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# Syntheses of Arenediacetic Esters and Acetonyl-Substituted Arylacetic Esters by Means of Friedel–Crafts Reaction with α-Acyl-α-chlorosulfides

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Friedel–Crafts reaction of the phenylacetates **5a**, **b** with ethyl α-chloro-α-(methylthio)acetate (**1**) in the presence of stannic chloride gave the α-methylthio-1,4-benzenediacetates **7a**, **b**. The reactions of biphenyl, diphenylmethane, and diphenyl ether with an excess amount of **1** gave directly the corresponding disubstituted products **10a**—**c**. Desulfurization of **7a**, **b** and **10a**—**c** gave the corresponding diacetates **8a**, **b** and **11a**—**c**. Methyl 4-(2-oxopropyl)phenylacetate (**14**) was prepared by reaction of methyl phenylacetate with α-chloro-α-(methylthio)acetone (**2**) followed by desulfurization of the resulting product. Methyl 2-(2-furyl)propionate (**19**) reacted with **2** in the presence of zinc chloride to give the 2,5-disubstituted furan **20**, whose desulfurization gave methyl 2-[5-(2-oxopropyl)-2-furyl]propionate (**21**).

**Keywords**——arenediacetic ester; Friedel–Crafts reaction; α-chlorosulfide; nonactic acid; nonactin; desulfurization; Raney nickel; stannic chloride; zinc chloride

We have recently developed a simple and high yield synthetic method for arylacetic esters **5** or arylacetones **6** which involves Friedel–Crafts reaction of arenes with α-acyl-α-chlorosulfide **1** (or **2**) and successive desulfurization of the resulting products **3** (or **4**).[1] In the present paper, we wish to report an application of this method to syntheses of arenediacetic esters and acetonyl-substituted arylacetic esters.

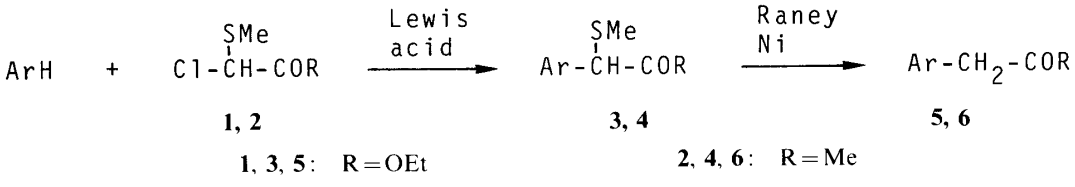
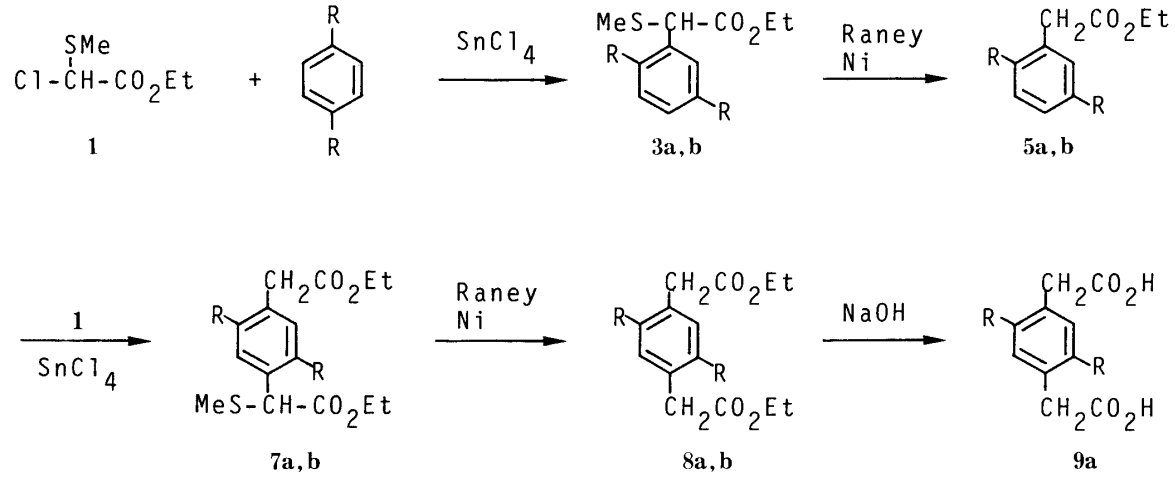



