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Selective Direct Synthesis of Organofunctionalized Dialkylgermanes from Solvochemically Activated Germanium**

Sabine Schlecht

Since the first successful experiments aimed at the direct synthesis of organosilanes and organogermanes, carried out by Rochow and Müller in 1942 – 1947,[1, 2] a whole industry for the production and application of alkylchlorosilanes has evolved. The direct synthesis of alkylgermanes, however, has received much less attention, although alkylated polygermylenes[3] and mixed polygermasilylenes,[4] the synthesis of which requires diorganogermanium compounds, have gained increasing importance as photoconducting polymers and photoresists. Their surface properties and their absorption maxima depend not only on the type of polymer backbone but also on the nature of the organic substituent on the diorganogermanium compound.[3, 4]

Like the direct synthesis of alkylchlorosilanes, the oxidative addition of alkyl halides to germanium is also clearly exothermic [Eqs. (1) – (3)].[5]

$$2\,CH_3Cl + Si \xrightarrow[300\,^\circ C]{Cu} (CH_3)_2SiCl_2 \qquad \Delta H_R = -304\ kJ\,mol^{-1}$$
$$2\,C_2H_5Cl + Si \xrightarrow[350\,^\circ C]{Cu} (C_2H_5)_2SiCl_2 \qquad (1)$$

$$2\,CH_3Cl + Ge \xrightarrow[320\,^\circ C]{Cu} (CH_3)_2GeCl_2 \qquad \Delta H_R = -97\ kJ\,mol^{-1} \qquad (2)$$

$$2\,CH_3Br + Si \xrightarrow[325\,^\circ C]{Cu} (CH_3)_2SiBr_2 \qquad \Delta H_R = -246\ kJ\,mol^{-1} \qquad (3)$$

Nonetheless, the direct reaction of alkyl halides with elemental silicon or germanium requires very drastic conditions (for the synthesis of an organoelement compound) and the use of a copper catalyst [Eqs. (1) – (3)]. This is due to the pronounced kinetic inertness of the elemental tetrels. The required reaction temperature of about 300 °C[5] prevents the use of functionalized organic halides in direct syntheses, because these compounds lack the necessary thermal stability. In the following, the application of solvochemically activated germanium in direct synthesis reactions is reported. When this very reactive form of germanium is used, the oxidative addition takes place at conditions mild enough to allow, for the first time, a direct synthesis of organofunctionalized germanes without the need for a copper-containing additive.

The solvochemical activation of germanium occurs through the reduction of its dichloride with a solution of $Li[Et_3BH]$ in THF[6] at room temperature [Eqs. (4) and (5)]. Completely X-ray amorphous, orange-colored germanium is obtained under these conditions.

$$GeCl_2 \cdot \text{dioxane} + 2\,Li[Et_3BH] \xrightarrow[RT]{THF} \underset{\text{orange}}{Ge^*} + 2\,LiCl + 2\,Et_3B + H_2 + \text{dioxane} \qquad (4)$$

$$GeI_2 + 2\,Li[Et_3BH] \xrightarrow[RT]{THF} \underset{\text{orange}}{Ge^*} + 2\,LiI + 2\,Et_3B + H_2 \qquad (5)$$

