	2.110(3)
Ge*–C(7)	1.970(3)	C(4)–C(8)	1.531(4)
Ge*–PL	0.222	Fe–PL	1.718
Ge–Fe–C(7)	99.28(8)	Fe–Ge–C(7*)	115.78(9)
Fe–C(7)–Ge*	132.0(2)	PL–PL*	68.7

**Fig. 3** Molecular structure of complex **2t**. Hydrogen atoms are omitted for clarity.**Fig. 4** Molecular structure of complex **2c**.

able in energy, while the molecular strain in substrates is likely to be the driving force of the rearrangement reaction. The Fe–Ge distance of **2t** [2.3741(8) Å] is close to that in its parent $[\text{Fe}_2(\text{CO})_4(\text{C}_5\text{H}_4\text{GeMe}_2)_2]$ **4** [2.379(2) Å].⁶

The molecular structure of complex **2c** is illustrated in Fig. 4. Selected bond distances and angles are listed in Table 4. The molecule of **2c** also consists of two $[\text{Me}_2\text{Ge}(\eta^5\text{-}t\text{-BuC}_5\text{H}_3)\text{-Fe}(\text{CO})_2]$ moieties linked to each other by two Ge–Fe bonds. Unlike the *trans* isomer **2t**, **2c** has C_2 symmetry and the corresponding six-membered ring is in a semi-chair conformation owing to the interaction between the two *cis tert*-butyls. The dihedral angle between two cyclopentadienyl rings is 68.7° , and the Fe–Ge distance [2.3760(6) Å] is almost equal to that in the *trans* isomer.

Experimental

Schlenk and vacuum line techniques were employed for all manipulations of air- and moisture-sensitive compounds. Reaction solvents were distilled from appropriate drying agents under argon before use. Tetrahydrofuran, toluene and xylene were distilled from sodium–benzophenone and purged with argon prior to use. The ^1H NMR spectra were recorded on a JEOL FX-90Q or Bruker AC-P200 spectrometer, infrared

spectra on a Nicolet 5DX FT-IR spectrometer as KBr disks. Elemental analyses were performed by a Perkin-Elmer 240C spectrometer. 1,2-dichlorotetramethyldigermane¹⁰ and *t*-BuC₅H₅¹¹ was prepared according to literature methods.

Syntheses

(*t*-Bu)C₅H₄Me₂GeGeMe₂C₅H₄(*t*-Bu). A solution of 0.8 g (6.56 mmol) of *t*-BuC₅H₅ in 40 mL of THF, cooled to -20°C , was treated dropwise with 3.45 mL of a 1.90 M hexane solution of *n*-BuLi (6.56 mmol) to give a light yellow solution, which was allowed to warm to room temperature and then stirred for 3 h. The mixture was cooled again to -20°C and a solution of 0.7 g (2.5 mmol) of $\text{ClMe}_2\text{GeGeMe}_2\text{Cl}$ in 20 mL of THF added dropwise. The reaction mixture was slowly warmed to room temperature and stirred for 12 h. Water (50 mL) was added, the aqueous layer separated and extracted twice with 20 mL of diethyl ether. The organic and ether extracts were combined and dried with sodium sulfate overnight. The solvent was removed under reduced pressure and the residue introduced to a silica column in the minimum of hexane. Elution with hexane developed a light yellow band which afforded a yellow oil. It was identified as $(t\text{-Bu})\text{C}_5\text{H}_4\text{Me}_2\text{GeGeMe}_2\text{C}_5\text{H}_4(t\text{-Bu})$ and could be used in the following reaction without further purification. Yield: 0.7 g, 60%. ^1H NMR: δ (CDCl₃): δ 0.12 (s, 12 H, GeMe₂), 1.17 (s, 18 H, CMe₃), 2.92 (br m, 2 H, C₅H₄) and 6.10–6.50 (br m, 6 H, C₅H₄).

