Synthesis of 2,5-Diaryl-3-halofurans via Regioselective Ring Cleavage of Aryl 3-Aryl-2,2-dihalocyclopropyl Ketones

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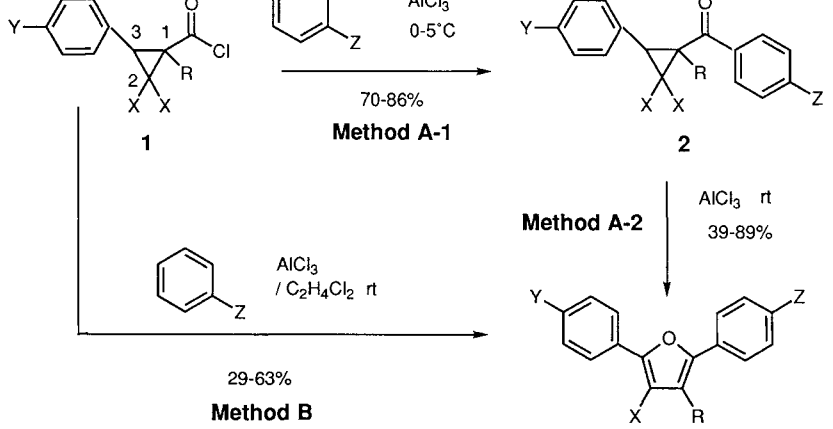
Several aryl 3-aryl-2,2-dihalocyclopropyl ketones were converted to 2,5-diaryl-3-halofurans in the presence of aluminum chloride via regioselective *gem*-dihalocyclopropane ring-cleavage. Friedel–Crafts acylation of substituted benzenes with 3-aryl-2,2-dihalocyclopropanecarbonyl chlorides followed by this furan formation also proceeded in a one-pot manner. For functionalization, bromine on a furan ring was easily replaced by methyl and carboxyl groups; lithiation using butyllithium followed by the treatment with iodomethane and carbon dioxide, respectively.

Reactions utilizing cyclopropane ring expansions have been developed in various types of characteristic synthetic methods.[1] Among the functionalized cyclopropanes, *gem*-dihalocyclopropanes should be noted for the following aspects: (1) ease of preparation by dihalocarbene addition to olefins;[2] (2) feasibility of the reductive dehalogenation giving halocyclopropanes or cyclopropanes;[3] and (3) utility for worthwhile transformations,[4] stereoselective C–C bond formation,[5] annulations via regioselective ring openings,[6] and radical cyclizations.[7] Following our interest in new reactions utilizing *gem*-dihalocyclopropanes,[6,7] we report here a novel synthesis of 2,5-diaryl-3-halofurans **3** from 3-aryl-2,2-dihalocyclopropyl ketones **2** and from 2-aryl-3,3-dihalocyclopropanecarbonyl chlorides **1** with substituted benzenes, wherein highly regioselective ring cleavage of these *gem*-dihalocyclopropane rings occurred. In addition, a further functionalization is described to demonstrate the present method: bromine of furan **3e** was easily replaced by methyl and carboxyl groups. Accordingly, tetrasubstituted 2,5-diarylfurans **3a–3f** were synthesized from readily available substrates and reagents.

Ketones **2** were prepared from 3-aryl-2,2-dihalocyclopropanecarbonyl chlorides **1** and substituted benzenes by Friedel–Crafts acylation using $AlCl_3$ (1.1–2.0 molar amounts) at 0–5 °C. Such mild conditions substantially prevented the cyclopropyl ketones **2** from undergoing further cyclopropane ring cleavage. However, in the case of the reaction of **1a** with benzene (Z = H), the Friedel–Crafts acylation was so sluggish that the desired ketone **2d** (X = Cl, Y = Z = H, R = Me) could not be obtained. Phenylmagnesium bromide (a molar amount) was found to couple with **1a** to give the ketone **2d** in 62% yield. Treatment of 3-aryl-2,2-dihalocyclopropyl aryl ketones **2** with $AlCl_3$ (1.1–3.2 molar amounts) at r.t. induced the furan cyclization to give the corresponding 2,5-diaryl-3-halofurans **3** (Method A). It is worth noting that the present synthesis of furans **3** was also