Chart 1

During the previous investigation, we noticed that benzene or *p*-xylene gave no di- or poly-alkylated products even if treated with an excess amount of **1**. Further treatment of the mono-alkylated product **3a** ($Ar=C_6H_5$) or **3b** ($Ar=2,5\text{-}Me_2C_6H_3$) with **1** (excess) in dichloromethane ($CH_2Cl_2$) in the presence of stannic chloride ($SnCl_4$) (excess) also resulted in recovery of the unreacted starting material even under refluxing conditions. This result can be explained by assuming that the initially introduced α-ethoxycarbonyl-α-(methylthio)methyl group functions as a strong electron-withdrawing group as a result of co-ordination of the Lewis acid to the sulfur and carbonyl oxygen atoms.

The desired diacetates **7a** and **7b** were found to be obtainable by starting from the esters

**5a** and **5b**, respectively. Thus, when the ester **5a** was allowed to react with **1** (1 eq) at room temperature in the presence of an equimolar amount of $SnCl_4$, the reaction was slow but the diester **7a** was obtained in 57% yield. The use of 2 mol eq of $SnCl_4$ improved the yield of **7a** to

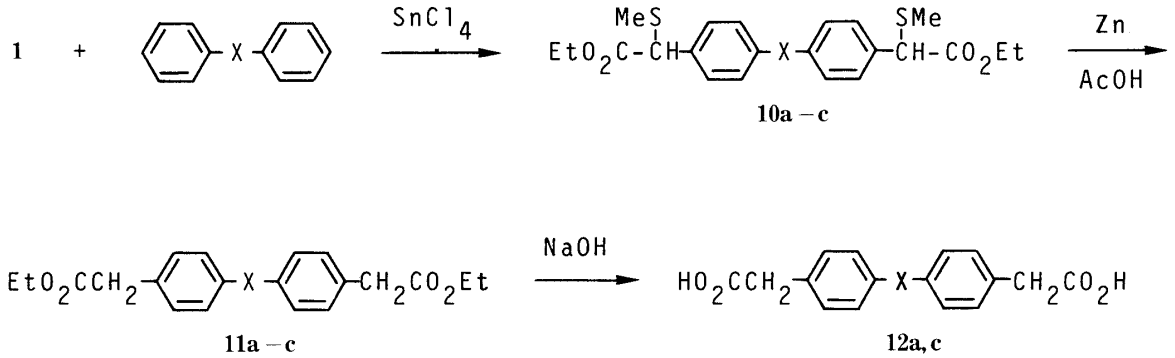



**a**: R=H, **b**: R=Me

Chart 2

78%. On the other hand, the reaction of **5b** with **1** occurred smoothly in the presence of an equimolar amount of $SnCl_4$, giving **7b** in 95% yield. The products **7a**, **b** were desulfurized with Raney nickel to give the diacetates **8a**, **b**. Alkaline hydrolysis of **8a** yielded the diacetic acid **9a**.

In contrast to the case of benzene, biphenyl gave directly the diacetate **10a** in 75% yield, when treated with 3 eq of **1**. Similarly, the reaction of diphenylmethane and diphenyl ether afforded **10b** (69%) and **10c** (75%), respectively. Desulfurization of **10a**—**c** with zinc dust in acetic acid gave the corresponding diacetates **11a**—**c**. Hydrolysis of **11a** and **11c** yielded the diacetic acids **12a** and **12c**, respectively.




**a**: X=no atom, **b**: $X=CH_2$, **c**: X=O

Chart 3

In principle, the stepwise procedure can be used for the introduction of two different $RCOCH_2$– groups into the aromatic ring. This possibility was tested by the reaction of methyl phenylacetate with α-chloro-α-(methylthio)acetone (**2**). This reaction was found to be more sluggish than that of **5a** with **1**. The best result was obtained by carrying out the reaction using 5 eq of **2** and 2 eq of $SnCl_4$, giving the product **13** in 75% yield.[2] Desulfurization of **13** with zinc dust in acetic acid gave the *p*-acetonylphenylacetate **14** in 95% yield.

