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Synthesis of 2-Arylsuccinates by Oxidative 1,2-Aryl Migration of 3-Aroylpropionic Acids or 5-Arylfuran-2(3*H*)-ones with Thallium(III) Nitrate

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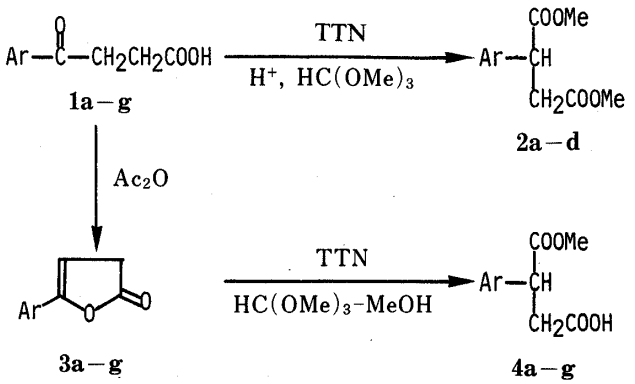
Treatment of 3-aroylpropionic acids (**1**) with thallium(III) nitrate causes smooth 1,2-aryl migration to give dimethyl 2-arylsuccinates (**2**) in good yields. Similar transformation of 5-arylfuran-2(3*H*)-ones (**3**) with thallium(III) nitrate to 1-methyl 2-arylsuccinates (**4**) is also described.

Keywords——oxidative 1,2-aryl migration; thallium(III) nitrate; dimethyl 2-arylsuccinate; 1-methyl 2-arylsuccinate; 2-arylsuccinic acid; indan-1-carboxylic acid

McKillop and Taylor reported the oxidative 1,2-aryl migration of alkyl aryl ketones in the presence of thallium(III) nitrate (TTN) to give 2-arylalkanoates.[1] The migration proceeds smoothly in high yield and provides a simple procedure for the preparation of 2-arylalkanoic acids, which are antiinflammatory agents.[2] The work has prompted us to apply the migration to the preparation of 2-arylsuccinates, which are key intermediates in the synthesis of indan-1-carboxylic acids possessing antiinflammatory activity.[3] We describe here the oxidative 1,2-aryl migration reaction of 3-aroylpropionic acids (**1**) and 5-arylfuran-2(3*H*)-ones (**3**) to dimethyl 2-arylsuccinates (**2**) and 1-methyl 2-arylsuccinates (**4**), respectively (Chart 1), using TTN.



1a–g **2a–d** **3a–g** **4a–g**

a: Ar = C_6H_5
b: Ar = *p*-Br–C_6H_4
c: Ar = *p*-iso-Bu–C_6H_4
d: Ar = 2-thienyl
e: Ar = *p*-Me–C_6H_4
f: Ar = *p*-MeO–C_6H_4
g: Ar = *p*-cyclohexyl–C_6H_4

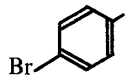
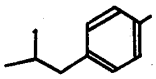
Chart 1

Transformation of 3-Aroylpropionic Acids (1) to Dimethyl 2-Arylsuccinates (2) with TTN

Treatment of 3-phenylpropionic acid (**1a**) with TTN in trimethyl orthoformate in the presence of perchloric acid at room temperature resulted in smooth 1,2-aryl migration to give dimethyl 2-phenylsuccinate (**2a**) in 90% yield. The transformation of **1b—d** to **2b—d** proceeded similarly. The results are summarized in Table I. The above approach to dimethyl 2-arylsuccinates (**2**) from 3-aroylpropionic acids (**1**) constitutes a versatile synthetic method for conversion of aromatic compounds to 2-arylsuccinic acids. Thus, benzene was converted into **1a** by Friedel–Crafts reaction with succinic anhydride.[4] The oxidative 1,2-aryl migration of **1a** with TTN then gave **2a**, which was readily hydrolyzed with alkaline solution to give 2-phenylsuccinic acid.[5] This conversion can be adapted to provide a simple preparation of 2-arylsuccinic acids, in contrast to classical methods.[6] 2-Arylsuccinic acids have been converted to indan-1-carboxylic acids in three steps.[3]

The mechanism of the above transformation[7] can be represented as shown in Chart 2. Initially esterification of **1** occurs to afford the methyl ester **5**, as confirmed by thin layer

TABLE I. Transformation of **1** to **2** with TTN

Compound No.	Ar	Reaction temp. (°C)	Reaction time (h)	Yield (%)
2a		R.T.	3	90
2b		R.T.	4	86
2c		R.T.	5	89
2d		R.T.	4	88

R.T. = room temp.

