2-Methoxy-1-methylnaphthalene (8).[12] **Method A.** 1-(Chloromethyl)-2-methoxynaphthalene (**7**)[6] (4.2 g, 0.02 mol) was dissolved in 200 mL of room temperature ethyl acetate and filtered from a small amount of insoluble material. Calcium carbonate (4 g) and 5% Pd/C (0.7 g) was added to the solution, and the mixture was hydrogenated at 50 psi for 2 h. The reaction was filtered through a Celite pad, and the filtrate was evaporated to dryness. The resulting oil was extracted with hot hexane (50 mL). The extracts were evaporated to an oil that formed white crystals of **8** (3.0 g, 87%) on cooling in an ice bath. Recrystallization from 12 mL of methanol gave analytically pure crystals (1.6 g): mp 39–39.5 °C (lit.[12] mp 39 °C); IR (thin film) 800, 740 cm^{-1}.

Method B. 6-Bromo-1-(chloromethyl)-2-methoxynaphthalene (**4**) (5.7 g, 0.02 mol) was dissolved in 125 mL of warm ethyl acetate. Triethylamine (5 mL) and 5% Pd/C (500 mg) were added, and the mixture was hydrogenated as in method A. The reaction was worked up as above and the white crystals of **8** (3.3 g, 96%); mp 35–7 °C, were identical by IR and NMR with the product prepared by method A.

2-(Hydroxymethyl)-6-methoxynaphthalene (9).[14] 6-Methoxy-2-naphthoic acid[13] (202 g, 1 mol) was dissolved in 1 L of dry THF (warm) and added dropwise to a mixture of dry THF (2 L) and 40 g (1 mol) of $LiAlH_4$ while cooling the reaction with an ice bath. The reaction mixture was stirred overnight at room temperature; cooled in an ice bath, and quenched by the consecutive dropwise addition of 40 mL of water, 40 mL of 15% aqueous NaOH, and 120 mL of water. The mixture was filtered and the precipitate washed with hot THF. The light yellow filtrate was evaporated to dryness and the solid was recrystallized from 800 mL of ethanol, yielding white crystals of **9** (137 g, 73%): mp 116–118 °C (lit.[14] mp 116–117 °C); NMR (Me_2SO-d_6) δ 3.85 (s, 3, OCH_3), 4.65 (d, 2, J = 5 Hz, CH_2), 5.23 (t, 1, J = 5 Hz, OH), 7.1–7.9 (m, 6).

2-(Bromomethyl)-6-methoxynaphthalene (10).[4] In a 1-L three-necked round-bottomed with football stirrer, gas inlet tube, and drying tube was added 56.4 g (0.3 mol) of finely powdered 2-(hydroxymethyl)-6-methoxynaphthalene (**9**). The mixture was stirred and kept between 15 °C and 20 °C in an ice bath while HBr gas was bubbled into the mixture. Hydrogen bromide addition was terminated when a homogenous light green solution was obtained. The reaction was poured over 500 mL of ice-cold 10% aqueous Na_2CO_3, and the organic layer was washed with water (300 mL), dried (Na_2SO_4), and evaporated to dryness (warm water bath). The resulting white solid was recrystallized from 250 mL of hexane by cooling to −10 °C; and the crystals of **10** were collected by filtration: mp 84–5 °C (lit.[4] mp 79–80 °C); TLC (SS2) showed one fast moving spot. The filter cake was air-dried for 2 min and used directly for the preparation of the nitrile **11**.

6-Methoxy-2-naphthaleneacetonitrile (11). The 2-(bromomethyl)-6-methoxynaphthalene (**10**) from the above reaction (assumed 0.3 mol) was dissolved in 500 mL of CH_2Cl_2. Benzyltriethylammonium chloride (13.7 g. 0.06 mol) and NaCN (45 g, 0.9 mol) dissolved in 75 mL of hot water were added to the reaction. The two-phase system was heated gently with a steam bath and stirred (overhead stirrer). The reaction was monitored by TLC (SS2). After 20 h the organic layer was washed with brine (3 × 100 mL), dried (Na_2SO_4), evaporated, and the residue was recrystallized from 600 mL of ethanol to give a 76% yield (45.0 g) of nitrile **11**: mp 102–3 °C. Recrystallization of 500 mg from 8 mL of ethanol gave an analytical sample: mp 103–4 °C; IR (KBr) 2230, 855, 820 cm^{-1}.

Anal. Calcd for $C_{13}H_{11}NO$: C, 79.16; H, 5.62; N, 7.10. Found: C, 78.97; H, 5.76; N, 7.05.