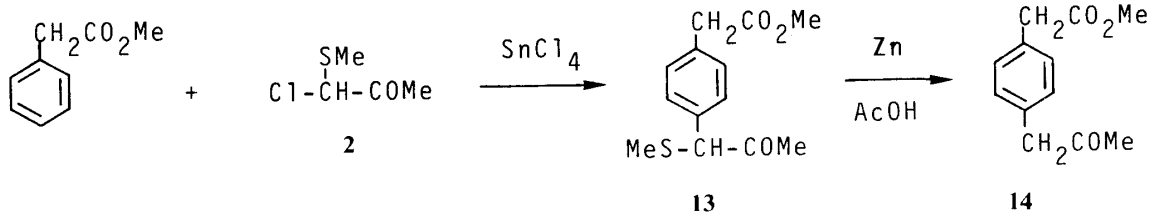


Chart 4

Nonactic acid (**22**) is the subunit of the macrotetrolide antibiotic nonactin. Among several syntheses[3] of compound **22**, the method described by Gerlach and Wetter[4] is of particular interest because it makes use of the 2,5-disubstituted furan **21** as a key intermediate. We applied our method to the synthesis of **21**. The synthetic route is outlined in Chart 5. The

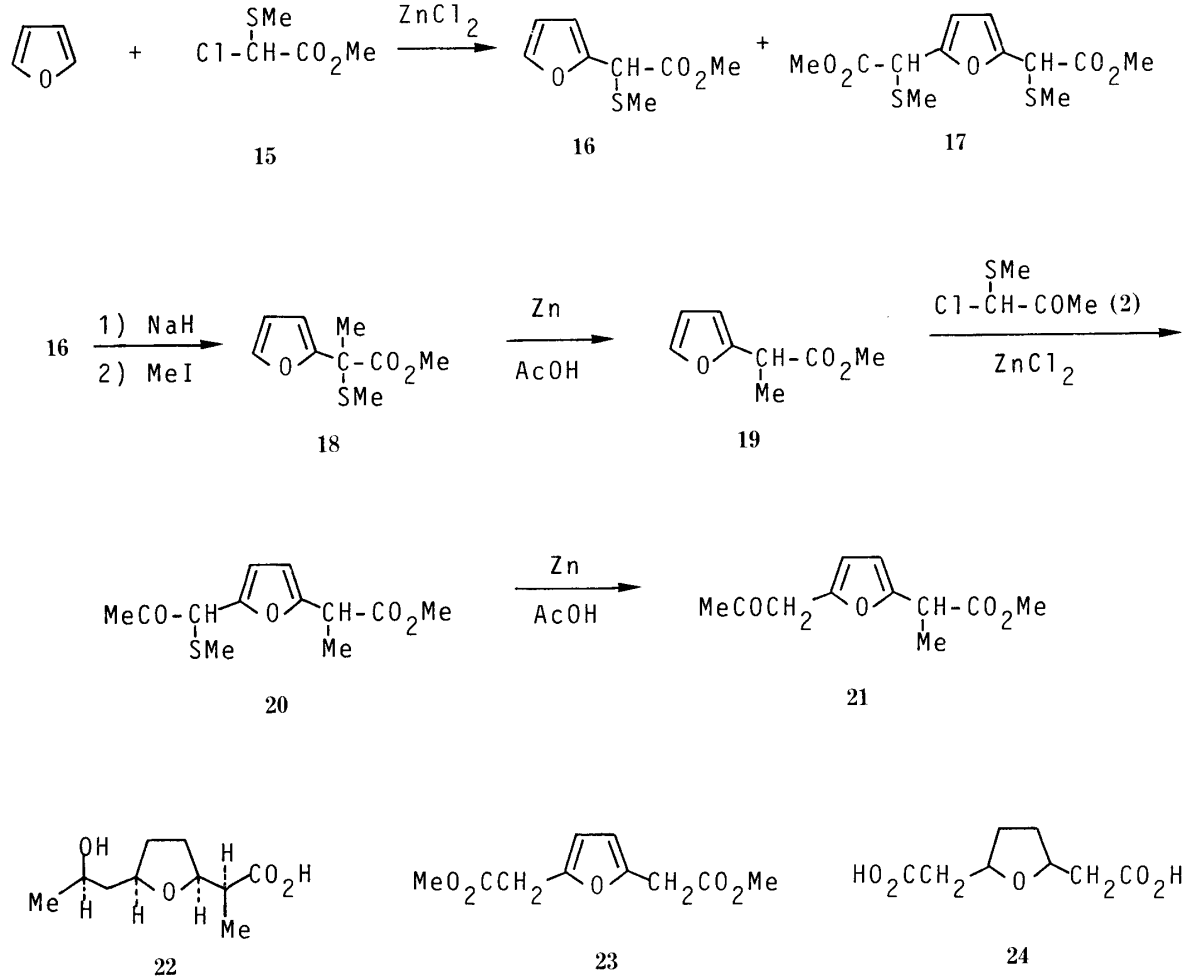


Chart 5

reaction of furan with methyl α-chloro-α-(methylthio)acetate (**15**) in the presence of zinc chloride ($ZnCl_2$) gave methyl α-methylthio-2-furylacetate (**16**) and the disubstituted furan **17**[5] in 59 and 12% yields, respectively. Methylation of **16** by treatment with sodium hydride and then methyl iodide in dimethylformamide afforded the 2-(2-furyl)propionate **18**. Reduction of **18** with zinc dust in acetic acid gave **19**, which was then subjected to the Friedel–Crafts reaction with the chloride **2** in the presence of $ZnCl_2$ to give the 2,5-disubstituted furan **20**. Desulfurization of **20** with zinc dust in acetic acid afforded **21**. A three-step conversion of **21**

into nonactic acid (**22**) has already been described in the literature.[4]

Finally, the by-product **17** obtained from the reaction of furan with **15** was converted into dimethyl 2,5-furandiacetate (**23**) by reduction with Raney nickel. The diacetate **23** has recently been described[6] as an intermediate for the synthesis of tetrahydro-2,5-furandiacetic acid (**24**: a mixture of *cis* and *trans* isomers), which is a compound isolated from human urine.