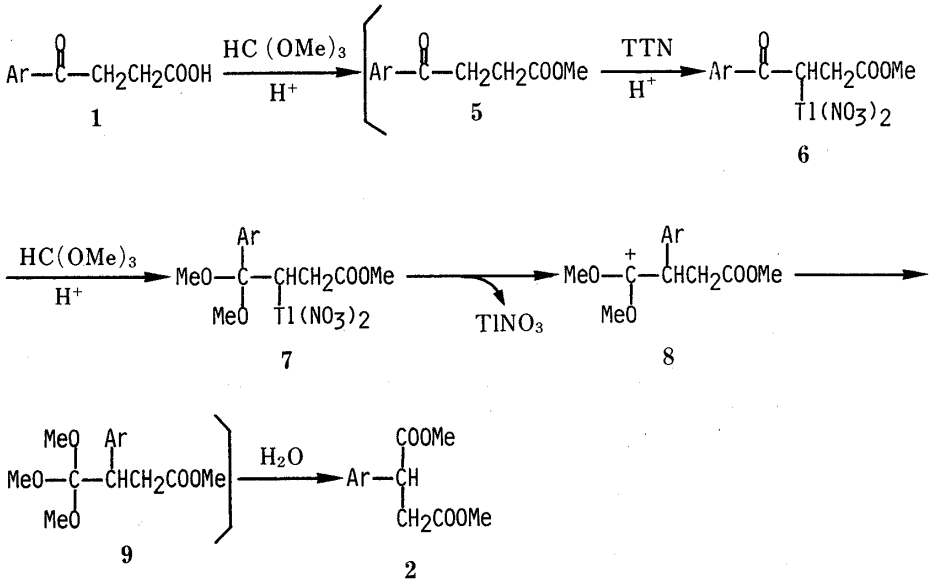


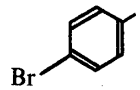
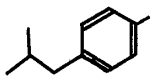
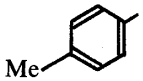
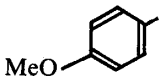
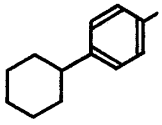
Chart 2

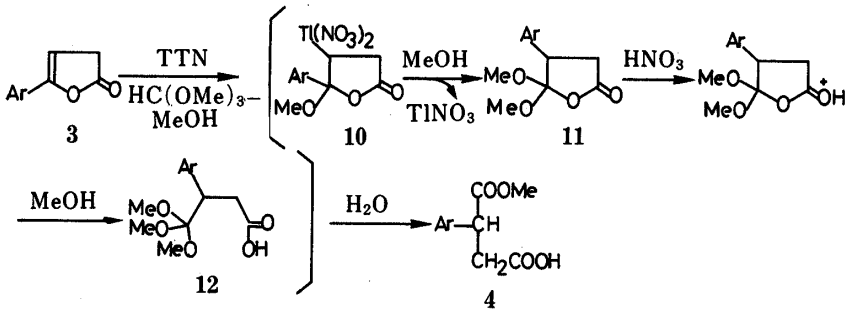
chromatography (TLC) and gas chromatography. The enol form of the ester **5**, which is produced by acid-catalyzed enolization, reacts with TTN to give the thallium (III) salt **6**, which is acetalized with trimethyl orthoformate to give the intermediate **7**. Loss of thallium (I) nitrate with concomitant 1,2-aryl migration leads to the stable carbonium ion **8**. Solvolysis of the ion **8** and hydrolysis of the resultant ortho-ester **9** gives **2**.