Scheme 1

Table 1. Synthesis of Ketones **2** from Acyl Chlorides **1** by Friedel–Crafts Acylation (Method A-1)[a]

Substrate	(R	X	Y)	Z	Molar Amount[b]	Solvent	Product	Yield (%)
1a	(Me	Cl	H)	Me	1.1	Carbon disulfide	**2a**	75
1a	(Me	Cl	H)	OMe	2.0	Chlorobenzene	**2b**	86
1b	(H	Cl	H)	Me	1.1	Carbon disulfide	**2c**	70

[a] The reactions were carried out at 0–5 °C for 10 h.
[b] $AlCl_3$ vs **1**.

Table 2. Synthesis of 2,5-Diaryl-3-halofurans **3** from Ketones **2** (Method A-2)[a]

Substrate	(R	X	Y	Z)	Molar Amount[b]	Solvent	Product	Yield (%)
2a	(Me	Cl	H	Me)	2.2	1,2-Dichloroethane	**3a**	61
2b	(Me	Cl	H	OMe)	3.2	1,2-Dichloroethane	**3b**	89
2c	(H	Cl	H	Me)	2.2	Chlorobenzene	**3c**	44
2d	(Me	Cl	H	H)	2.2	1,2-Dichloroethane	**3f**	62

[a] The reactions were carried out at r.t.
[b] $AlCl_3$ vs **2**.

Table 3. Direct Synthesis of 2,5-Diaryl-3-halofurans **3** from Acyl Chlorides **1** (Method B)[a]

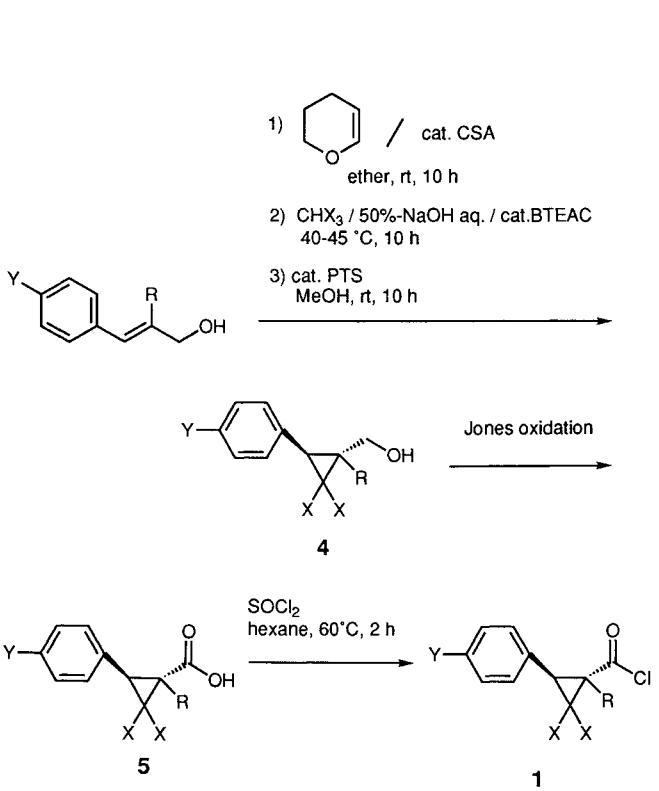
Substrate	(R	X	Y)	Z	Molar Amount[b]	Product	Yield (%)
1a	(Me	Cl	H)	OMe	2.0	**3a**	57
1a	(Me	Cl	H)	OMe	3.3	**3b**	60
1c	(Me	Cl	Me)	OMe	3.3	**3d**	29
1d	(Me	Br	Me)	OMe	3.3	**3e**	63

[a] The reactions were carried out at r.t. in 1,2-dichloroethane for 20 h.
[b] $AlCl_3$ vs **2**.

performed directly from 3-phenyl-2,2-dihalocyclopropane carbonyl chlorides **1** and substituted benzenes, catalyzed by $AlCl_3$ (2.2 molar amounts) in a one-pot manner (without the isolation of intermediary ketones **2**) at ambient temperature (Method B). Tables 1, 2 and 3 show these results.