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This "activated germanium" has been characterized by thermoanalysis, elemental analysis, and IR, Raman, and UV/Vis spectroscopy. The UV/Vis spectrum showed an absorption maximum at $\lambda = 415$ nm. Elemental analysis of the activated germanium revealed the presence of chlorine and hydrogen in the orange solid.[7] Thus, it can be concluded that the surface is terminated by chloride and hydride. This assumption is strongly supported by the results from vibrational spectroscopy. The Raman spectrum showed a very strong band for the Ge–Ge streching mode of the amorphous network at 292 cm^{-1} and two weaker bands at 545 (ν(Ge–Cl)) and 2011 cm^{-1} (ν(Ge–H)). In the IR spectrum two corresponding bands at 550 (ν(Ge–Cl)) and at 2002 cm^{-1} (ν(Ge–H)) were found. Thermoanalytic investigation of the amorphous germanium showed two distinct signals. At 490 °C a signal for an exothermic transition was detected and at 909 °C the peak for the endothermic melting process was found. Hence, the melting point of the activated germanium is significantly lowered compared to the one of pure elemental germanium ($T_m = 937$ °C).[8] Since for films of completely hydrogenated amorphous germanium (a-Ge:H) crystallization temperatures around 430 °C have been found,[9] and the crystallization temperatures of bulk material are expected to be higher than those of films, it is reasonable to assign the exothermic transition at 490 °C to the crystallization of the amorphous germanium.[10]

Clean reaction of the activated germanium with functionalized alkyl bromides can be achieved at 130–150 °C, depending on the substrate [Eq. (6)]. Polar substrates with a short hydrocarbon chain react at lower temperatures than nonpolar substrates with longer chains. The reaction times are between 2 and 9 h, depending on the scale of the reaction.

$$Ge^* + 2X(CH_2)_nBr \xrightarrow[130-150\,^\circ C]{} 2(X(CH_2)_n)_2GeBr_2 \quad (6)$$

X = CN, $COOCH_3$, Cl
n = 2–4

The yields of organogermanes vary between 82 and 94%. Unlike industrial direct synthesis, these reactions do not form mixtures of the whole family of organobromogermanes $R_{4-n}GeBr_n$, but yield the dialkyl products R_2GeBr_2 with very high selectivity (Table 1). This selectivity for the dialkyl products is maintained at lower reaction temperatures (Table 2), although the lower temperatures no longer allow a complete conversion of the activated germanium, not even at very prolonged reaction times. Yields and distributions of products at different reaction temperatures are given in Table 2 for the reaction of activated germanium with $BrC_2H_4COOCH_3$.

Table 1. Direct syntheses of organofunctionalized germanes from activated germanium Ge*.

Substrate	Product[a]	t [h][b]	T [°C]	Yield [%][b]
$H_3COOCC_2H_4Br$	$(H_3COOCC_2H_4)_2GeBr_2$	2	130	82
$H_3COOCC_4H_8Br$	$(H_3COOCC_4H_8)_2GeBr_2$	2	150	84
NCC_2H_4Br	$(NCC_2H_4)_2GeBr_2$	2	130	94
NCC_3H_6Br	$(NCC_3H_6)_2GeBr_2$	2	130	85
ClC_4H_8Br[c]	$(ClC_4H_8)_2GeBr_2$	5	150	84

[a] The corresponding monoalkyl product is either not observed or is found with a yield <2%. [b] Reaction times and yields for a reaction scale of 1 mmol $GeCl_2 \cdot$ dioxane. [c] The substrate was allowed to react in a 50:50 (v/v) mixture with diethyleneglycol dimethyl ether (diglyme).

Table 2. Dependence of the yield and the distribution of products on the reaction temperature in the reaction of activated germanium with $BrC_2H_4COOCH_3$.

Substrate	Products	T [°C]	t [h][a]	Yield [%][a]
$H_3COOCC_2H_4Br$	$(H_3COOCC_2H_4)_2GeBr_2$	130	2	82
	$(H_3COOCC_2H_4)GeBr_3$			1–2
$H_3COOCC_2H_4Br$	$(H_3COOCC_2H_4)_2GeBr_2$	120	2	74
	$(H_3COOCC_2H_4)GeBr_3$			1–2
$H_3COOCC_2H_4Br$	$(H_3COOCC_2H_4)_2GeBr_2$	100	2	34
	$(H_3COOCC_2H_4)GeBr_3$			0–1

[a] Reaction times and yields for a reaction scale of 1 mmol $GeCl_2 \cdot$ dioxane.