## Experimental[7]

**Ethyl 2,5-Dimethylphenylacetate (5b)**——Compound **3b**[1a] (4.31 g, 18.1 mmol) was heated under reflux in ethanol (100 ml) containing Raney nickel (W-2) (*ca.* 15 g) for 4 h. After removal of the Raney nickel, the solvent was evaporated off and the residue was distilled *in vacuo* to give **5b** (2.95 g, 85%), bp 104—106 °C (15 mmHg), lit.[8] 118—119 °C (12 mmHg). $^1$H-NMR ($CDCl_3$) $\delta$: 1.23 (3H, t, $J$=7 Hz, $OCH_2\underline{CH}_3$), 2.24 (6H, s, $ArC\underline{H}_3 \times 2$), 3.51 (2H, s, $ArC\underline{H}_2$), 4.18 (2H, q, $J$=7 Hz, $OC\underline{H}_2$), 6.90 (3H, s, arom.).

**Diethyl α-Methylthio-1,4-benzenediacetate (7a) and Diethyl α-Methylthio-2,5-dimethyl-1,4-benzenediacetate (7b)**——$SnCl_4$ (1.04 g, 4 mmol for **5a** or 0.52 g, 2 mmol for **5b**) was added to a solution of **1**[1a] (337 mg, 2 mmol) and **5a** or **5b** (2 mmol) in dry dichloromethane ($CH_2Cl_2$) (10 ml) at room temperature, and the mixture was stirred at the same temperature for 24 h. The reaction was quenched with water (5 ml) and the organic layer was separated, then the aqueous layer was extracted with $CH_2Cl_2$ (10 ml × 2). The combined organic layers were dried ($MgSO_4$), the solvent was evaporated off, and the residue was chromatographed on silica gel (benzene–ethyl acetate, 1 : 1) to give **7a** (485 mg, 78%) or **7b** (616 mg, 95%) as an oil, whose physical data are listed in Table I.

