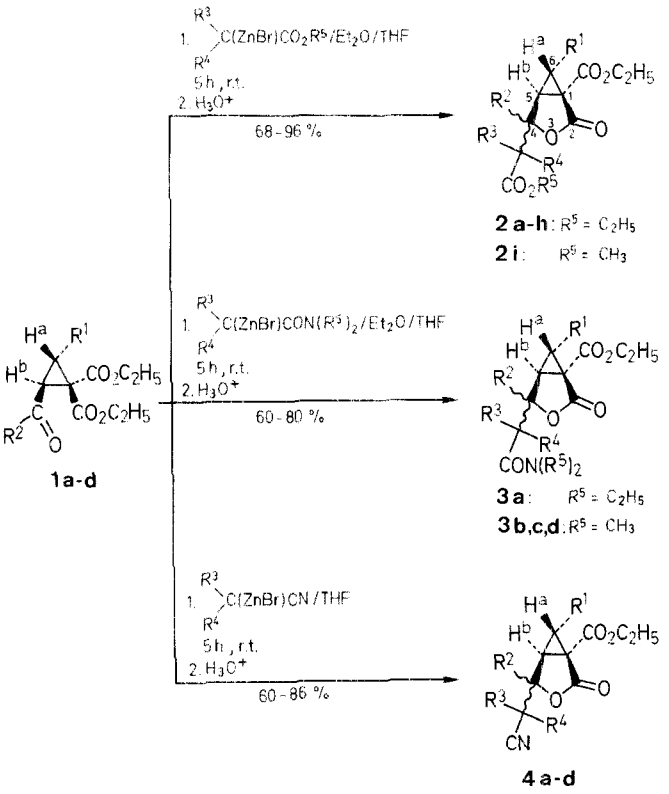

Synthesis of 4-Functionalized 1-Ethoxycarbonyl-2-oxo-3-oxabicyclo[3.1.0]hexanes by Reformatsky Reaction

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The title compounds are easily synthesized in good yields by reaction of the Reformatsky reagents derived from α-bromesters, α-bromocarboxamides, and α-bromonitriles with 2-acyl-3-phenyl(or 3-alkyl)-1,1-diethoxycarbonylcyclopropanes.

We have previously reported the synthesis and the structural elucidation of 1-ethoxycarbonyl-3-oxabicyclo[3.1.0]hexane-2-ones[1] which could be conveniently obtained from *trans*-2-acyl-3-methyl(or 3-phenyl)-1,1-diethoxycarbonylcyclopropanes **1**.

In connection with a pharmacological study of cyclopropane-fused γ-lactones we extended this methodology to the synthesis of new functionalized 3-oxabicyclo[3.1.0]hexane-2-ones (**2**, **3**, **4**) by reaction of organozinc reagents derived from α-bromesters, α-bromocarboxamides, and α-bromonitriles with the same compounds **1**.

In most of the cases shown in Table 1, the reaction is carried out in two steps (Method A): preparation of the intermediary Reformatsky reagent[2-5] and its addition to the carbonyl compound **1**. When the two-step procedure is not applicable (for example with the ester of α-bromophenylacetic acid[6]), the reaction may be carried out using the one-step Method B. Comparative experiments have shown that the reaction proceeds without changing the stereochemistry but that Method B affords slightly lower yields. We have thus prepared three new series of bicyclic lactones containing an unsubstituted or an alkyl(or phenyl)-substituted functional group, namely, an ethoxycarbonylmethyl group (**2**) or a dialkylaminocarbonylmethyl group (**3**) or a cyanomethyl group (**4**), in γ-position of the lactone ring. The IR and ^{1}H-NMR spectroscopic data listed in the Tables are in full agreement with the structures proposed for compounds **2**, **3**, and **4**.

