The Reaction of Lithium α-Lithiocarboxylates (Dianions of Carboxylic Acids) or α-Lithiocarboxylic Esters (Ester Enolates) with α-Halocarboxylic Esters or Lithium α-Halocarboxylates

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Since it has been found that α-carbanions of carboxylic acids and ester enolates can be prepared under mild conditions by the use of non-nucleophilic bases such as disubstituted lithium amides[1,2,3], their synthetic utility has been widely explored. Reactions of these intermediates with alkyl halides[1-11], dihaloalkanes[1,6,9,12,13], epoxides[1], halogen[14,15], carbon tetrahalides[16], silyl halides[17], sulfenylation[18,19,20] and selenylation[20,21,22] reagents, chlorophosphates[20], alkyl nitrates[23], ammonia derivatives[24], aldehydes and ketones[25-33], carbon dioxide[34], esters[23,35,36], acyl chlorides[37,38], carbonochloridates[20,39], anhydrides[40], α,β-unsaturated carbonyl compounds[33,36], and oxygen[41-44] have been described. However, the (very simple) application of such reactions to the preparation of succinate derivatives via alkylation of acid dianions and ester enolates with α-halocarboxylates and α-haloesters has not been widely exploited.

In continuation of our studies on ester enolate chemistry[15,20,28], we report here the results of the investigation of the following four reaction types:

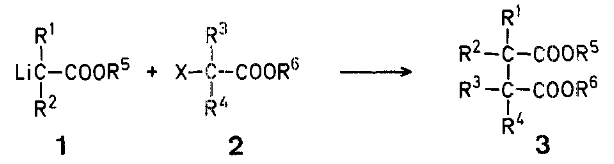
(1) Reaction of lithium α-lithiocarboxylates with lithium α-halocarboxylates;

(2) Reaction of α-lithiocarboxylic esters with lithium α-halocarboxylates;

(3) Reaction of α-lithiocarboxylic esters with α-halocarboxylic esters;

(4) Reaction of lithium α-lithiocarboxylates with α-halocarboxylic esters.

In spite of some limitations due to the occurrence of side reactions, the above reactions *(1)*-*(4)* have been found to be widely applicable to the synthesis of succinic acid derivatives (3). Hitherto, these derivatives have usually been prepared via alkylation of malonic esters, i.e., by a route involving a decarboxylation step[45,46]. Previous attempts to prepare succinic acid esters (3, R^5 = alkyl, R^6 = H, alkyl) by reaction of ester enolates with α-halocarboxylic esters or metal α-halocarboxylates had only given poor results[47,48].



1 2 3

(1) $R^5 = R^6 = Li$

(2) R^5 = alkyl ; R^6 = Li

(3) R^5, R^6 = alkyl

(4) R^5 = Li ; R^6 = alkyl

Due to the stability of dianions of carboxylic acids (in 1, $R^5 = Li$) even at a reaction temperature of 45 °C and to the inertness of the carboxylate group (in 1 and 2) towards nucleophilic reagents, *reaction (1)* affords the best results without significant dependence of the yields on the degree of α-substitution either at the nucleophilic (in 1) or the electrophilic center (in 2).

Reaction (2) requires milder conditions to avoid decomposition of the ester enolate (1, R^5 = alkyl), and it shows limitations in the case of the reaction of *t*-butyl lithioacetate with compounds 2 having a sterically highly hindered electrophilic center. Ethyl 2-lithiobutanoate, which lacks a protective bulky ester group, undergoes the desired reaction only with lithium bromoacetate; with lithium 2-bromopropanoate, which has a secondary electrophilic center, the reaction does not proceed and self-condensation of 1 occurs to a minor degree (10% by N.M.R. analysis).

Reaction (3) shows only slight dependence on steric factors. In the case of methyl 2-lithiohexanoate, the reaction with α-iodocarboxylic esters gives better results than with the bromo analogs; the yields decrease with increasing steric hindrance at the electrophilic center and the formation of β-oxocarboxylic esters (condensation products) is observed. In the light of these results, we reinvestigated the reaction of *t*-butyl lithioacetate with *t*-butyl bromoacetate under our reaction conditions and obtained a 58% yield of di-*t*-butyl succinate (**3u**) whereas the reported[47] yield was only 10%.