Registry No. **1**, 3401-47-6; **4**, 92643-16-8; **5**, 92643-17-9; **6**, 71056-97-8; **7**, 67367-39-9; **8**, 1130-80-9; **9**, 60201-22-1; **10**, 73022-40-9; **11a**, 71056-96-7; 6-methoxy-2-naphthoic acid, 2471-70-7.

Supplementary Material Available: (a) Crystallography methods discussion, (b) Figure 1, ORTEP drawing of **4**, (c) Table I, crystal data summary for **4**, (d) line drawing of **4** showing numbering scheme, (e) Table II listing fractional coordinates for atoms in **4**, (f) Table III listing angles in degrees for **4**, (g) Table IV listing bond distances in angstroms for **4**, (h) Table V listing anisotropic thermal parameters (7 pages). Ordering information is given on any current masthead page.

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A New Synthesis of Medium and Large Membered Lactones via Denitration of Nitro Lactones

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Although a number of methods for preparing macrolides have been reported,[1] the recent method based on ring expansion of 2-(3-hydroxypropyl)-2-nitrocyclohexanones **1** to nitro lactones **2** as in Scheme I is very attractive, for **2** can be prepared in good yields from readily available materials and without requirement of tedious procedures such as high dilution and/or very slow mixing of reactants.[2,3] It was originally reported that **2** could be converted into keto lactones by the Nef reaction, but we felt that conversion of **2** into nitro-free lactones **3** via direct replacement of the nitro group by hydrogen would be highly desirable for preparing macrolides. In this paper we report the realization of this conversion by treatment of **2** with tributyltin hydride, (Bu_3SnH).

We have reported that aliphatic nitro groups are efficiently replaced by hydrogen without affecting other functional groups on treatment with Bu_3SnH (1.3 equiv) in the presence of azobis(isobutyronitrile) (AIBN, 0.3 equiv) at 80 °C for 1–2 h.[4] This reaction is now being used as a useful strategy for organic synthesis.[5] However, this procedure cannot be applied to the denitrohydrogenation of all kinds of nitroalkanes. In general, primary and secondary nitro groups are not replaced by hydrogen in good yields by this procedure.[6] For example, heating a mixture of 6-nitro-9-nonanolide (**2a**), Bu_3SnH (1.3 equiv), and AIBN (0.3 equiv) in benzene for 2 h at 80 °C gave only a trace amount of 9-nonanolide (**3a**). And unidentified products were mainly obtained. The use of Bu_3SnH in large excess improved the yield of **3a**. Heating a mixture of **2a**, Bu_3SnH (5 equiv), and AIBN (0.8 equiv) in toluene at 110 °C for 30 min gave **3a** in 26% yield. Similarly, 12-nitro-15-pentadecanolide (**2b**, n = 10) was converted into 15-pentadecanolide (**3a**) in 25% yield.

The present denitration was applied to the synthesis of 9-decanolide (**3c**) (Scheme II), which is a natural product

(1) See the following reviews: (a) Nicolaou, K. C. *Tetrahedron* **1977**, 683. (b) Masamune, S.; Bates, G. S.; Corcoran, I. W. *Angew. Chem., Int. Ed. Engl.* **1977**, *16*, 585. (c) Back, T. G. *Tetrahedron* **1977**, 3041.
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(6) The nitro group of the following secondary nitro compounds is replaced by hydrogen in good yields by this procedure, i.e., benzylic and allylic nitro compounds, α-nitro ketones, and α-nitro esters.[4] The reason for this difficulty for denitration of secondary nitro compounds is not clear yet, but probably nitro compounds are reduced to the nitrogen derivatives on treatment with Bu_3SnH when the carbon–nitrogen bond is hard to break.

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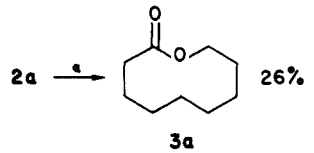


(a) Bu_3SnH (5 equiv), AIBN (0.8 equiv), 110 °C, 30 min.

isolated from *Phoracanthe synonyma*.[7] The requisite nitro lactone, 6-nitro-9-decanolide was prepared by ring expansion of the nitro alcohol **1c** in 81% yield.[3] Denitration of **2c** was carried out under similar conditions as that for **2a** to give **3c** in 48% yield. Although the yield of the denitration is only moderate (25–48% yield) in the present case, the method consisting of ring expansion of **2** and the subsequent denitration is useful to prepare certain macrolides, particularly ten-membered lactones, for they are generally recognized to be cyclized less efficiently than other cases.[8] On the other hand, ten-membered lactones are especially easily prepared by the present method, for the starting materials of ten-membered lactones are cyclohexanones in the present case. Considering this merit and the simplicity of the experimental procedures, the present synthesis of **3**, particularly **3c**, is fully compatible with the known methods.[7]

Further synthetic utility of **2** is nicely demonstrated in Scheme III, where hydroxymethyl derivatives of macrolides are regioselectively prepared. Treatment of **2** with 37% HCHO in the presence of a catalytic amount of base gave the hydroxymethylated nitro lactones which were further converted into the acetoxymethylated derivatives **4** of lactones via acetylation and the subsequent denitration with Bu_3SnH (1.3 equiv). Compounds **5** are versatile intermediates for further structual modifications.