$[\text{Fe}_2(\text{CO})_2(\mu\text{-CO})_2(\mu\text{-}t\text{-BuC}_5\text{H}_3\text{Me}_2\text{GeGeMe}_2\text{C}_5\text{H}_3\text{Bu-}t)]$. A solution of 1.90 g (4.2 mmol) of $(t\text{-Bu})\text{C}_5\text{H}_4\text{Me}_2\text{GeGeMe}_2\text{C}_5\text{H}_4(t\text{-Bu})$ and 2.5 mL (19.0 mmol) of $[\text{Fe}(\text{CO})_5]$ in 60 mL of toluene–xylene (3 : 1) was refluxed for 16 h. The solvent was removed under vacuum to give a dark crude product, which was introduced to a silica column (3 × 35 cm) in the minimum of dichloromethane. Elution with light petroleum (bp range $30\text{--}60^\circ\text{C}$)–CH₂Cl₂ (4 : 1) developed a yellow band which upon evaporation afforded 0.17 g of yellow crystals (**2**). The following red band afforded 0.73 g of a deep red solid (**1**) as a mixture of *cis* and *trans* isomers. Complex **1** was dissolved in the minimum volume of dichloromethane and chromatographed by preparative TLC to develop two red bands. The first gave 0.39 g (14%) of deep red crystals (**1t**), the second 0.28 g (10%) of deep red crystals (**1c**). The ^1H NMR of the yellow crystals **2** indicated that they were also a mixture of isomers **2c** and **2t** which could not be separated by chromatographic methods. For **1c**: mp 168°C (decomp.) (Found: C, 46.61; H, 5.49. C₁₃H₁₈FeGeO₂ requires C, 46.65; H, 5.42%); ^1H NMR (CDCl₃): δ 0.36 (s, 6 H, GeMe₂), 0.53 (s, 6 H, GeMe₂), 1.31 (s, 18 H, CMe₃), 4.03 (d, 2 H, $J = 1.94$, C₅H₃), 4.58 (s, 2 H, C₅H₃) and 5.12 (d, 2 H, $J = 1.94$ Hz, C₅H₃); IR ($\tilde{\nu}_{\text{CO}}/\text{cm}^{-1}$) 1974.1, 1931.9, 1793.0 and 1746.3. For **1t**: mp $175\text{--}176^\circ\text{C}$ (Found: C, 46.83; H, 5.35%); ^1H NMR (CDCl₃): δ 0.45 (s, 12 H, GeMe₂), 1.33 (s, 18 H, CMe₃), 4.25 (d, 2 H, $J = 1.84$, C₅H₃), 4.35 (s, 2 H, C₅H₃) and 5.20 (d, $J = 1.84$ Hz, 2 H, C₅H₃); IR ($\tilde{\nu}_{\text{CO}}/\text{cm}^{-1}$) 1990.6, 1942.4 and 1752.2.

The rearrangement reaction of complexes **1c** and **1t**

Complex **1t** (0.25 g) in 25 mL of xylene was refluxed for 10 h. The product was separated by column chromatography (Al₂O₃–hexane), giving 0.13 g (52%) of yellow crystals of **2c**, mp 147°C (decomp.) (Found: C, 46.76; H, 5.31. C₁₃H₁₈FeGeO₂ requires C, 46.65; H, 5.42%). ^1H NMR (CDCl₃): δ 0.53 (s, 12 H, GeMe₂), 1.24 (s, 18 H, CMe₃), 4.73 (s, 4 H, C₅H₃) and 4.83 (s, 2 H, C₅H₃). IR ($\tilde{\nu}_{\text{CO}}/\text{cm}^{-1}$) 1982.3 and 1922.3.