Transformation of 5-Arylfuran-2(3*H*)-ones (3) to 1-Methyl 2-Arylsuccinates (4) with TTN

As an alternative preparation of 2-arylsuccinates, 1,2-aryl migration of 5-phenylfuran-2(3*H*)-ones (**3**), readily prepared from **1a** and acetic anhydride,[8] with TTN was examined. The migration was found to proceed cleanly and smoothly in trimethyl orthoformate-methanol at room temperature to afford 1-methyl 2-phenylsuccinate (**4a**) in 90% yield. The structure of **4a** was determined from the spectral data and by comparison of the melting points with those of authentic samples of the 1-methyl ester (**4a**) and 4-methyl ester (**4a'**).[9] Some typical transformations under similar conditions are summarized in Table II. From these data it is evident that the present transformation provides a simple and rapid proce-

TABLE II. Transformation of **3** to **4** with TTN

Compound No.	Ar	Reaction temp. (°C)	Reaction time (min)	Yield (%)
4a		R.T.	30	90
4b		R.T.	20	71
4c		R.T.	20	92
4d		R.T.	15	79
4e		R.T.	20	90
4f		R.T.	20	79
4g		R.T.	20	76



3 10 11 12 4

Chart 3

dure for the preparation of 1-methyl 2-arylsuccinates (**4**), which can be cyclized to the methyl esters of 3-oxoindan-1-carboxylic acids by heating with polyphosphoric acid (PPA).[10] The methyl ester is useful for the preparation of indan-1-carboxylic acids.

The mechanism of the above transformation can be represented as shown in Chart 3. Methoxythallation of **3** occurs to give the intermediate **10**, then elimination of thallium (I) nitrate and subsequent 1,2-aryl migration followed by methanolysis results in the acetal (**11**). Protonation of the carbonyl group of the acetal **11** with nitric acid generated by loss of thallium (I) nitrate followed by methanolysis gives the ortho-ester (**12**). Hydrolysis of the ortho-ester **12** gives **4**.

The present transformations of **1** and **3** with TTN proceed smoothly under mild conditions to afford **2** and **4** in high yields, respectively. Consequently, both transformations provide versatile procedures for the preparation of key intermediates (**2** and **4**) for the synthesis of indan-1-carboxylic acids.

Experimental

All melting points are uncorrected. Infrared (IR) spectra were recorded on a JASCO IRA-1 spectrometer, and nuclear magnetic resonance (NMR) spectra on a Hitachi R-20A (60 MHz) or a Hitachi R-22 (90 MHz) spectrometer (with tetramethylsilane as an internal standard). Low- and high-resolution mass spectra (MS) were obtained with a JEOL JMS D-300 instrument, with a direct inlet system. Gas chromatography was run on a Shimadzu GC-4B instrument. Column chromatography was carried out on Merck Silica gel 60.

The starting 3-aroylpropionic acids (**1**) were prepared by the reported method.[4]

General Procedure for the Transformation of 3-Aroylpropionic Acids (1) to Dimethyl 2-Arylsuccinates (2) with TTN——A stirred solution of TTN (1 mmol) and **1** (1 mmol) in 5 ml of trimethyl orthoformate was treated dropwise with 70% $HClO_4$ (4 mmol). The mixture was stirred for several hours at room temperature, then the precipitate of thallium (I) nitrate was filtered off. The organic layer was washed with water and dried over $MgSO_4$. Removal of the solvent gave crude **2**. The crude **2** was purified by recrystallization or silica gel column chromatography. Yields of **2** are summarized in Table I.

Dimethyl 2-Phenylsuccinate (2a): mp 54—55 °C (hexane) (lit.[11] mp 54.5—55.5 °C). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1730. ^{1}H-NMR ($CDCl_3$) δ: 2.62 (1H, dd, J=6, 17 Hz, $-CH_2COOCH_3$), 3.26 (1H, dd, J=10, 17 Hz, $-CH_2COOCH_3$), 3.63 (6H, s, $-COOCH_3 \times 2$), 4.07 (1H, dd, J=6, 10 Hz, $-CHCOOCH_3$), 7.23 (5H, s, ArH).