Experimental Section

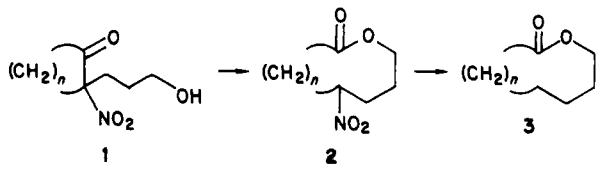
Materials. Nitro lactones **2** were prepared by literature methods.[2,3] The structure of **2** was confirmed by comparison with data reported in the literatures.[2,3] As these literatures did not describe the experimental details, preparation of **2a** was described here as a typical procedure.

6-Nitro-9-nonanolide (2a). A mixture of 2-nitro-2-(3-hydroxypropyl)cyclohexanone (**1a**, 0.77 g, 3.8 mmol) and sodium hydride (50% dispersion, 0.02 g, 0.4 mmol) in dimethoxyethane (5 mL) was refluxed for 45 min. The reaction mixture was then poured into cold water containing a small amount of acetic acid and extracted with ether. The ether extract was washed with water, dried with anhydrous magnesium sulfate, and concentrated in vacuo. The residue was purified by column chromatography (silica gel/benzene–hexane) to give **2a** (0.70 g, 91% yield): NMR ($CDCl_3$) δ 1.5–2.8 (m, 12 H), 3.90 (m, 1 H), 4.6–5.1 (m, 2 H); IR (neat) 1710, 1530, 1440, 1370, 1240, 1160, 1050 cm^{-1}.

12-Nitro-15-pentadecanolide (2b). A mixture of **1b** (4.72 g, 16.6 mmol) and sodium hydride (1.7 mmol) in dimethoxyetane (20 mL) was refluxed for 30 min. The same workup as in the preparation of **2a** gave **2b** (4.07 g, 87% yield): NMR ($CDCl_3$) δ 1.1–2.1 (m, 22 H), 2.32 (t, J = 7.5 Hz, 2 H), 4.1 (m, 2 H), 4.5 (m, 1 H); IR (eat) 1720, 1540, 1450, 1370, 1240, 1180 cm^{-1}.

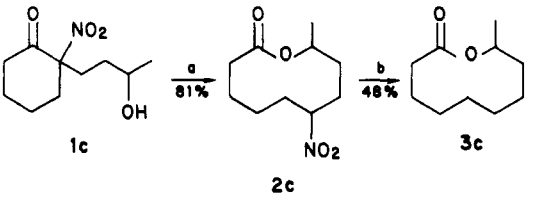
6-Nitro-9-decanolide (2c). A mixture of **1c** (1.78 g, 8.3 mmol) and sodium hydride (0.41 mmol) in dimethoxyetane (10 mL) was refluxed for 60 min. The same workup as described in the preparation of **2a** gave **2c** (1.43 g, 81% yield): NMR ($CDCl_3$) δ 1.1–2.7 (m, 12 H), 1.28 (d, J = 7.5 Hz, 3 H), 4.7–5.0 (m, 2 H); IR (neat) 1720, 1540, 1440, 1370, 1250 cm^{-1}.

Scheme I[a]



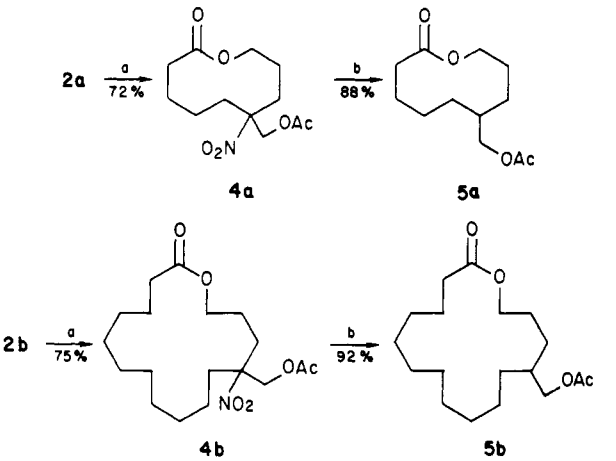
[a] a, n = 4; b, n = 10.

