Reactions of polychlorophenyllithium compounds with electrophiles*

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Polychloroaromatic compounds lithiated by BuⁿLi in THF react with several electrophilic agents of which aldehydes and epoxides seem to be the most promising from the preparative point of view.

Key words: polychloroarenes, hexachlorobenzene, *n*-butyllithium, organolithium compounds, carbonyl compounds, aldehydes, epoxides.

Polychloroaromatic compounds, such as insecticides of the first generation and polychlorodioxines, are ecologically hazardous materials. The most popular method for their chemical utilization is the catalytic hydrogenolysis of the C–Cl bond.¹ Other chemical transformations of polychloroarenes are little studied. At the same time, the development of methods for substitution of C–C and C–Het (Het is a heteroatom) bonds for C–Cl bonds usually of low reactivity can provide wide potentialities for polychloroarene processing to practically useful aromatic compounds. Available literature data indicate the mutual activating effect of chlorine atoms, due to which they undergo nucleophilic aromatic substitution (see, *e.g.*, Ref. 2).

A more rare type of reactions of polychloroarenes is the exchange of chlorine by a metal to give the corresponding organometallic compounds. For instance, they react with alkyllithium reagents (BuⁿLi, Bu^tLi) affording the expected polychlorophenyllithium derivatives,^{3–7} unlike monochloroarenes that are stable toward the action of AlkLi.⁸ Since the prepared organometallic compounds contain vicinal metal and chlorine atoms, these can be eliminated with formation of benzynes.⁹ Nevertheless, polychloroaryllithium derivatives are stable at -50 °C, whereas C₆Cl₅MgCl is stable even in boiling ether.¹⁰

The purpose of the present work is a comparative study of lithiation of hexachlorobenzene (1), 1,2-dimethoxytetrachlorobenzene (2), and compounds 3 and 4 containing hydrogen atoms in the aromatic ring and further reactions of the resulting $Ar^{Cl}Li$ with electrophiles. Different carbonyl compounds, halogenating agents, and methyloxirane were used as electrophiles. It should be noted that the reactions of $Ar^{Cl}Li$ with D_2O , CO_2 , $HgCl_2$, Me_3SiCl , and some other organoelement reagents are documented, ^{3-5,7} whereas carbonyl compounds as electrophiles are virtually unexplored.⁶



The interaction of hexachlorobenzene (1) with BuⁿLi in THF results in the Cl-Li exchange to form pentachlorophenyllithium (cf. Refs 3-5). We studied the reactions of *in situ* prepared C₆Cl₅Li with several electrophiles (Scheme 1, Table 1). Aldehydes with different structures add C_6Cl_5Li affording the corresponding secondary alcohols 5a-c in preparative yields (entries 1-3). The reactions of C₆Cl₅Li with ketones afford no tertiary alcohols. In this case, the main product is pentachlorobenzene C₆HCl₅ ($\delta_{\rm H}$ 7.54, [M]⁺ 248), which results from protonation of C_6Cl_5Li with either mobile α -H atoms of enolizable ketones or water during work-up (for instance, in the case of benzophenone that remains unchanged in the reaction). The reactions of benzoyl chloride (entry 4) and methyl benzoate (entry 5) even with excess C_6Cl_5Li stop at the step of formation of 2,3,4,5,6-pentachlorobenzophenone **6b** (yields 7–9%). N-Methoxy-N-methyl-

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R = Me (5a, 6a), Ph (5b, 6b), CH=CHMe (5c); Hal = Br (9a), I (9b)

Table 1. Reactions of lithiated polychloroaromatic compounds with electrophiles (THF, arene : $Bu^{n}Li \sim 1 : 0.85$)

Entry	Start-	Electrophile	Pro-	Yield
	ing	(molar ratio	duct	(%)
	arene	to arene)		
1	1	MeCHO (0.8)	5a	52
2	1	PhCHO (1.0)	5b	81
3	1	MeCH=CHCHO (1.0)	5c	68
4	1	PhC(O)Cl (0.32)	6b	9
5	1	$PhCO_{2}Me(0.32)$	6b	7
6	1	Methyloxirane ^{a} (1.2)	7	46
7	1	ClC(0)OEt (0.5 and 1.3)	8	34 and 18
8	1	BrCN (0.9)	9a	$\sim 45^{b}$
9	1	$I_2(0.8)$	9b	41
10	2	MeCHO (1.0)	10	17
11	3	MeCHO (1.6)	11a	51
12	3	PhCHO (1.2)	11b	23
13	3	PhCO ₂ Me (1.0 and 0.5)	12b	55 and 12
14	3	$PhC(\overline{O})Cl(0.5)$	12b	c
			13	12
15	4	MeCHO (1.0)	14	13

^{*a*} In the presence of 1 equiv. of HMPA.