Complex **1c** (0.22 g) in 20 mL of xylene was similarly refluxed for 10 h, yielding 0.14 g (62%) of yellow crystals of **2t**, mp 145°C (decomp.) (Found: C, 46.68; H, 5.25%). ^1H NMR (CDCl₃): δ 0.47 (s, 6 H, GeMe₂), 0.58 (s, 6 H, GeMe₂), 1.27 (s, 18 H, CMe₃), 4.61 (s, 2 H, C₅H₃), 4.66 (s, 2 H, C₅H₃) and 4.90 (s, 2 H, C₅H₃). IR ($\tilde{\nu}_{\text{CO}}/\text{cm}^{-1}$): 1969.5 and 1927.1.

Table 5 Summary of X-ray diffraction data

	1c	1t	2t	2c
Formula	C ₂₆ H ₃₆ Fe ₂ Ge ₂ O ₄	C ₂₇ H ₃₈ Cl ₂ Fe ₂ Ge ₂ O ₄	C ₂₆ H ₃₆ Fe ₂ Ge ₂ O ₄	C ₂₆ H ₃₆ Fe ₂ Ge ₂ O ₄
Formula weight	669.44	754.37	669.44	669.44
Space group	<i>P2₁/c</i>	<i>C2/c</i>	<i>P$\bar{1}$</i>	<i>C2/c</i>
Crystal system	Monoclinic	Monoclinic	Triclinic	Monoclinic
Z	4	8	1	4
<i>a</i> /Å	6.753(1)	25.199(8)	7.513(6)	20.591(3)
<i>b</i> /Å	21.428(4)	11.789(2)	10.811(4)	7.731(2)
<i>c</i> /Å	19.364(4)	20.939(8)	11.152(5)	20.784(4)
α /°			59.91(3)	
β /°	97.53(3)	104.20(3)	69.68(5)	117.529(9)
γ /°			70.10(4)	
<i>V</i> /Å ³	2778(1)	3030(3)	719.3(7)	2933(1)
<i>D</i> /g cm ⁻³	1.601	1.662	1.434	1.515
Crystal size/mm	0.2 × 0.2 × 0.3	0.2 × 0.2 × 0.3	0.2 × 0.3 × 0.3	0.2 × 0.2 × 0.5
λ (Mo-K α)/Å	0.71073	0.71069	0.71073	0.71069
μ /cm ⁻¹	31.68	31.24	30.52	30.24
Total no. observations	4124	4766	2119	2873
No. data with <i>I</i> > 3 σ (<i>I</i>)	1150 [<i>I</i> > 2 σ (<i>I</i>)]	3229	1295 [<i>I</i> > 3 σ (<i>I</i>)]	2051
No. variables	157	469	154	226
<i>R</i>	0.064	0.028	0.069	0.027
<i>R</i> '	0.068	0.033	0.069	0.030
Goodness of fit	1.03	1.25	6.16	1.45

Crystallography

Crystals suitable for X-ray diffraction were obtained from hexane-dichloromethane solution. All data sets were collected on Enraf-Nonius CAD-4 (for **1c** and **2t**) or Rigaku AFC 7R (for **1c** and **2t**) diffractometers with graphite monochromated Mo-K α radiation. Data were corrected in the usual fashion for Lorentz-polarization, and ψ absorption corrections were applied for **1t** and **2c**. All calculations for **1t** and **2c** were performed using *teXsan*,¹² for **1c** and **2t** on PDP11/44 and IBM4 86 computers using SDP-PLUS and Siemens SHELXTL-PC program packages.¹³ The structures were solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically except for **1c** for which there are not enough observed reflections [only 1150 for *I* > 2 σ (*I*)] due to the poor crystal quality. So only heavy atoms were refined anisotropically while other non-hydrogen atoms refined isotropically. Hydrogen atoms for **1t** and **2c** were refined isotropically. For **1c** and **2t** hydrogen atoms were included but not refined. Neutral atom scattering factors were taken from Cromer and Waber.¹⁴ A summary of the crystallographic results is presented in Table 5.

CCDC reference number 186/1342.

Acknowledgements

This work was financially supported by the National Science Foundation of China and State Key Laboratory of Elemento-Organic Chemistry of Nankai University.

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Paper 9/00027E