Dimethyl 2-(4-Bromophenyl)succinate (**2b**): A colorless oil (lit.[12] mp 26—28 °C). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1735. ^{1}H-NMR ($CDCl_3$) δ: 2.61 (1H, dd, J=6, 17 Hz, $-CH_2COOCH_3$), 3.19 (1H, dd, J=9.5, 17 Hz, $-CH_2COOCH_3$), 3.66 (6H, s, $-COOCH_3 \times 2$), 4.05 (1H, dd, J=6, 9.5 Hz, $CHCOOCH_3$), 7.10 (2H, d, J=8.5 Hz, ArH), 7.42 (2H, d, J=8.5 Hz, ArH). High-resolution MS Calcd for $C_{12}H_{13}BrO_4$: 299.9998. Found: 300.0003.

Dimethyl 2-(4-Isobutylphenyl)succinate (**2c**): mp 34.5—35.5 °C (pet. ether). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1730. ^{1}H-NMR ($CDCl_3$) δ: 0.88 [6H, d, J=7 Hz, $-CH(CH_3)_2$], 1.5—2.1 [1H, m, $-CH(CH_3)_2$], 2.44 [2H, d, J=7 Hz, $-CH_2-CH(CH_3)_2$], 2.60 (1H, dd, J=5, 17 Hz, $-CHCOOCH_3$), 3.19 (1H, dd, J=10, 17 Hz, $-CH_2COOCH_3$), 3.63 (6H, s, $-COOCH_3 \times 2$), 4.03 (1H, dd, J=5, 10 Hz, $-CHCOOCH_3$), 7.09 (4H, s, ArH). *Anal.* Calcd for $C_{16}H_{22}O_4$: C, 69.04; H, 7.97. Found: C, 68.97; H, 8.04.

Dimethyl 2-(2-Thienyl)succinate (**2d**): mp 31—32 °C (hexane). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1740. ^{1}H-NMR ($CDCl_3$) δ: 2.27 (1H, dd, J=6, 16.5 Hz, $-CH_2COOCH_3$), 3.22 (1H, dd, J=10, 16.5 Hz, $-CH_2COOCH_3$), 3.64 (3H, s, $-COOCH_3$), 3.67 (3H, s, $-COOCH_3$), 4.34 (1H, dd, J=6, 16.5 Hz, $-CHCOOCH_3$), 6.88—7.30 (3H, m, ArH). *Anal.* Calcd for $C_{10}H_{12}O_4S$: C, 52.62; H, 5.30; S, 14.04. Found: C, 52.35; H. 5.20; S, 13.99.

5-Arylfuran-2(3*H*)-ones (**3**) were prepared according to the literature procedure.[8]

5-(4-Isobutylphenyl)furan-2(3*H*)-one (3c)——A mixture of **1c** (234 mg, 1 mmol) and acetic anhydride (204 mg, 2 mmol) was warmed at 100 °C for 1 h. After cooling, the reaction mixture was poured into cold 10% K_2CO_3 solution and extracted with ether. The ether layer was washed with cold 10% K_2CO_3 solution, dried over $MgSO_4$ and concentrated. The residue was purified by column chromatography on silica gel with benzene as the eluent to give pure **3c** (208 mg, 86%). mp 124.5—126 °C. IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1795. ^{1}H-NMR ($CDCl_3$) δ: 1.89 [6H, d, J=6 Hz, $-C(CH_3)_2$], 1.6—2.3 [1H, m, $-CH(CH_3)_2$], 2.46 (2H, d, J=6 Hz, $ArCH_2$), 3.32 (2H, d, J=3 Hz, $=CHCH_2-$), 5.64 (1H, t, J=3 Hz, $=CH$), 7.08 (2H, d, J=8.5 Hz, ArH), 7.45 (2H, d, J=8.5 Hz, ArH). High-resolution MS Calcd for $C_{14}H_{16}O_2$: 216.1147. Found: 216.1145.