As regards the stereochemistry of the products **2**, **3**, and **4**, the following considerations should be kept in mind:

- The *cis*-configuration at C-1 and C-5 is imposed by the lactone ring closure leading to the *cis*-fused 3-oxabicyclo[3.1.0]hexane system.
- The *exo*-position of the substituent R^1 (C-6) derives from the *trans*-configuration of the starting compounds **1**.
- The relative configuration at C-4 has been established for part of the products, namely **2a–d, g, i, 3a, b, e** and **4d** by ^{1}H-NMR spectrometry. As in the previous paper we name a *cis*-isomer that isomer, in which the smallest substituents (according to the sequence rules) at positions 4 and 5 are on the same side of the lactone ring.

The configuration of compounds **2b** and **3b** are assigned on the basis of the coupling constants for protons H^b and H^c. Thus, for **2b** we assign the *cis*-configuration to the isomer with J_{H^b,H^c} = 5.4 Hz and the *trans*-configuration to the isomer with $J_{H^bH^c}$ = 0 Hz. For **3b**, the *cis*-isomer has J_{H^b,H^c} = 4.0 Hz and the *trans*-isomer again J_{H^b,H^c} = 0 Hz.

2	R^1	R^2	R^3	R^4
a	CH_3	CH_3	H	H
b	C_6H_5	H	H	H
c	C_6H_5	CH_3	H	H
d	C_6H_5	C_6H_5	H	H
e	C_6H_5	CH_3	CH_3	CH_3
f	C_6H_5	C_6H_5	CH_3	CH_3
g	C_6H_5	CH_3	C_2H_5	H
h	C_6H_5	C_6H_5	C_2H_5	H
i	C_6H_5	CH_3	C_6H_5	H

3	R^1	R^2	R^3	R^4	4	R^1	R^2	R^3	R^4
a	C_6H_5	CH_3	H	H	**a**	C_6H_5	CH_3	CH_3	CH_3
b	C_6H_5	H	CH_3	CH_3	**b**	C_6H_5	C_6H_5	CH_3	H
c	C_6H_5	CH_3	CH_3	CH_3	**c**	C_6H_5	C_6H_5	CH_3	CH_3
d	C_6H_5	C_6H_5	CH_3	CH_3	**d**	C_6H_5	CH_3	C_6H_5	H
e	C_6H_5	CH_3	C_6H_5	H	**e**	C_6H_5	C_6H_5	C_6H_5	H