A variation of the scale of a test system from 1–20 mmol showed that the selectivity found for small-scale reactions is completely preserved in the scale-up (Table 3). Again, the reaction of activated germanium with $BrC_2H_4COOCH_3$ was chosen as the test system. This system gives the lowest yield and should therefore be the most susceptible to changes in the reaction conditions. The reaction time is prolonged up to 9 h (20 mmol), but no reduction in yield or selectivity has been observed.

Table 3. Dependence of the reaction time on the reaction scale.

Amount of Ge*	Substrate	Products	T [°C]	t [h]	Yield [%]
1 mmol	$H_3COOCC_2H_4Br$	$(H_3COOCC_2H_4)_2GeBr_2$	130	2	82
		$(H_3COOCC_2H_4)GeBr_3$			1–2
7 mmol	$H_3COOCC_2H_4Br$	$(H_3COOCC_2H_4)_2GeBr_2$	130	6	81
		$(H_3COOCC_2H_4)GeBr_3$			2
20 mmol	$H_3COOCC_2H_4Br$	$(H_3COOCC_2H_4)_2GeBr_2$	130	9	83
		$(H_3COOCC_2H_4)GeBr_3$			1–2

For all reactions described herein it has been indispensable that the residual lithium chloride, which had coprecipitated with the amorphous germanium was washed out thoroughly and completely before the subsequent reaction with an alkyl bromide. Otherwise, the selectivity for the dialkylgermane could not be achieved. The formation of bromogermanes of the type $R_{(4-(n+m))}GeBr_nH_m$ has not been observed for any of the direct syntheses reported here. Organodigermanes or other higher germanes, which are found in the classical Rochow synthesis[2] as by-products, have not been detected. Criteria for the choice of the different alkyl bromides were a variation of the chain length and an alteration of the functional group. The direct synthesis is of particular synthetic value in the case of functional groups that contain a carbonyl functionality and can therefore not be introduced at the germanium center through the use of Grignard reagents or organolithium compounds. The use of functionalized organozinc compounds such as $BrZn(CH_2)_nCOOCH_3$,[11] which are easily accessible, is generally possible here, but would be unfavorable and not competitive with a selective direct synthesis because of the lack of selectivity of the subsequent metathesis with a germanium halide GeX_4 (X = Cl, Br). This

metathesis never yields exclusively the dialkyl product. In addition, a selective direct synthesis represents the more elegant approach due to its ability to form all four new Ge–E bonds (E = C, halogen) in the target molecule in *one* synthetic step.

The variation of chain length presented here constitutes a considerable advance compared to the current synthetic route to functionalized organogermanes,[12] because satisfactory yields were only obtained when vinyl or allyl compounds were used in the hydrogermylation step. Thus, all organofunctionalized halogermanes that are commercially available contain a C_3 chain between the tetrel and the functional groups.

The reactions described in this work require the use of the orange-colored, amorphous germanium that is obtained in the reduction of $GeCl_2 \cdot$dioxane (or of GeI_2) with $Li[Et_3BH]$. Previously reported black activated germanium, which can be obtained from GeI_2 and elemental potassium,[13] did not react with the alkyl bromides used in this work under the given reaction conditions (Table 1), although it reacts with α-haloesters and other Reformatsky substrates at room temperature.[13] Brown amorphous germanium, which was obtained from $GeCl_4$ and lithium naphthalide, did not show a reactivity analogous to the one of the orange material either. With the direct synthesis from appropriately activated germanium, a synthetic method becomes available that allows an easy, mild, and selective preparation of organofunctionalized dialkylgermanes in a single step. Furthermore, this approach shows that the kinetic inertness of the elemental tetrels can be overcome under appropriate reaction conditions and that the exothermal nature of the direct synthesis can be exploited in a very controlled manner.