5-(2-Thienyl)furan-2(3*H*)-one (**3d**): Yield: 72%. mp 82—83 °C. IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1800. ^{1}H-NMR ($CDCl_3$) δ: 3.38 (2H, d, J=3 Hz, $=CHCH_2-$), 5.56 (1H, t, J=3 Hz, $=CH$), 6.91—7.42 (3H, m, ArH). High-resolution MS Calcd for $C_8H_6O_2S$: 166.0086. Found: 166.0083.

5-(4-Cyclohexylphenyl)furan-2(3*H*)-one (**3g**): Yield: 75%. mp 85—86 °C. IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1780 (S). ^{1}H-NMR

($CDCl_3$) δ: 1.1—3.0 (11H, m, cyclohexyl), 3.36 (2H, d, J=3 Hz, =CHC$\underline{H}_2$–), 5.65 (1H, t, J=3 Hz, =CH), 7.17 (2H, d, J=8 Hz, ArH), 7.48 (2H, d, J=8.5 Hz, ArH). *Anal.* Calcd for $C_{16}H_{18}O_2$: C, 79.31; H, 7.49. Found: C, 79.13; H, 7.56.

General Procedure for the Transformation of 5-Arylfuran-2(3*H*)-ones (3) to 1-Methyl 2-Arylsuccinates (4) with TTN——TTN (445 mg, 1 mmol) was added slowly to a solution of **3** (1 mmol) in 5 ml of methanol–trimethyl orthoformate (1 : 3). The resulting mixture was stirred for 15—30 min at room temperature. The precipitate of thallium (I) nitrate was filtered off. A solution of KI–$NaHSO_3$–K_2CO_3 (1 g : 1 g : 3 g, 30 ml) was added to the filtrate and the whole was stirred for 5 min. The aqueous layer was washed with ether, then the aqueous layer was acidified with conc. HCl and extracted with CH_2Cl_2 (10 ml × 3). The CH_2Cl_2 layer was dried over $MgSO_4$ and concentrated to afford crude **4**, which was recrystallized from H_2O–EtOH to give pure **4**. Reaction conditions and yields are summarized Table II.

1-Methyl 2-Phenylsuccinate (**4a**): mp 102—103 °C (H_2O–EtOH) (lit.[9] mp 102 °C, lit.[13] mp 100—101 °C). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1710, 1730. ^{1}H-NMR ($CDCl_3$) δ: 2.67 (1H, dd, J=5.5, 17 Hz, –C$\underline{H}$COOH), 3.26 (1H, dd, J=9.5, 17 Hz, –C$\underline{H}$COOH), 3.64 (3H, s, –$COOCH_3$), 4.05 (1H, dd, J=5.5, 9.5 Hz, –C$\underline{H}$$COOCH_3$), 7.25 (5H, s, ArH), 9.80 (1H, br s, –COOH). MS m/z: 208 (M^+).

1-Methyl 2-(4-Bromophenyl)succinate (**4b**): mp 154.5—155.5 °C (H_2O–EtOH). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1710, 1730. ^{1}H-NMR ($CDCl_3$) δ: 2.56 (1H, dd, J=6, 17 Hz, –C$\underline{H}$COOH), 3.03 (1H, dd, J=9.5, 17 Hz, –C$\underline{H}$COOH), 3.58 (3H, s, –$COOCH_3$), 4.02 (1H, dd, J=6, 9.5 Hz, –C$\underline{H}$$COOCH_3$), 7.20 (2H, d, J=8.5 Hz, ArH), 7.48 (2H, d, J=8.5 Hz, ArH). *Anal.* Calcd for $C_{11}H_{11}BrO_4$: C, 46.02; H, 3.86; Br, 27.83. Found: C, 45.90; H, 3.78; Br, 27.85.