Table 1. Bicyclic Lactones **2**, **3**, and **4** Prepared

Product	Method	Yield[a] (%)	*cis*/*trans* (*M*/*m* or *A*/*B*) ratio	m.p. (°C)[b] b.p. (°C)/Torr (solvent)	Molecular Formula[c]	Isomer	IR ($CHCl_3$)[d] ν (cm^{-1})
2a	A	70	>95/5	126–127/0.05	$C_{14}H_{20}O_6$ (284.3)	*cis*	1772, 1725
2b	A	70	65/35			*cis*	
				205–209/0.05	$C_{18}H_{20}O_6$ (332.3)	*trans*	1775, 1720
2c	A	96	92/8	70–71 (EtOH)	$C_{19}H_{22}O_6$ (346.4)	*cis*	1775, 1725
						trans	
2d	A	84	< 5/95	122–123 ($CHCl_3$/hexane)	$C_{24}H_{24}O_6$ (408.4)	*trans*	1780, 1720
2e	A	90	>95/5	89–90 ($CHCl_3$/hexane)	$C_{21}H_{26}O_6$ (374.4)	*M*	1770, 1710
2f	A	75	>95/5	118.5–119.5 ($CHCl_3$/hexane)	$C_{26}H_{28}O_6$ (436.5)	*M*	1780, 1720
2g	A	68	50/50	92–93 (EtOAc/hexane)		*cis-A*	1775, 1718
				117–118 (EtOAc/hexane)	$C_{21}H_{26}O_6$ (374.4)	*cis-B*	1775, 1720
2h	A	69	52/48	117–118 (EtOAc/hexane)		*M-A*	1778, 1718
				110–111 (EtOAc/hexane)	$C_{26}H_{28}O_6$ (436.5)	*M-B*	1780, 1720
2i	B	74	50/50	167–168 ($CHCl_3$/hexane)		*cis-A*	1775, 1722
				170–172 ($CHCl_3$/hexane)	$C_{24}H_{24}O_6$ (408.4)	*cis-B*	1778, 1722
3a	A	60	>95/5	130–131 (EtOAc/hexane)	$C_{21}H_{27}NO_5$ (373.4)	*cis*	1770, 1722, 1700
3b	A	80	50/50	154–155 ($CHCl_3$/hexane)		*cis*	1780, 1720, 1610
				156–157 ($CHCl_3$/hexane)	$C_{20}H_{25}NO_5$ (359.4)	*trans*	1775, 1720, 1605
3c	A	75	>95/5	120–121 ($CHCl_3$/hexane)	$C_{21}H_{27}NO_5$ (373.4)	*M*	1780, 1720, 1610
3d	A	80	>95/5	170–171 ($CHCl_3$/hexane)	$C_{26}H_{29}NO_5$ (435.5)	*M*	1782, 1720, 1603
3e	B	71	33/67			*cis-A*	
				232–233	$C_{25}H_{27}NO_5$ (421.5)	*cis-B*	1770, 1720, 1630
4a	A	77	67/33	126–127 ($CHCl_3$/hexane)		*M*	2230, 1780, 1720
				111–111.5 ($CHCl_3$/hexane)	$C_{19}H_{21}NO_4$ (327.4)	*m*	2230–1780, 1720
4b	A	75	17/83			*M-A*	
				165–166 (EtOAc/hexane)	$C_{23}H_{21}NO_4$ (375.4)	*M-B*	2230, 1785, 1720
4c	A	86	>95/5	174–175 ($CHCl_3$/hexane)	$C_{24}H_{23}NO_4$ (398.4)	*M*	2230, 1778, 1720
4d	B	61	36/64	177–178 ($CHCl_3$/hexane)		*cis-A*	2240, 1785, 1723
				137–139 ($CHCl_3$/hexane)	$C_{23}H_{21}NO_4$ (375.4)	*cis-B*	2240, 1780, 1720
4e	B	60	≈15/85			*M-A*	
				176–177 ($CHCl_3$/hexane)	$C_{28}H_{23}NO_4$ (437.5)	*M-B*	2240, 1790, 1720

[a] Yield of isolated product based on **1**.
[b] m.p.s were measured in sealed capillaries and are uncorrected.
[c] Satisfactory microanalyses obtained: C ±0.31, H ±0.29, N ±0.29; except **3d**, C −0.49.
[d] Recorded on a specord 75 IR Carl Zeiss Jena spectrophotometer.

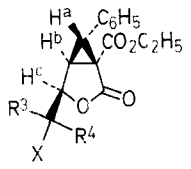
Table 2. ^{1}H-NMR Data for Bicyclic Lactones **2**, **3**, and **4**