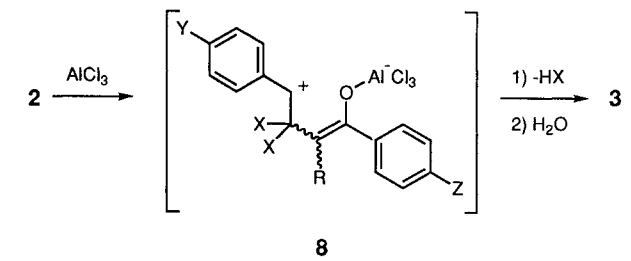
The reaction of aryl 2-arylcyclopropyl ketones catalyzed by $SnCl_4$, $BF_3 \cdot OEt_2$, or CF_3CO_2H was reported to give mainly the corresponding tetralones via regioselective cyclopropane ring cleavage, wherein the related furan-formation was limited to a sole specific example.[8] Our previous report also described that aryl(*gem*-dihalocyclopropyl)methanols were transformed into α- and β-halonaphthalenes by an acid-catalyzed reaction.[6] In contrast to these facts, we found that the present system gave no substantial formation of plausible naphthol-type products[6,8] under the aforementioned reaction conditions.

The starting acyl chlorides **1** were easily prepared by the following sequence (Scheme 2): (1) dihalocarbene addition to THP ethers of 3-aryl-2-propen-1-ols; (2) deprotection of these THP ethers giving 3-aryl-2,2-dihalocyclopropyl methanols **4**; and (3) Jones oxidation of **4** giving 3-aryl-2,2-dihalocyclopropanecarboxylic acids **5**, followed by conversion to acyl chlorides **1**. The structural proof of furans **3a–3f** was confirmed by ^1H NMR, IR, MS (in the case of **3a**), and elemental analyses. The physical and spectral data of the acyl chlorides **1**, ketones **2**, and furans **3** are listed in Table 4. Table 5 shows those of intermediary alcohols **4** and carboxylic acids **5**.



Scheme 2

The key step of the reactions, i.e., the transformation of ketones **2** to halofurans **3** would proceed via enolate intermediates **8** (Scheme 3), wherein regioselective bond fission between C-1 and C-3 of **2** occurred due to the feasible formation of a benzyl cation compared with the dichloromethylium ion (via bond fission between C-1 and C-2). Subsequent cyclization of the enolates **8** gives ha-



Scheme 3

Table 4. Physical and Spectral Data of Acyl Chlorides **1a–1d**, Ketones **2a–2d**, and Furans **3a–3f**