Experimental Section

The syntheses required exclusion of air and humidity and were carried out under an argon atmosphere. Typical protocol for a direct synthesis with activated germanium: $GeCl_2 \cdot$dioxane (232 mg, 1.0 mmol) was dissolved in THF (50 mL) and was reduced to the element with $Li[Et_3BH]$ (2.0 mL of a 1M solution in THF). After the evolution of hydrogen had ceased, the solution was decanted from the precipitated element and the precipitate was washed with THF (3×50 mL). Then, five equivalents of the alkyl bromide were added and the reaction mixture was heated to 130 °C for 2 h. The orange suspension gradually turned into a colorless solution. The excess alkyl bromide was removed under vacuum.

$(H_3COOCC_2H_4)_2GeBr_2$: ^{1}H NMR (CD_3CN): $\delta = 2.08$ (t, $^3J(^1H,^1H) = 7.2$ Hz, 4H), 2.64 (t, $^3J(^1H,^1H) = 7.2$ Hz, 4H), 3.58 (s, 6H); ^{13}C NMR (CD_3CN): $\delta = 26.2$, 30.3, 52.9, 175.5; MS (EI): *m*/*z* (%): 377 (9) [$M-(MeO)^+$], 327 (85) [$M-Br^+$], 321 (100) [$MeOOCC_2H_4GeBr_2^+$]; yield: 82%, colorless oil.

$(H_3COOC_4H_8)_2GeBr_2$: ^{1}H NMR (CD_3CN): $\delta = 1.65$(m, 8H), 1.83 (t, $^3J(^1H,^1H) = 6.9$ Hz, 4H), 2.34 (t, $^3J(^1H,^1H) = 6.9$ Hz, 4H), 3.61 (s, 6H); ^{13}C NMR (CD_3CN): $\delta = 24.5$, 27.4, 28.2, 33.9, 52.0, 174.5; MS (EI): *m*/*z* (%): 431 (7) [$M-(MeO)^+$], 383 (16) [$M-Br^+$], 349 (56) [$MeOOCC_4H_8GeBr_2^+$], 115 (100) [$MeOOCC_4H_8^+$]; yield: 84%, colorless oil.

$(NCC_2H_4)_2GeBr_2$: ^{1}H NMR (CD_3CN): $\delta = 2.20$ (t, $^3J(^1H,^1H) = 8.1$ Hz), 4H), 2.73 (t, $^3J(^1H,^1H) = 8.1$ Hz, 4H); ^{13}C NMR(CD_3CN): $\delta = 13.7$, 23.2, 120.4; MS (EI): *m*/*z* (%): 342 (3) [M^+], 288 (100) [$CC_2H_4GeBr_2^+$], 153 (49) [$GeBr^+$]; yield: 94%, colorless solid.

$(NCC_3H_6)_2GeBr_2$: ^{1}H NMR (CD_3CN): $\delta = 1.86$ (m, 8H), 2.45 (m, 4H); ^{13}C NMR (CD_3CN): $\delta = 19.1$, 21.4, 26.6, 120.2; MS (EI): *m*/*z* (%): 369 (1) [M^+], 342 (3) [$M-(CN)^+$], 302 (100) [$CC_3H_6GeBr_2^+$], 289 (52) [$(NCC_3H_6)_2GeBr^+$]; yield: 85%, colorless solid.

$(ClC_4H_8)_2GeBr_2$: ^{1}H NMR (CD_3CN): $\delta = 1.92-2.04$ (m, 8H), 2.05–2.11 (m, 4H), 3.64 (t, $^3J(^1H,^1H) = 6.7$ Hz, 4H); ^{13}C NMR (CD_3CN): $\delta = 26.4$, 36.9, 40.8, 45.2; MS (EI): *m*/*z* (%): 381 (3) [M^+-Cl], 325 (25) [$ClC_4H_8GeBr_2^+$], 289 (23) [$C_4H_8GeBr_2^+$], 243 (10) [$ClC_4H_8GeBr^+$], 55 (100) [$C_4H_7^+$]; yield: 84%, colorless oil.

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