Product	Isomer	^{1}H-NMR ($CDCl_3$/TMS)[a,b] δ, J(Hz)
2a	*cis*	[a]1.29 (t, 3H, J = 7.2); 1.33 (t, 3H, J = 7.2); 1.32 (d, 3H, J = 6.4); 1.59 (s, 3H); 1.77 (m, H^a); 2.62 (d, 1H, J = 15.3, HCHCOOEt); 2.74 (d, 1H, J = 15.3, HCHCOOEt); 2.61 (d, H^b, J = 5.4); 4.19 (q, 2H, J = 7.2); 4.29 (q, 2H, J = 7.2)
2b	*cis*	[a,c] 0.91 (t, 3H, J = 7.1); 1.29 (t, 3H, J = 7.0); 2.67 (dd, 1H, J = 16.5, 8.2, HCH–COOEt); 2.88 (dd, 1H, J = 16.5, 5.4, HCHCOOEt); 2.92 (d, H^a, J = 5.48); 3.52 (dd, H^b, J = 5.48, 5.40); 3.89–4.0 (m, 2H); 4.23 (q, 2H, J = 7.0); 5.09–5.17 (m, H^c); 7.30 (s, 5H)
	trans	[a,c] 0.91 (t, 3H, J = 7.1); 1.30 (t, 3H, J = 7.2); 2.83–2.97 (m, 3H, H^a + CH_2COOEt); 3.23 (d, H^b, J = 5.5); 3.89–4.0 (m, 2H); 4.23 (q, 2H, J = 7.0); 4.85 (t, H^c, J = 6.13); 7.26 (s, 5H)
2c	*cis*	[a] 0.90 (t, 3H, J = 7.1); 1.26 (t, 3H, J = 7.1); 1.69 (s, 3H); 2.72 (d, 1H, J = 15.9, HCHCOOEt); 2.87 (d, 1H, J = 15.9, HCHCOOEt); 2.97 (d, H^a, J = 5.6); 3.46 (d, H^b, J = 5.6); 3.95 (q, 2H, J = 7.1); 4.19 (q, 2H, J = 7.1); 7.30 (s, 5H)
	trans	[a,c] 1.56 (s, 3H); 2.44 (s, 2H); 3.42 (d, H^b, J = 5.6); 7.27 (s, 5H)
2d	*trans*	[a] 0.87 (t, 3H, J = 7.1); 1.12 (t, 3H, J = 7.2); 3.06 (d, 1H, J = 15.5, HCHCOOEt); 3.20 (d, 1H, J = 15.5, HCHCOOEt); 3.20 (d, H^a, J = 5.6); 3.98 (d, H^b, J = 5.6); 3.90 (q, 2H, J = 7.1); 4.06 (q, 2H, J = 7.2); 7.28–7.64 (m, 10H)
2e	*M*	[b] 0.86 (t, 3H, J = 7.2); 1.24 (t, 3H, J = 7.2); 1.32 (s, 6H); 1.50 (s, 3H); 3.23 (s, 2H, H^a + H^b); 3.94 (q, 2H, J = 7.2); 4.16 (q, 2H, J = 7.2); 7.32 (s, 5H)