Scheme II. Preparation of 9-Decanolide (3c)[a]



[a] (a) NaH/DME; (b) Bu_3SnH (5 equiv), AIBN (0.8 equiv), 110 °C, 30 min.

Scheme III. Preparation of Hydroxymethyl Derivatives of Macrolides[a]



[a] (a) i, 37% HCHO, NaOH/*i*-PrOH; ii, Ac_2O/pyridine; (b) Bu_3SnH (1.3 equiv), AIBN (0.3 equiv), 80 °C, 90 min.

Synthesis of Macrolides 3 by Denitration. The structure of **3** was assigned on the basis of spectral data, which are in good agreement with those reported.[7,9]

9-Nonanolide (3a). A mixture of **2a** (0.91 g, 4.4 mmol), Bu_3SnH (6.55 g, 22 mmol), and AIBN (0.59 g, 3.6 mmol) in toluene (5 mL) was refluxed for 30 min. The reaction mixture was subjected to column chromatography to give **3a** (0.18 g, 26% yield): NMR ($CDCl_3$) δ 1.2–1.9 (m, 12 H), 2.38 (t, J = 7 Hz, 3 H), 4.25 (t, J = 7 Hz, 2 H); IR (neat) 1720, 1460, 1240, 1160 cm^{-1}.

15-Pentadecanolide (3b). From the reaction of **2b** (0.94 g, 3.3 mmol) with Bu_3SnH (4.80 g, 16.5 mmol), **3b** was obtained in 25% yield (0.20 g): NMR ($CDCl_3$) δ 0.8–1.8 (m, 24 H), 2.36 (t, J = 7 Hz, 2 H), 4.14 (t, J = 7 Hz, 2 H); IR (neat) 1720, 1453, 1240, 1131, 1100 cm^{-1}.

9-Decanolide (3c). A mixture of **2c** (0.68 g, 3.2 mmol), Bu_3SnH (4.66 g, 16 mmol), and AIBN (0.42 g, 2.6 mmol) in toluene (3 mL) was refluxed for 30 min. Pure **3c** was isolated in 48% yield (0.29 g) after column chromatography (silica gel/benzene–hexane): NMR ($CDCl_3$) δ 1.27 (d, J = 7.5 Hz, 3 H), 0.8–2.7 (m, 14 H), 5.05 (m, 1 H); IR (neat) 1715, 1455, 1240, 1150 cm^{-1}.

Synthesis of 6-(Acetoxymethyl)-6-nitro-9-nonanolide (4a). A mixture of **2a** (0.68 g, 3.4 mmol), 37% HCHO (0.68 g, 3.4 mmol), and NaOH (0.05 g, 1.1 mmol) in isopropyl alcohol (10 mL) was stirred at room temperature for 30 min. Then the reaction mixture

(7) Synthesis of **3c** has been done by various methods, see: (a) Wakamatsu, T.; Akasaka, K.; Ban, Y. *J. Org. Chem.* **1979**, *44*, 2008. (b) Takahasi, T.; Hashiguchi, S.; Kasuga, K.; Tsuji, J. *J. Am. Chem. Soc.* **1978**, *100*, 7424. (c) Barbier, M. *J. Chem. Soc., Chem. Commun.* **1982**, 668. (d) Mahajan, J. R.; Araujo, H. C. *Synthesis* **1981**, 49.

(8) Ring closure of cesium carboxylates is a good method for preparing macrolides, but C_9 and C_{10} cesium ω-halogenoalkanoates do not give lactones in good yields, see: Kruizinga, W. H.; Kellogg, R. M. *J. Am. Chem. Soc.* **1981**, *103*, 5183 and references therein.

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was poured into water, acidified by dilute hydrochrolic acid, and extracted with ether. The crude product after the usual workup was added to a solution of acetic anhydride (0.35 g, 3.4 mmol) in pyridine (1 mL). The resulting solution was stirred at room temperature for 30 min, then poured into 5 mL of 2 N hydrochrolic acid, and extracted with ether. After the usual workup, the crude product was subjected to column chromatography (silica gel/benzene–hexane) to give **4a** (0.68 g, 72% yield): NMR ($CDCl_3$) δ 1.2–1.5 (m, 12 H), 2.04 (s, 3 H), 4.05 (m, 2 H), 4.30 (d, 2 H); IR (neat) 1720, 1530, 1460, 1360, 1240 cm^{-1}.