^b According to the GLC data.

^c Not determined.

benzamide, which usually reacts with organolithium compounds to form the corresponding phenones,¹¹ does not react with C_6Cl_5Li . Like ketones, acetyl chloride acts as a proton donor in the reaction with C_6Cl_5Li to form considerable amounts of pentachlorobenzene. Methyloxirane reacts smoothly with C_6Cl_5Li in the presence of 1 equiv. of HMPA to form secondary alcohol 7 (entry 6). Ethyl chloroformate, like other derivatives of carboxylic acids, reacts with C_6Cl_5Li to afford, even with an excess of the latter, ethyl pentachlorobenzoate (8) as the main product (entry 7; however, according to the data from mass spectrometry, the reaction mixture contains certain amount of diethyl tetrachlorobenzenedicarboxylates with $[M]^+$ 358). Cyanogen bromide reacts with C₆Cl₅Li as a brominating agent to form bromopentachlorobenzene (9a) (entry 8). When C₆Cl₅Li is quenched with a solution of iodine, pentachloroiodobenzene (9b) is formed (entry 9). An attempt to synthesize pentachlorophenylboronic acid by the treatment of C₆Cl₅Li with trimethyl borate or triisopropyl borate was unsuccessful.

1,2-Dimethoxytetrachlorobenzene deactivated by two methoxy groups (2) is lithiated less efficiently than hexachlorobenzene (1) (Scheme 2, see Table 1). When acetaldehyde was used as a trap for $Ar^{Cl}Li$, 1-(3,4-dimethoxy-2,5,6-trichlorophenyl)ethan-1-ol (10) was obtained in a low yield (entry *10*). The position of the 1-hydroxyethyl substituent in the product (10) was inferred on the basis of 2D NMR experiments (NOESY and HMBC). The NOESY experiment showed no interaction between protons of the methoxy groups and CH(OH)Me fragment. No correlation between the C<u>H</u>(OH) proton and aromatic carbon atoms of =<u>C</u>—OMe were observed in the HMBC experiment but correlations of this proton

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with the carbon atoms of the =C-Cl fragment of the benzene ring were found. The direction of the attack of BuⁿLi on the chlorine atom in position 4 is related, most likely, to its greater spatial accessibility than that in position 3, although lithium can preliminarily be complexed by the oxygen atom in the case of the alternative lithiation route. Noticeable amounts of the dimerization products of polychloroarene 2 are formed in the reaction along with compound 10.

Scheme 2



Unlike polychloroarenes 1 and 2, 1,2,4,5-tetrachlorobenzene (3) and 3,4-dichloro-1-methoxybenzene (4) con-

taining hydrogen atoms in the benzene ring react with BuⁿLi with replacement of one or two hydrogen atoms by metal. Monolithium arene derivative **3** was identified following its reactions with aldehydes and methyl benzoate to give products **11** and **12** (Scheme 3, see Table 1, entries 11-13). The dilithium derivative was identified by the isolation of diketone **13**, although in low yield, when 0.5 equiv. of benzoyl chloride was added to the reaction mixture (entry 14). Similarly to C₆Cl₅Li, lithiated **3** does not react with ketones.

The lithiation product of dichloroarene **4** does not react with acetaldehyde; instead of the expected alcohol, biaryl **14** is formed (Scheme 4, see Table 4, entry *15*). The structure of compound **14** was established by 2D NMR experiments (COSY, HSQC, and HMBC). The HMBC experiment identified the junction carbon atom C(1) correlating with the protons H(5) (doublet) and H(6[°]) (singlet).

ortho-Deprotonation of aryl ethers by alkyllithium reagents is well known.¹² In our case, the preferential deprotonation of the proton from position 2, which is more hindered but activated by the adjacent chlorine, is noteworthy. Probably, aryllithium 15 that formed is less stable than organolithium derivatives of compounds 1-3 and, hence, it reacts with excess dichloroarene 4 substituting the chlorine atom in position 3, which is not deactivated by the mesomeric effect of the methoxy group. An alternative dimerization mechanism including the formation of 6-chloro-3-methoxybenzyne (16) and addition of aryllithium 15 is also probable.