2f	*M*	[a] 0.78 (t, 3H, J = 7.1); 1.14 (s, 3H); 1.24 (t, 3H, J = 7.2); 1.46 (s, 3H); 3.54 (d, H^a, J = 6.0); 3.80 (d, H^b, J = 6.0); 3.82 (q, 2H, J = 7.1); 4.17 (q, 2H, J = 7.2); 7.34–7.49 (m, 10H)
2g	*cis-A*	[a] 0.91 (t, 3H, J = 7.1); 0.95 (t, 3H, J = 7.2); 1.11 (t, 3H, J = 7.1); 1.62 (s, 3H); 1.75–1.95 (m, 2H); 2.62 (dd, 1H, J = 6.4, 10.4); 3.08 (d, H^a, J = 5.7); 3.13 (d, H^b, J = 5.7); 3.95 (dq, 2H, J = 7.1, 2.3); 4.10 (q, 1H, J = 7.2); 4.18 (q, 1H, J = 7.2); 7.22–7.35 (m, 5H)
	cis-B	[a] 0.91 (t, 3H, J = 7.2); 0.95 (t, 3H, J = 7.3); 1.28 (t, 3H, J = 7.1); 1.63 (s, 3H); 1.78–1.93 (m, 2H); 2.62 (dd, 1H, J = 3.6, 11.5); 3.09 (d, H^a, J = 5.7); 3.16 (d, H^b, J = 5.7); 3.96 (dq, 2H, J = 7.2, 2.4); 4.16 (q, 1H, J = 7.1); 4.23 (q, 1H, J = 7.1); 7.20–7.35 (m, 5H)
2h	*M-A*	[a] 0.83 (t, 3H, J = 7.2); 0.85 (t, 3H, J = 7.2); 1.13 (t, 3H, J = 7.1); 1.53–1.66 (m, 2H); 2.89 (dd, 1H, J = 4.4, 11.0); 3.35 (d, H^a, J = 5.8); 3.72 (d, H^b, J = 5.8); 3.85 (q, 2H, J = 7.2); 4.13 (q, 1H, J = 7.1); 4.19 (q, 1H, J = 7.1); 7.32–7.57 (m, 10H)
	M-B	[a] 0.85 (t, 3H, J = 7.1); 0.87 (t, 3H, J = 7.3); 1.08 (t, 3H, J = 7.1); 1.60–1.72 (m, 1H); 1.83–1.95 (m, 1H); 2.96 (dd, 1H, J = 3.06, 11.9); 3.34 (d, H^a, J = 5.87); 3.66 (d, H^b, J = 5.87); 3.84 (q, 2H, J = 7.1); 3.95 (q, 1H, J = 7.1); 4.04 (q, 1H, J = 7.1); 7.34–7.54 (m, 10H)
2i	*cis-A*	[b] 0.83 (t, 3H, J = 7.2); 1.47 (s, 3H); 3.17 (d, H^a, J = 6.0); 3.43 (d, H^b, J = 6.0); 3.62 (s, 3H); 3.90 (q, 2H, J = 7.2); 4.07 (s, 1H); 7.30 (s, 10H)