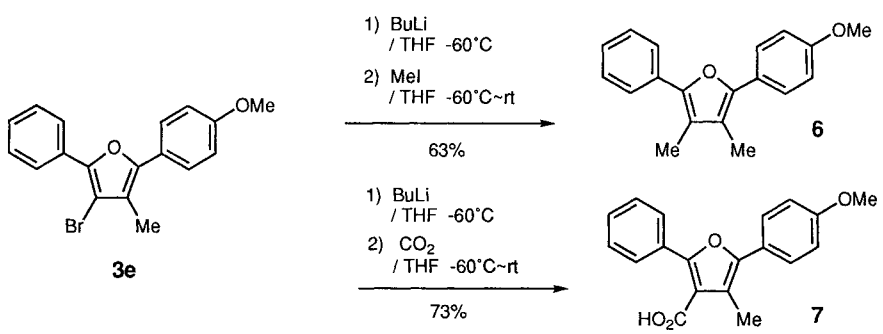
Compound[a]	R	X	Y	Z	mp or bp[b] (°C)	IR (KBr, cm^{-1})	^1H NMR ($CDCl_3$, TMS)
1a	Me	Cl	H	–	175–220/0.4 Torr	–	1.55 (3H, s), 3.65 (1H, s), 7.15–7.70 (5H, m)
1b	H	Cl	H	–	130/0.35 Torr	–	3.40 (1H, d, J = 9.0 Hz), 3.60 (1H, d, J = 9.0 Hz), 7.05–7.70 (5H, m)
1c	Me	Cl	Me	–	130/0.22 Torr	–	1.55 (3H, s), 2.35 (3H, s), 3.60 (1H, s), 7.10–7.25 (4H, m)
1d	Me	Br	H	–	210–215/0.3 Torr	–	1.55 (3H, s), 3.65 (1H, s), 7.20–7.80 (5H, m)
2a	Me	Cl	H	Me	84–85	1680, 1290, 1180	1.40 (3H, s), 2.45 (3H, s), 3.55 (1H, s), 7.15–7.45 (5H, m), 7.35 (2H, J = 9.0 Hz), 7.95 (2H, d, J = 9.0 Hz)
2b	Me	Cl	H	OMe	63–64	1680, 1270, 1175	1.45 (3H, s), 3.50 (1H, s), 3.90 (3H, s), 7.05 (2H, d, J = 8.0 Hz), 7.20–7.50 (5H, m), 8.05 (2H, d, J = 8.0 Hz)
2c	H	Cl	H	Me	125–128	1680, 1610, 1230	2.45 (3H, s), 3.65 (1H, d, J = 9.0 Hz), 3.75 (1H, d, J = 9.0 Hz), 7.20–7.50 (7H, m), 8.05 (2H, d, J = 9.0 Hz)
2d[c]	Me	Cl	H	H	87–89	1680, 1290, 1170	1.40 (3H, s), 3.55 (1H, s), 7.15–7.80 (8H, m), 7.90–8.10 (2H, m)
3a[d]	Me	Cl	H	Me	71.0–72.0	1600, 1490, 950	2.25 (3H, s), 2.40 (3H, s), 7.00–8.15 (9H, m)
3b	Me	Cl	H	OMe	93.5–94.0	1620, 1520, 1260	2.25 (3H, s), 3.85 (3H, s), 6.90–8.10 (9H, m)
3c	H	Cl	H	Me	83.5–84.0	1610, 1520, 820	2.30 (3H, s), 6.60 (1H, s), 7.05–7.95 (9H, m)
3d	Me	Cl	Me	OMe	100–100.5	1520, 1260, 820	2.25 (3H, s), 2.40 (3H, s), 3.85 (3H, s), 6.90–8.00 (9H, m)
3e	Me	Br	H	OMe	105–106	1505, 1260, 840	2.30 (3H, s), 3.90 (3H, s), 6.90–8.20 (9H, m)
3f	Me	Cl	H	H	65.5–68.0	1600, 1500, 940	2.25 (3H, s), 7.20–8.10 (10H, m)

[a] All elemental analyses were C, H ± 0.3%.
[b] Oven temp. of bulb-to-bulb distillation.
[c] Prepared by Grignard reaction (shown in Text).
[d] MS (70 eV): m/z (rel intensity) = 282 (M^+, 100), 91 (C_7H_7, 23), 77 (C_6H_6, 32).

Table 5. Physical and Spectral Data of Alcohols **4a–d** and Carboxylic Acids **5a–d**