1-Methyl 2-(4-Isobutylphenyl)succinate (**4c**): mp 87—88.5 °C (H_2O–EtOH). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1715, 1730. ^{1}H-NMR ($CDCl_3$) δ: 0.87 [6H, d, J=7 Hz, –$C(CH_3)_2$], 1.6—2.1 [1H, m, –C$\underline{H}$$(CH_3)_2$], 2.45 (2H, d, J=7 Hz, –CH_2Ar), 2.64 (1H, dd, J=5.5, 17 Hz, –C$\underline{H}$COOH), 3.24 (1H, dd, J=9.5, 17 Hz, –C$\underline{H}$COOH), 4.01 (1H, dd, J=5.5, 9.5 Hz, –C$\underline{H}$$COOCH_3$), 7.08 (4H, s, ArH), 11.18 (1H, br s, –COOH). *Anal.* Calcd for $C_{15}H_{20}O_4$: C, 68.16; H, 7.63. Found: C, 68.11, H, 7.77.

1-Methyl 2-(2-Thienyl)succinate (**4d**): mp 69—71 °C (H_2O–EtOH). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1715, 1730. ^{1}H-NMR ($CDCl_3$) δ: 2.78 (1H, dd, J=6, 17 Hz, –C$\underline{H}$COOH), 3.29 (1H, dd, J=9, 17 Hz, –C$\underline{H}$COOH), 3.70 (3H, s, –$COOCH_3$), 4.83 (1H, dd, J=6, 9 Hz, –C$\underline{H}$$COOCH_3$), 6.80—7.25 (3H, m, ArH), 10. 65 (1H, br s, –COOH). High-resolution MS Calcd for $C_9H_{10}O_4S$: 214.0281. Found: 214.0279.

1-Methyl 2-(4-Methylphenyl)succinate (**4e**): mp 145 °C (H_2O–EtOH). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1710, 1730. ^{1}H-NMR ($CDCl_3$) δ: 2.30 (3H, s, Ar-CH_3), 2.62 (1H, dd, J=6, 17 Hz, –C$\underline{H}$COOH), 3.23 (1H, dd, J=9.5, 17 Hz, –C$\underline{H}$COOH), 3.63 (3H, s, –$COOCH_3$), 4.00 (1H, dd, J=6, 9.5 Hz, –C$\underline{H}$$COOCH_3$), 7.08 (4H, s, ArH), 8.26 (1H, br s, –COOH). *Anal.* Calcd for $C_{12}H_{14}O_4$: C, 64.85; H, 6.35. Found: C, 64.81; H, 6.37.

1-Methyl 2-(4-Methoxyphenyl)succinate (**4f**): mp 112—113 °C (H_2O–EtOH). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1710, 1730. ^{1}H-NMR ($CDCl_3$)δ: 2.63 (1H, dd, J=6, 17 Hz, –C$\underline{H}$COOH), 3.23 (1H, dd, J=9.5, 17 Hz, –C$\underline{H}$COOH), 3.63 (3H, s, –$COOCH_3$), 3.75 (3H, s, Ar-OCH_3), 4.00 (1H, dd, J=6, 9.5 Hz, –C$\underline{H}$$COOCH_3$), 6.79 (2H, d, J=8 Hz, ArH), 7.16 (2H, d, J=8 Hz, ArH), 10.70 (1H, br s, –COOH). *Anal.* Calcd for $C_{12}H_{14}O_5$: C, 60.50; H, 5.92. Found: C, 60.27; H, 5.95.

1-Methyl 2-(4-Cyclohexylphenyl)succinate (**4g**): mp 107—108 °C (H_2O–EtOH). IR $\nu_{max}^{CHCl_3}$ cm^{-1}: 1715, 1730. ^{1}H-NMR ($CDCl_3$) δ: 1.1—2.2 (11H, m, cyclohexyl), 2.60 (1H, dd, J=6, 17 Hz, –C$\underline{H}$COOH), 3.16 (1H, dd, J=10, 17 Hz, –C$\underline{H}$COOH), 3.58 (3H, s, –$COOCH_3$), 4.02 (1H, dd, J=6, 10 Hz, –C$\underline{H}$$COOCH_3$), 7.17 (4H, s, ArH), 8.2 (1H, br s, –COOH). *Anal.* Calcd for $C_{17}H_{22}O_4$: C, 70.32; H, 7.64. Found: C, 70.07; H, 7.69.

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