In *reaction (4)*, we used lithium 2-lithio-2-methylpropanoate (the dianionic derivative of isobutyric acid) as a model compound. With this nucleophile, we obtained a good yield (70%) of the corresponding succinic monoester only from the reaction with *t*-butyl bromoacetate, a complex mixture of products being formed in the other cases.

Melting points were taken on a Kofler block and are uncorrected. Boiling points refer to bulb-to-bulb destillation (Kugelrohr). G.L.C. analyses were performed on a Varian 2800 instrument equipped with flame-ionization detectors and a 6′ × 1/8″ column packed with 3% OV-17 on Varaport 30 (100/120 mesh) with nitrogen as carrier gas. ^{1}H-N.M.R. spectra were recorded at 60 MHz on a Varian T-60 spectrometer (TMS as internal standard).

All the reactions are performed in a nitrogen atmosphere.

Succinic Acids and Esters (3) by *CC* Coupling of α-Lithiocarboxylic Acid Derivatives (1) with α-Halocarboxylic Acid Derivatives (2); General Procedure:

Lithium Diisopropylamide Solution: A 2 molar solution (5 ml, 10 mmol) of butyllithium in hexane is added to a stirred mixture of

0039-7881/80/0932-0710 $ 03.00

Table. *CC* Coupling of α-Lithiocarboxylic Acid Derivatives (**1**) with α-Halocarboxylic Acid Derivatives (**2**) to give Succinic Acids or Esters (**3**)