Compound[a]	R	X	Y	Yield (%)	mp (°C)	IR (KBr, cm^{-1})	^1H NMR ($CDCl_3$, TMS)
4a	Me	Cl	H	63	101–101	3320, 1450, 1050	1.30 (3H, s), 1.75 (1H, br s), 2.70 (1H, s), 3.85 (1H, d, J_{gem} = 13.5 Hz), 4.05 (1H, d, J_{gem} = 13.5 Hz), 7.10–7.55 (5H, m)
4b	H	Cl	H	55	oil	3400, 1510, 1060	1.70–2.00 (1H, br), 2.30 (1H, dt, J = 9.0 Hz, J = 9.0 Hz), 2.65 (1H, d, J = 9.0 Hz), 3.65–4.30 (2H, m), 7.10–7.55 (5H, m)
4c	Me	Cl	Me	54	113–116	3250, 1520, 1050	1.30 (3H, s), 1.65–1.95 (1H, br), 2.35 (3H, s), 2.65 (1H, s), 3.65–4.20 (2H, m), 7.00–7.40 (4H, m)
4d	Me	Br	H	52	116–118	3310, 1460, 1160	1.35 (3H, s), 1.70–2.10 (1H, br), 2.75 (1H, s), 3.90 (1H, d, J_{gem} = 11.0 Hz), 4.05 (1H, d, J_{gem} = 11.0 Hz), 7.10–7.55 (5H, m)
5a	Me	Cl	H	87	125–126	3000–2300, 1710, 1310	1.45 (3H, s), 3.65 (1H, s), 7.10–7.60 (5H, m)
5b	H	Cl	H	80	100–101	3100–2300, 1720, 1460	2.90 (1H, d, J = 9.0 Hz), 3.50 (1H, d, J = 9.0 Hz), 7.10–7.60 (5H, m)
5c	Me	Cl	Me	92	145–147	3100–2400, 1720, 1305	1.40 (3H, s), 2.35 (3H, s), 3.60 (1H, s), 2.65 (1H, s), 7.15–7.25 (4H, m)
5d	Me	Br	H	94	169–170	3050–2300, 1710, 1310	1.45 (3H, s), 3.65 (1H, s), 7.05–7.60 (5H, m)

[a] All elemental analyses were C, H ± 0.3%.



Scheme 4

lofurans **3** with elimination of the hydrogen halide. This result of the regioselective cleavage of aryl-substituted cyclopropanes coincides with those of the tetralone[8] and the naphthalene annulations.[6]

Finally, to show the utility of a new class of halofurans **3**, further functionalizations of bromofuran **3e** were performed (Scheme 4). Treatment of **3e** with BuLi at $-60\,^{\circ}C$ (bromine–lithium exchange) followed by the addition of excess MeI gave 3,4-dimethyl-2-(4-methoxyphenyl)-5-phenylfuran **(6)** in 63% yield. In a similar type of reaction using excess amount of CO_2 in the place of MeI, 4-methyl-5-(4-methoxyphenyl)-2-phenylfuran-3-carboxylic acid **(7)** was obtained in 73% yield.

Boiling points are uncorrected. Melting points were determined using a hot-stage apparatus and are uncorrected. ^1H NMR spectra were recorded on a JEOL EX-90 (90 MHz) spectrometer using TMS as an internal standard in $CDCl_3$. IR spectra were recorded on a Hitachi 270-30 spectrophotometer. Mass spectra were obtained using a Hitachi GC/MS M-80 instrument. Reagents were of commercial grade and were used without further purification. The solvents were purified by standard methods. Silica gel column chromatography was performed on Merck Art. 7734 and/or 9385.

Satisfactory C, H analyses were recorded for products **6** and **7** ($\pm 0.3\%$).

2,2-Dichloro-1-methyl-*t*-3-phenyl-*r*-1-cyclopropyl(*p*-tolyl)methanone (2a); Typical Procedure for Method A-1:
A mixture of 2,2-dichloro-1-methyl-*t*-3-phenyl-*r*-1-cyclopropanecarbonyl chloride (**1a**; 500 mg, 1.90 mmol), toluene (1.57 g, 20 mmol), and $AlCl_3$ (415 mg, 3.11 mmol) in CS_2 (7.2 mL) was stirred at 0–5 °C for 10 h under a nitrogen atmosphere. 1 M HCl was added to the reaction mixture with stirring for several minutes. The mixture was extracted with Et_2O, the organic phase was washed with water and brine, dried (Na_2SO_4), and concentrated. The crude oil obtained was purified by silica gel column chromatography (hexane/Et_2O 30:1) to give ketone **2a**; yield: 436 mg (75%).