**Diethyl 1,4-Benzenediacetate (8a) and Diethyl 2,5-Dimethyl-1,4-benzenediacetate (8b)**——By a procedure similar to that described for the preparation of **5b**, compounds **7a** (332 mg, 1.12 mmol) and **7b** (502 mg, 1.55 mmol) were desulfurized with Raney nickel (W-2) (*ca.* 2 g) to give **8a** (241 mg, 86%) and **8b** (422 mg, 98%), respectively. **8a**: mp 57.5—58.5 °C (from *n*-hexane), lit.[9] 58—59 °C. $^1$H-NMR ($CDCl_3$) $\delta$: 1.25 (6H, t, $J$=7 Hz, $OCH_2\underline{CH}_3 \times 2$), 3.58 (4H, s, $ArC\underline{H}_2 \times 2$), 4.14 (4H, q, $J$=7 Hz, $OC\underline{H}_2 \times 2$), 7.25—7.35 (4H, m, arom). **8b**: mp 55—56 °C (from *n*-hexane–ethyl ether), lit.[8] 55.5—56.5 °C. $^1$H-NMR ($CDCl_3$) $\delta$: 1.24 (6H, t, $J$=7 Hz, $OCH_2\underline{CH}_3 \times 2$), 2.25 (6H, s, $ArC\underline{H}_3 \times 2$), 3.45 (4H, s, $ArC\underline{H}_2 \times 2$), 4.08 (4H, q, $J$=7 Hz, $OC\underline{H}_2 \times 2$), 6.90 (2H, s, arom.).

**1,4-Benzenediacetic Acid (9a)**——A solution of **8a** (375 mg, 1.5 mmol) in ethanol (3 ml) was added to a solution of sodium hydroxide (152 mg, 3.8 mmol) in water (2 ml), and the mixture was heated under reflux for 9 h, then cooled. The reaction mixture was acidified to pH 1 with concentrated hydrochloric acid and the precipitate was collected and recrystallized to give **9a** (166 mg, 57%), mp 249—251 °C (from ethanol), lit.[10] 253—254 °C.

**Friedel–Crafts Reaction of Biphenyl, Diphenylmethane, and Diphenyl Ether with 1**——$SnCl_4$ (2.34 g, 9 mmol) was added to a solution of **1** (1.01 g, 6 mmol) and biphenyl, diphenylmethane, or diphenyl ether (2 mmol) in dry $CH_2Cl_2$ (10 ml) at 0 °C (−40 °C for diphenyl ether), and the mixture was stirred at the same temperature for 30 min. The reaction was quenched with water (5 ml) and the organic layer was separated. The aqueous layer was extracted with $CH_2Cl_2$ (10 ml × 2) and the combined organic layers were washed successively with saturated sodium bicarbonate solution (10 ml) and water (10 ml), then dried ($MgSO_4$). The solvent was evaporated off and the residue was chromatographed on silica gel (*n*-hexane–ethyl acetate, 3 : 1) to give **10a** (628 mg, 75%), **10b** (597 mg, 69%), or **10c** (652 mg, 75%) as an oil, whose physical data are listed in Table I.

**Diethyl [1,1′-Biphenyl]-4,4′-diacetate (11a), Diethyl 4,4′-Methylenebisbenzeneacetate (11b), and Diethyl 4,4′-Oxybisbenzeneacetate (11c)**——Zinc dust (4.55 g) was added to a solution of **10a**, **10b**, or **10c** (3 mmol) in acetic acid (6 ml), and the mixture was heated with vigorous stirring at 100—110 °C for 1 h, then cooled. Water (20 ml) and $CH_2Cl_2$ (30 ml) were added and the inorganic materials were filtered off. The organic layer was separated and the aqueous layer was extracted with $CH_2Cl_2$, then the combined organic layers were dried ($MgSO_4$). The solvent was evaporated off and the residue was recrystallized (for **10a** or **10b**) or chromatographed (for **10c**, silica gel, *n*-hexane–ethyl acetate, 3 : 1) to give **11a** (881 mg, 90%), **11b** (868 mg, 85%), or **11c** (882 mg, 80%). **11a**: mp 49—50 °C (from *n*-hexane). **11b**: mp 19.5—20.5 °C (from benzene–*n*-hexane). **11c**: Oil. Other physical data for **11a**—**c** are listed in Table I.

**[1,1′-Biphenyl]-4,4′-diacetic Acid (12a) and 4,4′-Oxybisbenzeneacetic Acid (12c)**——By a procedure similar to that described for the preparation of **9a**, compounds **11a** and **11c** (1.5 mmol) were hydrolyzed with sodium hydroxide to give **12a** (271 mg, 67%) and **12c** (292 mg, 68%), respectively. **12a**: mp 270—275 °C (from ethanol), lit.[10] 282—284 °C or lit.[11] 270—273 °C. **12c**: mp 226—228 °C (from ethanol), lit.[12] 227.5—230 °C.