Scheme 3



R = Me (11a, 12a), Ph (11b, 12b)





The reaction of the lithiation products of 1,2-dichlorobenzene with MeCHO affords a complex mixture of nonpolar compounds with low volatilities, probably, of the biaryl type. We failed to isolate individual components from this mixture.

Thus, for the first time we performed a comparative study of the lithiation processes of polychloroaromatic compounds of different structures and the reactivities of aryllithium derivatives that formed. The preparative value of the studied reactions is determined, in particular, by the reaction of Ar^{Cl}Li with aldehydes and epoxides. This process can be considered as a method for the preparation of secondary alcohols containing polychloroaromatic fragments. The latter can serve as intermediates in the syntheses of polychlorinated ketones by oxidation, while these compounds are poorly accessible by other methods.

Experimental

Melting points were not corrected. Standard NMR spectra were recorded on a Bruker AM-300 spectrometer in CDCl₃ at 300.13 MHz (¹H) and 75.47 MHz (¹³C). 2D NMR experiments (COSY, NOESY, HSQC, and HMBC) were carried out on a Bruker DRX-500 AVANS spectrometer at 500.13 MHz (¹H) and 125.76 MHz (¹³C) in DMSO-d₆ at 30 °C. All NMR experiments were performed using standard Bruker procedures. 2D HSQC and HMBC spectra were recorded using a gradient procedure. Mass spectra were obtained on a Kratos MS-30 instrument (ionizing voltage 70 eV) with the direct sample inlet and heating $0\rightarrow$ 220 °C; mass numbers are presented for the ³⁵Cl and ⁷⁹Br isotopes. Elemental analyses were carried out at the Analytical Laboratory of the N. D. Zelinsky Institute of Organic Chemistry (Russian Academy of Sciences). Hexane, AcOEt, and CH₂Cl₂ were pre-distilled; THF was dried by refluxing over

Table 2. Physicochemical characteristics of the synthesized compounds

Compound	NMR (CDCl ₃), δ (J/Hz)		M.p./°C	Composition ^a
	¹ H	¹³ C	(literature data)	
1-(Pentachlorophenyl)- ethan-1-ol (5a)	1.67 (d, 3 H, Me, $J = 8$); 3.00 (d, 1 H, OH, $J = 10$); 5.69 (br.quintet, 1 H, OCH, J = 8)	20.9 (Me); 69.8 (CH); 131.5, 132.6, 132.9, 140.0 (all C)	124—126 (123—124, ⁶ 126 ¹³)	_
2,3,4,5,6-Pentachloro- benzhydrol (5b)	3.43 (d, 1 H, OH, <i>J</i> = 10); 6.76 (d, 1 H, OCH, <i>J</i> = 10); 7.20–7.42 (m, 5 H, Ph)	73.9, 125.2, 127.7 (all CH); 132.9, 133.0, 133.8, 139.2, 140.2 (all C)	114—116 (117 ¹⁴)	_
1-(2,3,4,5,6- Pentachlorophenyl)but- 2-en-1-ol (5c)	1.75 (d, 3 H, Me, $J = 6$); 3.04 (d, 1 H, OH, $J = 10$); 5.65–6.10 (m, 3 H, OCH and CH=CH)	17.9 (Me); 73.8, 128.7, 130.0 (all CH); 132.0, 132.8, 133.1, 138.7 (all C)	104—106	Found (%): C, 37.26; H, 2.33. $C_{10}H_7Cl_5O.$ Calculated (%): C, 37.48; H, 2.20
2,3,4,5,6-Pentachloro- acetophenone (6a)	2.60 (s, 3 H, Me)	30.5 (Me); 127.6, 132.5, 134.4, 139.9, 196.7 (all C)	90—92 (90 ¹⁵)	_
2,3,4,5,6-Pentachloro- benzophenone (6b)	7.53 (t, 2 H, CH, <i>J</i> = 8); 7.71 (t, 1 H, CH, <i>J</i> = 7.3); 7.86 (d, 2 H, CH, <i>J</i> = 8)	129.3 (CH); 129.4 (C); 129.7 (CH); 132.7, 134.4, 134.8 (all C); 134.9 (CH); 138.0, 189.9 (both C)	149—151 (154 ¹⁴)	[M] ⁺ 352

(to be continued)