In a similar procedure, ketone **2b** (1.06 g, 86%) was obtained using **1a** (1.00 g, 3.1 mmol), anisole (0.60 g, 5.5 mmol), $AlCl_3$ (0.83 g, 6.2 mmol), and chlorobenzene (15 mL) in the place of CS_2. Ketone **2c** was prepared using **1b** in an almost similar procedure to that for the preparation of **2a**.

2,2-Dichloro-1-methyl-*t*-3-phenyl-*r*-1-cyclopropyl(phenyl)methanone (2d); (by Grignard Reaction):
To a stirred solution of phenylmagnesium bromide (1.0 M THF solution; 0.76 mL) was added 2,2-dichloro-1-methyl-*t*-3-phenyl-*r*-1-cyclopropanecarbonyl chloride (**1a**; 200 mg, 0.76 mmol) in THF (0.76 mL) at 0–5 °C under a nitrogen atmosphere. The mixture was stirred for 1 h at r.t. and then poured onto ice-sat. aq NH_4Cl solution. The mixture was extracted with Et_2O, the organic phase was washed with water and brine, dried (Na_2SO_4), and concentrated. The crude oil obtained was purified by silica gel column chromatography (hexane/Et_2O 35:1) to give ketone **2d**; yield: 144 mg (62%).

3-Chloro-4-methyl-2-phenyl-5-(*p*-tolyl)furan (3a); Typical Procedure for Method A-2:
To a stirred solution of ketone **2a** (158 mg, 0.50 mmol) in 1,2-dichloroethane (2.5 mL) was added $AlCl_3$ (145 mg, 1.09 mmol) at r.t. under a nitrogen atmosphere and the mixture was stirred for 20 h at r.t. After a similar workup as described for **2a** (Method A-1), the crude oil obtained was purified by silica gel column chromatography (hexane/Et_2O 50:1) to give furan **3a**; yield: 85 mg (61%).

In a similar procedure, furan **3b** (79 mg, 89%) was obtained using **2b** (100 mg, 0.31 mmol) and $AlCl_3$ (133 mg, 1.0 mmol). Furan **3c** was prepared from **2c** in a similar procedure using chlorobenzene as solvent in the place of 1,2-dichloroethane.

3-Chloro-4-methyl-2-phenyl-5-(*p*-tolyl)furan (3a); Typical Procedure for Method B:
To a stirred solution of acyl chloride **1a** (150 mg, 0.57 mmol) in 1,2-dichloroethane (2.2 mL) and toluene (0.47 mg, 5.1 mmol) was added $AlCl_3$ (152 mg, 1.14 mmol) at r.t. under a nitrogen atmosphere and the mixture was stirred for 20 h at r.t. After a similar work-up to that described for **2a** (Method A-1), the crude oil obtained was purified by silica gel column chromatography (hexane) to give furan **3a**; yield: 86 mg (57%).

In a similar manner, furans **3b**, **3d**, and **3e** were prepared from anisole and acyl chlorides **1a**, **1c**, and **1d**, respectively, wherein $AlCl_3$ (3.3 molar amounts vs. **1**) and anisole (1.2 molar amounts vs. **1**) were used in the place of toluene.

2,2-Dichloro-1-methyl-*t*-3-phenyl-*r*-1-cyclopropylmethanol (4a); Typical Procedure:
A mixture of (*E*)-2-methyl-3-phenylprop-2-en-1-ol (5.40 g, 40 mmol), 3,4-dihydro-2*H*-pyran (5.00 g, 59 mmol), and a small amount of camphorsulfonic acid in Et_2O (40 mL) was allowed to stand at r.t. for 10 h. After sat. aq $NaHCO_3$ solution had been added, the mixture was extracted with Et_2O (100 mL × 2). The organic phase was washed with water and brine, dried (Na_2SO_4) and concentrated to give 8.31 g of the crude tetrahydropyranyl (THP) ether. To a vigorously stirred mixture of the THP ether, benzyltriethylammonium chloride (456 mg, 2.0 mmol) and chloroform (47.8 g) was added 50% aq NaOH solution (32.0 g) at 35–40 °C and the mixture was stirred for 16 h at the same temperature. Water (200 mL) was added and then the mixture was extracted with CH_2Cl_2 (100 mL × 2). The organic phase was washed with water, brine, and dried (Na_2SO_4). After evaporation of the solvent, MeOH (50 mL) and a small amount of *p*-toluenesulfonic acid were added to the mixture which was then allowed to stand overnight. Sat. aq $NaHCO_3$ solution (10 mL) was added, followed by evaporation of MeOH to give the residue, which was extracted with Et_2O. The organic phase was washed with water and brine, dried (Na_2SO_4), and concentrated to give 5.75 g of the crude crystals; yield: 5.17 g (63%).