**Methyl 4-(1-Methylthio-2-oxopropyl)phenylacetate (13)**——$SnCl_4$ (1.04 g, 4 mmol) was added to a solution of methyl phenylacetate (300 mg, 2 mmol) and **2**[1b] (1.39 g, 10 mmol) in dry $CH_2Cl_2$ (10 ml) at room temperature, and the mixture was stirred at the same temperature for 24 h. The reaction mixture was worked-up in a similar manner to that described for the preparation of **10a**—**c** to give **13** (353 mg, 70%) as an oil, whose physical data are listed in Table I.

**Methyl 4-(2-Oxopropyl)phenylacetate (14)**——Zinc dust (1 g) was added to a solution of **13** (300 mg, 1.19 mmol) in acetic acid (3 ml), and the mixture was heated with vigorous stirring at 100—110 °C for 1 h, then cooled. The reaction mixture was worked up in a similar manner to that described for the preparation of **11a**—**c** to give **14**

TABLE I. Arenediacetic Esters (**7a—b**, **10a—c**, **11a—c**, **17**), Acetonyl-Substituted Phenylacetic Esters (**13**, **14**), and 2-(2-Furyl)propionic Esters (**18**, **20**)

| Compd. No. | Analysis (%) Calcd (Found) C | H | Formula | IR $\nu_{max}^{CHCl_3}$ cm$^{-1}$ | $^1$H-NMR ($CDCl_3$, $\delta$) |
|---|---|---|---|---|---|
| **7a** | 60.79 (60.97 | 6.80 6.83) | $C_{15}H_{20}O_4S$ | 1730 | 1.26 (3H, t), 2.07 (3H, s), 3.60 (2H, s), 4.15 (2H, q), 4.20 (2H, q), 4.47 (1H, s), 7.2—7.4 (4H, m) |
| **7b** | 62.94 (62.88 | 7.46 7.57) | $C_{17}H_{24}O_4S$ | 1730 | 1.25 (6H, t), 2.10 (3H, s), 2.27 (3H, s), 2.35 (3H, s), 3.55 (2H, s), 4.15 (2H, q), 4.20 (2H, q), 4.67 (1H, s), 6.99 (1H, br s), 7.26 (1H, br s) |
| **10a** | 63.13 (62.65 | 6.26 6.18) | $C_{22}H_{26}O_4S$ | 1725 | 1.27 (6H, t), 2.10 (6H, s), 4.20 (4H, q), 4.53 (2H, s), 7.55 (8H, s) |
| **10b** | 63.86 (63.69 | 6.52 6.56) | $C_{23}H_{28}O_4S$ | 1725 | 1.25 (6H, t), 2.03 (6H, s), 3.93 (2H, s), 4.18 (4H, q), 4.44 (2H, s), 7.05—7.45 (8H, m) |
| **10c** | 60.81 (60.73 | 6.03 5.98) | $C_{22}H_{26}O_4S$ | 1725 | 1.28 (6H, t), 2.08 (6H, s), 4.22 (4H, q), 4.48 (2H, s), 6.9—7.5 (8H, m) |
| **11a** | 73.60 (73.42 | 6.79 6.80) | $C_{20}H_{22}O_4$ | 1725 | 1.26 (6H, t), 3.57 (4H, s), 4.18 (4H, q), 7.25—7.65 (8H, m) |
| **11b** | 74.09 (73.85 | 7.11 7.03) | $C_{21}H_{24}O_4$ | 1725 | 1.23 (6H, t), 3.56 (4H, s), 3.93 (2H, s), 4.13 (4H, q), 7.16 (8H, s) |
| **11c** | 70.16 (70.17 | 6.48 6.40) | $C_{20}H_{22}O_5$ | 1725 | 1.25 (6H, t), 3.57 (4H, s), 4.16 (4H, q), 6.85—7.35 (8H, m) |
| **13** | 252.0820[a] (252.0826) | | $C_{13}H_{16}O_3S$ | 1725 1705 | 2.01 (3H, s), 2.18 (3H, s), 3.61 (2H, s), 3.69 (3H, s), 4.49 (1H, s), 7.30 (4H, br s) |
| **14** | 206.0940[a] (206.0925) | | $C_{12}H_{14}O_3$ | 1725 1705 | 2.08 (3H, s), 3.61 (2H, s), 3.69 (5H, s), 7.20—7.35 (4H, m) |
| **17** | 304.0438[a] (304.0447) | | $C_{12}H_{16}O_5S_2$ | 1725 | 2.13 (6H, s), 3.74 (6H, s), 4.60 (2H, s), 6.43 (2H, s) |
| **18** | 200.0507[a] (200.0513) | | $C_9H_{12}O_3S$ | 1730 | 1.85 (3H, s), 2.03 (3H, s), 3.79 (3H, s), 6.25—6.60 (2H, m), 7.41 (1H, br s) |
| **20** | 256.0767[a] (256.0766) | | $C_{12}H_{16}O_4S$ | 1735 1705 | 1.54 (3H, d), 2.07 (3H, s), 2.29 (3H, s), 3.6—4.0 (4H, br), 3.73 (3H, s), 3.81 (1H, q), 4.51 (1H, s), 6.19 (1H, br s), 6.39 (1H, br s) |