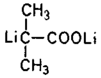
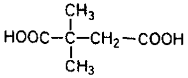
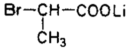
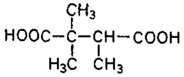
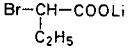
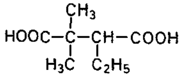
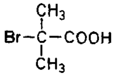
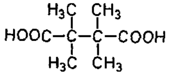
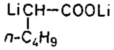
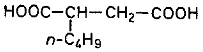
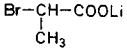
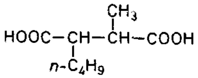
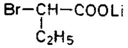
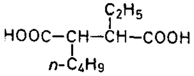
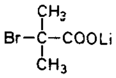
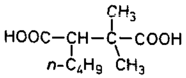
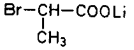
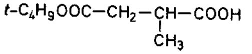
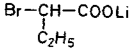
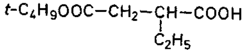
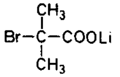
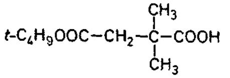
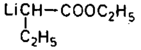
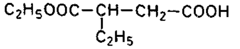
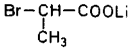
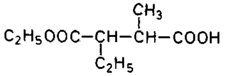
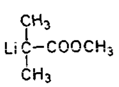
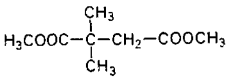
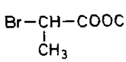
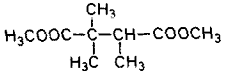
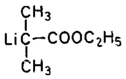
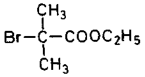
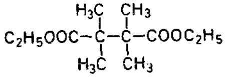
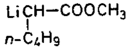
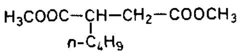
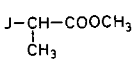
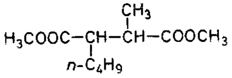
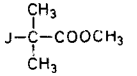
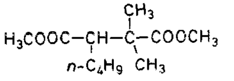
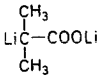
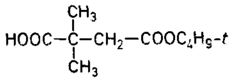
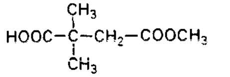
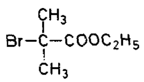
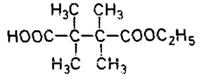
Reaction	1	2	3		Yield[a] [%]	m.p. [°C] or b.p. [°C]/torr	Molecular formula[c] or physical data [°C] reported	^{1}H-N.M.R. δ [ppm]
(1)	 $LiC(CH_3)_2-COOLi$	$Br-CH_2-COOLi$	 $HOOC-C(CH_3)_2-CH_2-COOH$	**3a**	63	137–138°	137–138°[49]	(F_3C—COOH): 2.90 (s, 2H); 1.47 (s, 6H)
		 $Br-CH(CH_3)-COOLi$	 $HOOC-C(CH_3)_2-CH(CH_3)-COOH$	**3b**	73	149–150°	148–149°[50]	(F_3C—COOH): 3.18 (q, 1H); 1.41 (s, 6H); 1.38 (d, 3H)
		 $Br-CH(C_2H_5)-COOLi$	 $HOOC-C(CH_3)_2-CH(C_2H_5)-COOH$	**3c**	56	140–141°	139°[51]	(F_3C—COOH): 2.90 (q, 1H); 1.50–2.00 (m, 2H); 1.44 (s, 6H); 1.07 (t, 3H)
		 $Br-C(CH_3)_2-COOH$	 $HOOC-C(CH_3)_2-C(CH_3)_2-COOH$	**3d**	78	194–196°	192°[52]	(F_3C—COOH): 1.38 (s, 12H)
	 $LiCH(n\text{-}C_4H_9)-COOLi$	$Br-CH_2-COOLi$	 $HOOC-CH(n\text{-}C_4H_9)-CH_2-COOH$	**3e**	74	79–81°	81°[53]	($CDCl_3$): 10.90 (s, 2H); 2.37–3.22 (m, 3H); 1.08–1.92 (m, 6H); 0.92 (t, 3H)
		 $Br-CH(CH_3)-COOLi$	 $HOOC-CH(n\text{-}C_4H_9)-CH(CH_3)-COOH$	**3f**	67	140–141°	$C_9H_{16}O_4$ (188.2)	(F_3C—COOH): 2.80–3.30 (m, 2H); 1.17–2.17 (m, 6H); 0.84–1.19 (m, 6H)
		 $Br-CH(C_2H_5)-COOLi$	 $HOOC-CH(n\text{-}C_4H_9)-CH(C_2H_5)-COOH$	**3g**	60	135–136°	$C_{10}H_{18}O_4$ (202.3)	(F_3C—COOH): 2.72–3.05 (m, 2H); 1.25–2.25 (m, 8H); 1.07 (t, 6H)
		 $Br-C(CH_3)_2-COOLi$	 $HOOC-CH(n\text{-}C_4H_9)-C(CH_3)_2-COOH$	**3h**	47	125–126°	oil[54]	($CDCl_3$): 10.73 (s, 2H); 2.60–2.96 (m, 1H); 1.12–1.78 (m, 12H); 0.90 (t, 3H)
(2)	$LiCH_2-COOC_4H_9\text{-}t$	$Br-CH_2-COOLi$	$t\text{-}C_4H_9OOC-CH_2-CH_2-COOH$	**3i**	75	49–51°	51.5–52°[55]	(CCl_4): 11.04 (s, 1H); 2.47–2.63 (m, 4H); 1.44 (s, 9H)
		 $Br-CH(CH_3)-COOLi$	 $t\text{-}C_4H_9OOC-CH_2-CH(CH_3)-COOH$	**3j**	51	38–40°	$C_9H_{16}O_4$ (188.2)	(CCl_4): 11.40 (s, 1H); 2.13–3.05 (m, 3H); 1.43 (s, 9H); 1.24 (d, 3H)
		 $Br-CH(C_2H_5)-COOLi$	 $t\text{-}C_4H_9OOC-CH_2-CH(C_2H_5)-COOH$	**3k**	52	105–110°/0.75	$C_{10}H_{18}O_4$ (202.3)	(CCl_4): 11.70 (s, 1H); 2.11–3.00 (m, 3H); 1.20–1.90 (m, 2H); 1.43 (s, 9H); 0.99 (t, 3H)
		 $Br-C(CH_3)_2-COOLi$	 $t\text{-}C_4H_9OOC-CH_2-C(CH_3)_2-COOH$	**3l**	—			
	 $LiCH(C_2H_5)-COOC_2H_5$	$Br-CH_2-COOLi$	 $C_2H_5OOC-CH(C_2H_5)-CH_2-COOH$	**3m**	54[d]	80–85°/0.75	$C_8H_{14}O_4$ (174.2)	(CCl_4): 11.17 (s, 1H); 4.13 (q, 2H); 2.25–2.97 (m, 3H); 1.40–1.78 (m, 2H); 1.26 (t, 3H); 0.94 (t, 3H)
		 $Br-CH(CH_3)-COOLi$	 $C_2H_5OOC-CH(C_2H_5)-CH(CH_3)-COOH$	**3n**	—			