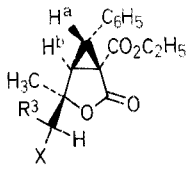
Table 2. (continued)

Product	Isomer	^{1}H-NMR ($CDCl_3$/TMS)[a,b] δ, J(Hz)
	cis-B	[b] 0.80 (t, 3H, J = 7.1); 1.72 (s, 3H); 2.78 (d, H^a, J = 6.0); 2.98 (d, H^b, J = 6.0); 3.70 (s, 3H); 3.87 (q, 2H, J = 7.1); 4.06 (s, 1H); 7.08–7.48 (m, 10H)
3a	*cis*	[b] 0.84 (t, 3H, J = 7.1); 0.90 (t, 3H, J = 7.1); 1.26 (t, 3H, J = 7.2); 1.60 (s, 3H); 2.88 (d, H^a, J = 6.0); 3.24 (d, 1H, J = 14.0, HCHCONEt$_2$); 3.40 (d, H^b, J = 6.0); 3.56 (d, 1H, J = 14.0, HCHCONEt$_2$); 3.95 (dq, 4H, J = 7.1, 2.2); 4.25 (q, 2H, J = 7.2); 7.32 (s, 5H)
3b	*cis*	[b] 0.80 (t, 3H, J = 7.1); 1.36 (s, 3H); 1.42 (s, 3H); 3.02 (s, 6H); 3.12 (d, H^a, J = 4.4); 3.56 (dd, H^b, J = 4.0, 4.4); 3.88 (q, 2H, J = 7.1); 4.92 (d, H^c, J = 4.0); 7.28 (s, 5H)
	trans	[b] 0.84 (t, 3H, J = 7.0); 1.32 (s, 3H); 1.40 (s, 3H); 2.82 (d, H^a, J = 5.9); 3.08 (s, 6H); 3.20 (d, H^b, J = 5.9); 3.96 (q, 2H, J = 7.0); 4.80 (s, H^c); 7.28 (s, 5H)
3c	*M*	[a] 0.89 (t, 3H, J = 7.1); 1.49 (s, 3H); 1.51 (s, 3H); 1.57 (s, 3H); 3.07 (s, 6H); 3.15 (d, H^a, J = 5.9); 3.29 (d, H^b, J = 5.9); 3.94 (q, 2H, J = 7.1); 7.29 (s, 5H)
3d	*M*	[b] 0.70 (t, 3H, J = 7.0); 1.36 (s, 3H); 1.42 (s, 3H); 2.78 (s, 6H); 3.40 (d, H^a, J = 5.8); 3.74 (d, H^b, J = 5.8); 3.77 (q, 2H, J = 7.0); 7.20–7.68 (m, 10H)
3e	*cis-A*	[a] 0.82 (t, 3H, J = 7.0); 1.76 (s, 3H); 2.87 (s, 3H); 3.01 (s, 3H); 3.56 (d, H^a, J = 5.9); 3.83 (d, H^b, J = 5.9); 3.88 (q, 2H); 4.32 (s, 1H); 7.20–7.32 (m, 10H)
	cis-B	[a] 0.84 (t, 3H, J = 7.2); 1.51 (s, 3H); 2.72 (s, 3H); 2.96 (s, 3H); 3.14 (d, H^a, J = 5.7); 3.67 (d, H^b, J = 5.7); 3.88 (q, 2H, J = 7.2); 4.14 (s, 1H); 7.20–7.32 (m, 10H)
4a	*M*	[b] 0.88 (t, 3H, J = 7.4); 1.48 (s, 6H); 1.52 (s, 3H); 3.08 (d, H^a, J = 6.0); 3.80 (d, H^b, J = 6.0); 3.96 (q, 2H, J = 7.4); 7.21 (s, 5H)
	m	[b] 0.88 (t, 3H, J = 7.2); 1.42 (s, 3H); 1.46 (s, 3H); 1.56 (s, 3H); 2.93 (d, H^a, J = 5.8); 3.28 (d, H^b, J = 5.8); 3.96 (q, 2H, J = 7.2); 7.30 (s, 5H)
4b	*M-A*	[a] 0.82 (t, 3H, J = 7.1); 1.15 (d, 3H, J = 7.0); 4.02 (q, 2H, J = 7.1)
	M-B	[a] 0.86 (t, 3H, J = 7.2); 1.36 (d, 3H, J = 7.3); 3.32 (q, 1H, J = 7.3); 3.55 (d, H^a, J = 5.8); 3.63 (d, H^b, J = 5.8); 3.89 (q, 2H, J = 7.2); 7.32 (s, 5H); 7.30–7.68 (m, 5H)
4c	*M*	[a] 0.84 (t, 3H, J = 7.1); 1.32 (s, 3H); 1.54 (s, 3H); 3.68 (d, H^a, J = 5.9); 3.85 (q, 2H, J = 7.1); 4.09 (d, H^b, J = 5.9); 7.36 (s, 5H); 7.28–7.65 (m, 5H)
4d	*cis-A*	[a] 0.80 (t, 3H, J = 7.3); 1.66 (s, 3H); 2.80 (d, H^a, J = 5.8); 3.08 (d, H^b, J = 5.8); 3.86 (q, 2H, J = 7.3); 4.18 (s, 1H); 7.38 (s, 5H); 7.04–7.44 (m, 5H)
	cis-B	[a] 0.79 (t, 3H, J = 7.2); 1.70 (s, 3H); 2.86 (d, H^a, J = 5.9); 3.26 (d, H^b, J = 5.9); 3.68 (q, 2H, J = 7.2); 4.22 (s, 1H); 7.24 (s, 5H); 7.38 (s, 5H)
4e	*M-A*	[a] 0.83 (t, 3H, J = 7.2); 4.46 (s, 1H)
	M-B	[a] 0.81 (t, 3H, J = 7.1); 3.39 (d, H^a, J = 5.8); 3.50 (d, H^b, J = 5.8); 3.83 (q, 2H, J = 7.1); 4.48 (s, 1H); 7.20–7.43 (m, 15H)