*a*) High-resolution MS ($M^+$).

(232 mg, 95%) as an oil, whose physical data are listed in Table I.

**Methyl α-Methylthio-2-furanacetate (16) and Dimethyl α,α′-Bis(methylthio)-2,5-furandiacetate (17)**——$ZnCl_2$ (243 mg, 1.78 mmol) was added to a stirred solution of furan (121 mg, 1.78 mmol) and **15**[13] (275 mg, 1.78 mmol) in $CH_2Cl_2$ (20 ml) at 0 °C, and stirring was continued at room temperature for 1 h. The reaction mixture was worked-up in a similar manner to that described for the preparation of **7a**, **b** and the crude products were purified by chromatography on silica gel (benzene). The first eluate gave **16**[14] (195 mg, 59%) as an oil. IR $\nu_{max}^{CHCl_3}$ cm$^{-1}$: 1725. $^1$H-NMR ($CDCl_3$) $\delta$: 2.11 (3H, s, SMe), 3.78 (3H, s, OMe), 4.61 (1H, s, ArCH), 6.2—6.6 (2H, m, arom.), 7.38 (1H, br s, arom.). The second eluate gave **17** (65 mg, 12%) as an oil, whose physical data are listed in Table I.

**Methyl 2-(2-Furyl)-2-(methylthio)propionate (18)**——A solution of **16** (470 mg, 2.53 mmol) in dimethylformamide (4 ml) was added to a suspension of sodium hydride (60% mineral oil dispersion) (110 mg, 2.75 mmol) in dimethylformamide (3 ml) at 0 °C under an argon atmosphere, and the mixture was stirred at the same temperature until the evolution of hydrogen ceased. Methyl iodide (800 mg, 5.6 mmol) was then added and the mixture was stirred at 0 °C for 30 min and at room temperature for 40 min. The reaction mixture was poured into a solution of ammonium chloride (700 mg) in water (14 ml) and extracted with ethyl ether (20 ml × 2). The extract was washed with water (10 ml × 2) and dried ($MgSO_4$), then the solvent was evaporated off. The residue was chromatographed on silica gel (benzene) to give **18** (440 mg, 87%) as an oil, whose physical data are listed in Table I.

**Methyl 2-(2-Furyl)propionate (19)**——Zinc dust (1.5 g) was added to a solution of **18** (220 mg, 1.1 mmol) in acetic acid (3 ml), and the mixture was heated with vigorous stirring at 100 °C for 1 h, then cooled. The reaction mixture was

worked-up in a similar manner to that described for the preparation of **10a**—**c** and the crude product was purified by chromatography on silica gel (benzene) to give **19**[15] (130 mg, 77%). $^1$H-NMR ($CDCl_3$) $\delta$: 1.51 (3H, d, $J$=7 Hz, CMe), 3.70 (3H, s, OMe), 3.82 (1H, q, $J$=7 Hz, ArC$\underline{H}$), 6.1—6.4 (2H, m, arom.), 7.35 (1H, br s, arom.).