Table. (continued)

Reaction	1	2	3		Yield[a] [%]	m.p. [°C] or b.p. [°C]/torr	Molecular formula[c] or physical data [°C] reported	^{1}H-N.M.R. δ [ppm]
(3)	 $\mathrm{LiC(CH_3)_2-COOCH_3}$	$\mathrm{Br-CH_2-COOCH_3}$	 $\mathrm{H_3COOC-C(CH_3)_2-CH_2-COOCH_3}$	**3o**	77[b]	25–30°/13	201–202°/760[56]	(CCl$_4$): 3.67 (s, 3H); 3.63 (s, 3H); 2.50 (s, 2H); 1.24 (s, 6H)
		 $\mathrm{Br-CH(CH_3)-COOCH_3}$	 $\mathrm{H_3COOC-C(CH_3)_2-CH(CH_3)-COOCH_3}$	**3p**	75[b,e]	55–60°/11	$C_9H_{16}O_4$ (188.2)	(CCl$_4$): 3.64 (s, 3H); 3.62 (s, 3H); 2.85 (q, 1H); 1.17 (s, 6H)
	 $\mathrm{LiC(CH_3)_2-COOC_2H_5}$	 $\mathrm{Br-C(CH_3)_2-COOC_2H_5}$	 $\mathrm{C_2H_5OOC-C(CH_3)_2-C(CH_3)_2-COOC_2H_5}$	**3q**	70	45–50°/48	219°/760[57]	(CCl$_4$): 4.06 (q, 4H); 1.25 (t, 6H); 1.18 (s, 12H)
	 $\mathrm{LiCH(\mathit{n}\text{-}C_4H_9)-COOCH_3}$	$\mathrm{J-CH_2-COOCH_3}$	 $\mathrm{H_3COOC-CH(\mathit{n}\text{-}C_4H_9)-CH_2-COOCH_3}$	**3r**	75[b]	42–47°/11	100°/6[58]	(CCl$_4$): 3.66 (s, 3H); 3.63 (s, 3H); 2.17–2.85 (m, 3H); 1.07–1.87 (m, 6H); 0.92 (t, 3H)
		 $\mathrm{J-CH(CH_3)-COOCH_3}$	 $\mathrm{H_3COOC-CH(\mathit{n}\text{-}C_4H_9)-CH(CH_3)-COOCH_3}$	**3s**	42[b]	28–33°/0.2	120–130° (bath)/0.5[59]	(CCl$_4$): 3.65 (s, 6H); 2.42–2.87 (m, 2H); 0.77–1.62 (m, 12H)
		 $\mathrm{J-C(CH_3)_2-COOCH_3}$	 $\mathrm{H_3COOC-CH(\mathit{n}\text{-}C_4H_9)-C(CH_3)_2-COOCH_3}$	**3t**	34[b,f]	33–38°/0.1	$C_{12}H_{22}O_4$ (230.3)	(CCl$_4$): 3.63 (s, 6H); 2.55–2.82 (m, 1H); 1.15–1.52 (m, 6H); 1.17 (s, 3H); 1.13 (s, 3H); 0.80–1.02 (m, 3H)
	$\mathrm{LiCH_2-COOC_4H_9\text{-}\mathit{t}}$	$\mathrm{Br-CH_2-COOC_4H_9\text{-}\mathit{t}}$	$\mathrm{\mathit{t}\text{-}C_4H_9OOC-CH_2-CH_2-COOC_4H_9\text{-}\mathit{t}}$	**3u**	58	32–33°	31.5–35°[60]	(CCl$_4$): 2.40 (s, 4H); 1.42 (s, 18H)
(4)	 $\mathrm{LiC(CH_3)_2-COOLi}$	$\mathrm{Br-CH_2-COOC_4H_9\text{-}\mathit{t}}$	 $\mathrm{HOOC-C(CH_3)_2-CH_2-COOC_4H_9\text{-}\mathit{t}}$	**3v**	70	77–79°	$C_{10}H_{18}O_4$ (202.3)	(CCl$_4$): 11.63 (s, 1H); 2.47 (s, 2H); 1.42 (s, 9H); 1.27 (s, 6H)
		$\mathrm{Br-CH_2-COOCH_3}$	 $\mathrm{HOOC-C(CH_3)_2-CH_2-COOCH_3}$	**3w**	10	30–35°/0.2	$C_7H_{12}O_4$ (160.2)	(CCl$_4$): 11.48 (s, 1H); 3.62 (s, 3H); 2.54 (s, 2H); 1.28 (s, 6H)
		 $\mathrm{Br-C(CH_3)_2-COOC_2H_5}$	 $\mathrm{HOOC-C(CH_3)_2-C(CH_3)_2-COOC_2H_5}$	**3x**	—			