[a] Recorded at 250 MHz on a Brucker WM spectrometer.
[b] Recorded at 100 MHz on a JEOL-JNM-PS-100 spectrometer.
[c] Values from the spectrum of the mixture of the two isomers.



cis-**2b**: X = $CO_2C_2H_5$, R^3 = R^4 = H
cis-**3b**: X = $CON(CH_3)_2$, R^3 = R^4 = CH_3



2g,i; 3e; 4d
R^3 = C_2H_5 or C_6H_5;
X = $CO_2C_2H_5$ or $CON(CH_3)_2$ or CN

For compounds **2g, 2i, 3e,** and **4d** it can be shown by NOE experiments that the configuration at C-4, C-5 is always *cis*. Two isomers are obtained, generally non-stereoselectively, due to the chiral center originating from the Reformatsky reagent. In all cases where diastereoisomers of this kind are obtained, the isomer with the higher R_f value is designated as *A*, the one with the lower R_f value as *B* (TLC on silica gel, eluent ether/hexane: 50/50 for **2g, h**; 60/40 for **2i, 4b, d, e**; 80/20 for **3e**).

For the compounds obtained with zinc-reagents prepared from acetic acid derivatives, the configuration at C-4 can be resolved on the basis of the diastereotopicity of the methylene protons adjacent to C-4. The magnetic behavior of these methylene protons depends on the relative positions of the CH_2 group and the cyclopropane ring. For similar compounds with unambiguously established configurations, marked magnetic non-equivalence is observed for the CH_2 protons in *endo*-position to the cyclopropane ring.[1,7] We thus assign the *cis*-configuration to **2a, b, c** and the *trans* configuration to **2d**. This assignment was confirmed for **2b** (see above).

^{1}H-NMR data were insufficient to assign the configurations of **2e, f, h, 3c, d,** and **4a, b, c, e**. For this reason we designate the respective two isomers *M* (major) and *m* (minor).

The isomer ratios were estimated from the ^{1}H-NMR spectra using the integration curves refering to the signals underlined in the Table. In most cases, formation of the chiral centre at C-4 proceeds with good stereoselectivity.

All reactions are carried out under nitrogen. The intermediate Reformatsky reagents are prepared as reported in the literature: esters and amides in THF/Et_2O (1:4),[5] nitriles in THF.[4] Compounds **1** are prepared according to Ref.[8].

4-Substituted 1-Ethoxycarbonyl-2-oxo-3-oxabicyclo[3.1.0]hexanes (2, 3, 4); General Procedures:

Method A, Two-Step Procedure: To a stirred solution of the Reformatsky reagent (10 mmol) in the respective solvent (7.5 mL) cooled to −10 °C is added dropwise a solution of the 2-acylcyclopropane-1,1-dicarboxylic ester **1** (7 mmol) in THF (5 mL). The cooling bath is then removed and the mixture stirred at room temperature for 5 h. The mixture is then hydrolyzed with ice-cold H_2O (25 mL) containing conc. HCl/H_2O (1 mL) and extracted with Et_2O (3 × 20 mL). The organic layer is washed with H_2O (10 mL), dried (Na_2SO_4), and evaporated under reduced pressure.

Method B, One-Step Procedure: A mixture of zinc turnings (0.65 g, 1 mmol), the α-bromophenylacetic acid derivative (10 mmol), the 2-acylcyclopropane-1,1-dicarboxylic ester **1** (8 mmol), and a few crystals of mercury(II) chloride (15 mg) in THF (10 mL) is stirred at room temperature for 5 h. Work-up is as in Method A.

The crude products obtained by Methods A or B are purified by vacuum distillation or recrystallization. The isomers are separated by fractional recrystallization (**2i, 3b, e, 4b, d, e**) or recrystallization combined with preparative TLC (**2g, h, 4a**; silica gel, eluent Et_2O/hexane, 50:50 for **2g** and **2h**; 60:40 for **4a**).

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