Compound	NMR (CDCl ₃),	M.p./°C	Composition ^a	
	¹ H	¹³ C	(literature data)	
1-(Pentachlorophenyl)- propan-2-ol (7)	1.36 (d, 3 H, Me, $J = 5.9$); 1.53 (s, 1 H, OH); 3.16 (dd, 1 H, CH, $J = 5.6$, J = 12.9); 3.31 (dd, 1 H, CH, $J = 7.1$, $J = 12.9$); 4.24 (settet OCH $J = 7.6$)	23.9 (Me); 42.8 (CH ₂); 67.4 (CH); 132.1, 133.6, 136.6 (all C)	119—120	Found (%): C, 35.44; H, 2.21. C ₉ H ₇ Cl ₅ O. Calculated (%): C, 35.05; H, 2.29
Ethyl pentachloro- benzoate (8)	$\begin{array}{l} 4.24 \text{ (state, OCH, J = 7.60 \\ 1.43 \text{ (t, 3 H, Me, } J = 8); \\ 4.50 \text{ (q, 2 H, OCH}_2, J = 6.7) \end{array}$	14.1 (Me); 63.0 (CH ₂); 129.4, 132.4, 134.3, 135.0, 162.9 (all C)	75—76	[M] ⁺ 320
1-Bromo-2,3,4,5,6-penta- chlorobenzene ^b (9a)	No signals	123.8 (C–Br); 132.1, 133.0, 138.1 (all C)		[M] ⁺ 326
1,2,3,4,5-Pentachloro- 6-iodobenzene ^b (9b)	No signals	103.2 (C-1); 130.6, 134.1, 138.1 (all C)	$186 - 188^{a}$ (207 ¹⁶)	
1-(3,4-Dimethoxy- 2,5,6-trichlorophenyl)- ethan-1-ol (10)	1.63 (d, 3 H, Me, $J = 6.7$); 3.12 (d, 1 H, OH, $J = 10$); 3.89 (s, 3 H, OMe); 3.91 (s, 3 H, OMe); 5.58 (br. quintet, 1 H, OCH, $J = 6.0$)	21.3 (Me); 61.1 (2 OMe); 69.0 (CH); 126.9, 127.3, 127.7, 136.1, 149.5, 150.1 (all C)	Oil	[M] ⁺ 284
1-(2,3,5,6-Tetrachloro- phenyl)ethan-1-ol (11a)	1.64 (d, 3 H, Me, $J = 7$); 3.00 (d, 1 H, OH, $J = 8.1$); 5.68 (br.quintet, 1 H, OCH, J = 7.4); 7.56 (s, 1 H, OCH)	20.9 (Me); 69.7, 129.9 (both CH); 130.7, 133.0, 142.2 (all C)	78—80	Found (%): C, 36.97; H, 2.18. $C_8H_6Cl_4O$. Calculated (%): C 36.96; H 2.33
2,3,5,6-Tetrachloro- benzhydrol (11b)	3.44 (d, 1 H, OH, <i>J</i> = 8.4); 6.66 (d, 1 H, OCH, <i>J</i> = 11.1); 7.20–7.43 (m, 5 H, Ph); 7.64 (s, 1 H, =CH)	73.9, 125.3, 127.7, 128.6, 130.6 (all CH); 132.2, 133.1, 140.4, 141.6 (all C)	88—89	Found (%): C, 48.22; H, 2.39. $C_{13}H_8Cl_4O.$ Calculated (%): C, 48.49; H, 2.50
2,3,5,6-Tetrachloro- acetophenone (12a)	2.58 (s, 3 H, Me); 7.59 (s, 1 H, CH)	30.6 (Me); 127.0 (C); 131.1 (CH); 132.6, 142.4, 197.5 (all C)	55-57	_
2,3,5,6-Tetrachloro- benzophenone (12b)	7.51 (t, 2 H, CH, <i>J</i> = 8.3); 7.65 (t, 2 H, CH, <i>J</i> = 8.8); 7.71 (s, 1 H); 7.83 (d, 2 H, CH, <i>J</i> = 9.3)	128.7 (C); 129.2, 129.7, 131.4 (all CH); 132.7, 134.5 (both C); 134.8 (CH); 140.4, 190.5 (both C)	129	[M] ⁺ 318
1,4-Dibenzoyl-2,3,5,6- tetrachlorobenzene (13)	7.58 (t, 4 H, CH, <i>J</i> = 8.3); 7.69 (t, 2 H, CH, <i>J</i> = 8.3); 7.90 (2 H, CH, <i>J</i> = 9.0)	129.4 (CH); 129.7 (C); 129.8 (CH); 134.2 (C); 135.1 (CH); 140.4, 190.3 (both C)	212	[M] ⁺ 422
2,2',3-Trichloro-5',6- dimethoxybiphenyl ^c (14)	3.72 (s, 3 H, 6-OMe); 3.78 (s, 3 H, 5'-OMe); 6.84 (br.s, 1 H, H(6')); 7.01 (br.d, 1 H, H(4'), $J = 8.3$); 7.18 (d, 1 H, H(4), $J = 8.7$); 7.43 (d, 1 H, H(3'), $J = 8.3$); 7.64 (d, 1 H, H(5), $J = 8.7$)	55.5, 56.3 (both OMe); 111.6 (CH(5)); 115.2 (CH(4')) 116.8 (CH(6')); 122.9 (C(3)); 124.0 (C(2')); 128.5 (C(1)); 129.8 (CH(3')); 130.3 (CH(4)) 131.2 (C(2)); 135.0 (C(1')); 156.3 (OC(6)); 158.0 (OC(5'))	122—124););	[M] ⁺ 316