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[a] Yield of isolated pure compound if not stated otherwise.

[b] The yield was determined by G.L.C. using as internal standard an authentic sample of the ester, prepared by esterification of the corresponding succinic acid with diazomethane.

[c] The microanalyses of the new compounds were in satisfactory agreement with the calculated values: C, ±0.23; H, ±0.29.

[d] Hydrolysis of the product with 10% aqueous sodium hydroxide gave ethylsuccinic acid; m.p. 97 °C (Ref.[61], m.p. 98 °C).

[e] Hydrolysis of the product with 20% methanolic sodium hydroxide gave trimethylsuccinic acid (**3h**); m.p. 151 °C. Reesterification with diazomethane gave **3p**.

[f] Hydrolysis of the product with 20% methanolic sodium hydroxide gave 3-butyl-2,2-dimethylsuccinic acid (**3h**); m.p. 125–126 °C. Reesterification with diazomethane gave **3t**.

diisopropylamine (1.012 g, 10 mmol) and anhydrous tetrahydrofuran (10 ml) and the mixture is held at 0 °C for 15 min.

Lithium α-Lithiocarboxylates (**1**, $R^5 = Li$): The carboxylic acid (5 mmol) is added, at 0 °C with stirring, to the lithium diisopropylamide solution (10 mmol). Stirring is continued for 15 min at 0 °C and for 1 h at 45 °C.

Alkyl α-Lithiocarboxylates (**1**, $R^5 =$ alkyl): The alkyl carboxylate (5 mmol) is added, at −78 °C with stirring, to the lithium diisopropylamide solution (5 mmol) and stirring is continued for 40 min at −78 °C.

Lithium α-Bromocarboxylates (**2**, $R^6 = Li$): The α-bromocarboxylic acid (5 mmol) is added with stirring to lithium hydride (48 mg, 6 mmol) in anhydrous tetrahydrofuran (5 ml) at 0 °C.

Condensation Reaction: Hexamethylphosphoric triamide (HMPT; 0.90 g, 5 mmol) is added to the solution of compound **2** (5 mmol) and the resultant mixture is added to the solution of **1** (5 mmol) with stirring.

For *reaction (1):* Addition at 0 °C, stirring for 2 h at 35 °C.

For *reactions (2), (3), and (4):* Addition at −78 °C, stirring for 2 h at −78 °C.

Work-up:

Reactions (1), (2), and (4): A 1 normal hydrochloric acid solution (~15 ml) is saturated with sodium chloride and added to the stirred reaction mixture at 0 °C. The mixture is extracted with ether (3 × 20 ml). The extract is washed with saturated sodium chloride solution (20 ml) [washing with plain water is to be avoided because of the solubility of the products], dried with sodium sulfate, and evaporated. The residual crude product is purified by column chromatography on silica gel (70–230 mesh) using ether as eluent (HMPT is thus removed). The solid products are recrystallized from petroleum ether (30–50 °C) or ethyl acetate. Liquid products are distilled in vacuo.

Compound **3h** is purified by dehydration to the anhydride (b.p. 75–80 °C/5 torr) using phosphorus pentoxide, and subsequent hydrolysis to the acid.

Reaction (3): Saturated aqueous ammonium chloride (20 ml) is added to the stirred reaction mixture at 0 °C. The mixture is then diluted with water (20 ml) and extracted with ether (3 × 20 ml). The organic phase is washed with water (30 ml) and saturated sodium chloride solution (20 ml), dried with magnesium sulfate, and evaporated. The residual crude products are distilled in vacuo.

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