**Methyl 2-[5-(1-Methylthio-2-oxopropyl)-2-furyl]propionate (20)**——$ZnCl_2$ (172 mg, 1.26 mmol) was added to a stirred solution of **19** (130 mg, 0.84 mmol) and **2** (175 mg, 1.26 mmol) in dry $CH_2Cl_2$ (8 ml) at $-20$ °C, and the mixture was stirred at 0 °C for 10 min. The reaction was quenched with water (10 ml) and the reaction mixture was worked-up in a similar manner to that described for the preparation of **7a**, **b**. The crude product was purified by chromatography on silica gel (*n*-hexane–ethyl acetate) to give **20** (58 mg, 27%) as an oil, whose physical data are listed in Table I.

**Methyl 2-[5-(2-Oxopropyl)-2-furyl]propionate (21)**——Zinc dust (1 g) was added to a solution of **20** (88 mg, 0.34 mmol) in acetic acid (2 ml), and the mixture was heated with vigorous stirring at 100 °C for 1 h. Work-up as described above for the preparation of **11a**—**c** afforded **21**[4] (60 mg, 84%) as an oil. IR $\nu_{max}^{CCl_4}$ cm$^{-1}$: 1740, 1720. $^1$H-NMR ($CCl_4$) $\delta$: 1.49 (3H, d, $J$=7 Hz, CMe), 2.07 (3H, s, COMe), 3.66 (3H, s, OMe), 3.5—3.9 (3H, br, ArC$\underline{H}$, ArC$\underline{H}_2$), 6.03 (2H, s, arom.).

**Dimethyl 2,5-Furandiacetate (23)**——Raney nickel (W-2) (*ca.* 1 g) was added to a solution of **17** (149 mg, 0.49 mmol) in ethanol (10 ml), and the mixture was heated under reflux for 3 h, then cooled. After removal of the Raney nickel, the solvent was evaporated off and the residue was chromatographed on silica gel (benzene) to give **23**[6] (61 mg, 59%) as an oil. $^1$H-NMR ($CDCl_3$) $\delta$: 3.63 (4H, s, ArC$\underline{H}_2$ × 2), 3.71 (6H, s, OMe × 2), 6.16 (2H, s, arom.).

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## References and Notes

1) *a*) Y. Tamura, H.-D. Choi, H. Shindo, and H. Ishibashi, *Chem. Pharm. Bull.*, **30**, 915 (1982); *b*) Y. Tamura, H.-D. Choi, M. Mizutani, Y. Ueda, and H. Ishibashi, *ibid.*, **30**, 3574 (1982).
2) The reaction of phenylacetone with **1** in the presence of $SnCl_4$ afforded only a slight amount of ethyl α-methylthio-4-(2-oxopropyl)phenylacetate.
3) A. G. M. Barrett and H. G. Sheth, *J. Chem. Soc., Chem. Commun.*, **1982**, 170 and references cited therein.
4) H. Gerlach and H. Wetter, *Helv. Chim. Acta*, **57**, 2306 (1974).
5) Activated aromatic compounds such as furan and anisole sometimes give disubstituted products, depending on the reaction conditions.
6) O. Rau, W. Kern, and G. Spiteller, *Justus Liebigs Ann. Chem.*, **1984**, 1504.
7) All melting and boiling points are uncorrected. The infrared (IR) spectra were recorded with a JASCO IRA-1 spectrophotometer. The proton nuclear magnetic resonance ($^1$H-NMR) spectra were measured on a Hitachi R-20A spectrometer using tetramethylsilane as an internal standard. High-resolution mass spectra (MS) were obtained with a JMS-D-300 instrument at 70 eV.
8) B. van Zanten and W. Th. Nauta, *Recl. Trav. Chim. Pays-Bas*, **79**, 1211 (1960).
9) K. Sakamoto and M. Oki, *Bull. Chem. Soc. Jpn.*, **47**, 2623 (1974).
10) E. Schwenk and D. Papa, *J. Org. Chem.*, **11**, 798 (1946).
11) J. V. Braun, G. Irmisch, and J. Nelles, *Chem. Ber.*, **66**, 1471 (1933).
12) W. A. W. Cummings and K. Whittaker, *J. Appl. Chem.*, **12**, 86 (1962).
13) H. Böhme and W. Krack, *Justus Liebigs Ann. Chem.*, **1977**, 51.
14) G. Tsuchihashi and K. Ogura, German Patent 2709504 (1977) [*Chem. Abstr.*, **87**, 201300 (1977)].
15) G. Beck and E. Henseleit, *Chem. Ber.*, **104**, 21 (1971).