Table 2 (continued)

^{*a*} Data from mass spectrometry (m/z) or elemental analysis.

 b The samples contained C₆Cl₆ (δ_{C} 132.5) in amounts of 55 (**9a**) and 20% (**9b**).

^c The NMR spectra were recorded in DMSO-d₆.

LiAlH₄ followed by distillation under argon. Hexachlorobenzene (1) (Aldrich), 1,2,4,5-tetrachlorobenzene (3) (Acros), and cyanogen bromide were used as received. Acetaldehyde was prepared by detrimerization of paraldehyde with H_3PO_4 , a fraction with b.p. 20-21 °C was collected through a high Vigreux

column. Benzaldehyde, methyl benzoate, benzoyl chloride, acetyl chloride, crotonaldehyde, and ethyl chloroformate were freshly distilled. *N*-Methoxy-*N*-methylbenzamide was synthesized by the reaction of benzoyl chloride with N,O-dimethylhydroxylamine hydrochloride in the presence of 2 equiv. of pyri-

dine and 1 equiv. of triethylamine in CH_2Cl_2 according to a known procedure.¹¹ 1,2-Dimethoxytetrachlorobenzene (**2**) and 3,4-dichloro-1-methoxybenzene (**4**) were synthesized by methylation of tetrachloropyrocatechol (Lancaster) and 3,4-dichlorophenol (Aldrich), respectively, with excess of MeI and K₂CO₃ in boiling acetone. TLC analysis was carried out on aluminum plates with Kieselgel 60 F₂₅₄ (Merck, 1.05554). Column chromatography was carried out on silica gel L (100/160).

Lithiation of polychloroarenes followed by the reaction with electrophiles (general procedure). A 1.7 M solution of BuⁿLi (2.5 mL, ~4.25 mmol) in hexane (Merck) was added dropwise for 5 min at -50 °C under argon to a mixture of polychloroarene (5 mmol) in anhydrous THF (25 mL) (hexachlorobenzene was not completely dissolved). The mixture was kept for 20 min. During this time, the solution became gray. Then the mixture was cooled to -78 °C, and a solution of the corresponding electrophile (2–5 mmol, see Table 1) in THF (3 mL) was added. Cooling was discontinued, and the mixture was warmed to room temperature. The mixture was quenched with a solution of sodium hydrogencarbonate, the organic layer was separated, the aqueous layer was extracted with ether, and the combined extracts were dried with MgSO₄ and concentrated in vacuo. The residues were chromatographed on silica gel (gradient $10 \rightarrow 100\%$ CH_2Cl_2 in hexane). Compounds 5–14 were thus obtained (Tables 1 and 2).

Ketones 6a,b and 12a,b (independent synthesis). A solution of $Na_2Cr_2O_7$ (1.5 g) and H_2SO_4 (1.13 mL) in water (7.5 mL) was added for 10 min to a solution of the corresponding secondary alcohol (15 mmol) in ether (4 mL), and the mixture was stirred for 2–3 h (TLC monitoring). The ethereal layer was separated, the aqueous layer was extracted with ether, and the combined extracts were dried with CaCl₂ and concentrated *in vacuo*. Ketones were isolated by crystallization or, if necessary, column chromatography in 51–99% yields.

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