2,2-Dichloro-1-methyl-*t*-3-phenyl-*r*-1-cyclopropanecarboxylic Acid (5a); Typical Procedure:
To a stirred solution of alcohol **4a** (1.68 g, 7.3 mmol) in acetone (15 mL) was added the Jones reagent (5 mL) at 0–5 °C and the mixture was stirred at r.t. for 24 h. Then, propan-2-ol (5 mL) was added to the mixture at 0–5 °C followed by stirring for 30 min. After acetone had been evaporated from the mixture, water was added. The mixture was extracted with EtOAc (20 mL × 2) and the organic phase was washed with water and brine, dried (Na_2SO_4) and concentrated to give carboxylic acid **5a** as colorless crystals; yield: 1.59 g (87%).

2,2-Dichloro-1-methyl-*t*-3-phenyl-*r*-1-cyclopropanecarbonyl Chloride (1a); Typical Procedure:
A mixture of carboxylic acid **5a** (1.34 g, 5.5 mmol), thionyl chloride (0.79 g, 6.6 mmol), and a drop of DMF in hexane (11 mL) was heated under reflux for 16 h. The mixture was concentrated under reduced pressure and distilled (bulb-to-bulb distillation) to give acid chloride **1a** as a colorless oil; yield: 1.10 g (76%).

In a similar procedure, **1b** (65%), **1c** (78%), and **1d** (70%) were obtained.

3,4-Dimethyl-2-(4-methoxyphenyl)-5-phenylfuran (6):
To a stirred solution of bromofuran **3e** (50 mg, 0.15 mmol) in dry THF (0.3 mL) was added BuLi (1.5 M hexane solution; 0.19 mL, 0.29 mmol) at $-60\,^{\circ}C$ and the mixture was stirred for 1 h. After methyl iodide (213 mg, 1.5 mmol) had been added at $-60\,^{\circ}C$, the mixture was allowed to warm at r.t. and stirred for 16 h. Water was added and the mixture was extracted with EtOAc (10 mL × 2). The organic phase was washed with water and brine, dried (Na_2SO_4), and concentrated. The crude oil was purified by silica gel column chromatography (hexane/EtOAc 40:1) to give 41 mg of furan (**3e:6** = 1:2 mixture, conversion yield 63%). Colorless crystals (recryst. from CH_2Cl_2/hexane 1:1); mp 54.0–55.5 °C.
IR (KBr): $\nu = 1510, 1245\ cm^{-1}$.

^{1}H NMR ($CDCl_3$, TMS): δ = 2.20 (3 H, s), 2.25 (3 H, s), 3.85 (3 H, s), 6.85–7.90 (9 H, m).

4-Methyl-5-(4-methoxyphenyl)-2-phenylfuran-3-carboxylic Acid (7): Similar to the procedure for preparing **6** using excess amounts of carbon dioxide in the place of methyl iodide, carboxylic acid **7** was obtained after silica gel column chromatography (hexane/EtOAc 1:1); yield: 73%, colorless crystals (recryst. from CH_2Cl_2); mp 185.0–187.0°C.

IR (KBr): ν = 3600–2200, 1690, 1260 cm^{-1}.

^{1}H NMR ($CDCl_3$, TMS): δ = 2.45 (3 H, s), 3.85 (3 H, s), 6.90–8.00 (9 H, m).

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