Anal. Calcd for $C_{12}N_{19}NO_6$: C, 52.74; H, 7.01; N, 5.13. Found: C, 52.53; H, 6.76; N, 5.09.

Synthesis of 12-(Acetoxymethyl)-12-nitro-15-pentadecanolide (4b). The same procedure starting from **2b** (1.13 g, 4.0 mmol) as in the preparation of **4a** gave **4b** (1.05 g, 75% yield): NMR ($CDCl_3$) δ 1.0–2.2 (m, 22 H), 2.04 (s, 3 H), 2.32 (t, J = 7 Hz, 2 H), 4.08 (t, J = Hz, 2 H), 4.36 (s, 2 H); IR (neat) 1715, 1700, 1530, 1440, 1230 cm^{-1}.

Anal. Calcd for $C_{18}H_{31}NO_6$: C, 60.48; H, 8.74; N, 3.92. Found: C, 60.43; H, 8.49; N, 3.88.

Synthesis of 6-(Acetoxymethyl)-9-nonanolide (5a). A mixture of **4a** (0.64 g, 2.3 mmol), Bu_3SnH (0.96 g, 3.28 mmol), and AIBN (0.12 g, 0.70 mmol) in benzene (5 mL) was heated at 80 °C for 90 min. Then the reaction mixture was subjected to column chromatography (silica gel/benzene–hexane) to give **5a** (0.47 g, 88% yield; NMR ($CDCl_3$) δ 0.8–2.6 (m, 13 H), 2.06 (s, 3 H), 3.66–4.04 (m, 2 H), 4.48–4.74 (m, 2 H); IR (neat) 1720, 1700, 1240 cm^{-1}.

Anal. Calcd for $C_{12}H_{20}O_4$: C, 63.14; H, 8,83. Found: C, 63.16; H, 8.66.

Synthesis of 12-(Acetoxymethyl)-15-pentadecanolide (5b). The same procedure as in the preparation of **5a** starting from **4b** (0.60 g, 1.7 mmol), Bu_3SnH (0.64 g, 2.2 mmol), and AIBN (0.09 g, 0.56 mmol) gave **5b** (0.48 g, 92% yield): NMR ($CDCl_3$) δ 0.9–1.8 (m, 23 H), 2.02 (s, 3 H), 2.32 (t, J = 7 Hz, 2 H), 3.92 (d, J = 7.5 Hz, 2 H), 4.10 (t, J = 7 Hz, 2 H); IR (neat) 1720, 1240 cm^{-1}.

Anal. Calcd for $C_{18}H_{32}O_4$: C, 69.19; H, 10.32. Found: C, 68.81; H, 10.09.

Registry No. **1a**, 84246-77-5; **1b**, 84246-80-0; **1c**, 92643-60-2; **2a**, 84246-81-1; **2b**, 84246-84-4; **2c**, 81590-80-9; **3a**, 6008-27-1; **3b**, 106-02-5; **3c**, 61448-27-9; **4a**, 92643-61-3; **4a**-ol, 92643-65-7; **4b**, 92643-62-4; **5a**, 92643-63-5; **5b**, 92643-64-6; Bu_3SnH, 688-73-3.

Reaction of 2-Nitrobenzaldehydes with Diethyl (Diethoxyphosphinyl)succinate: A New Synthesis of Quinoline-2,3-dicarboxylic Acid Esters via Their *N*-Oxides

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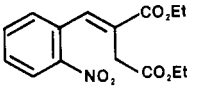
The synthesis of quinoline-2,3-dicarboxylic acid derivatives can be accomplished by the Friedlander reaction of 2-aminobenzaldehydes with diethyl oxaloacetate[1,2] or by an analogous reaction that utilizes the same aldehydes and acetylenedicarboxylic acid esters.[3] However, the difficulties encountered in preparing and storing 2-aminobenzaldehydes have severely limited the application of these synthetic procedures.

We report that when 2-nitrobenzaldehydes **1**, which are much more readily available and stable than their corresponding amino analogues, are treated with diethyl (diethoxyphosphinyl)succinate under alkaline conditions, a reaction frequently referred to as the modified Wittig reaction,[4] the appropriate diethyl quinoline-2,3-dicarboxylate *N*-oxides **2** are obtained. Subsequent reduction of the *N*-oxides with phosphorous trichloride provides, in toto, a novel and facile route to the preparation of quinoline-2,3-dicarboxylic acid esters **3** (Scheme I) that circumvents the problems associated with the use of 2-aminobenzaldehydes. Furthermore, while the participation of 2-nitrobenzaldehyde in the modified Wittig reaction[5] and the condensation of carbanions with nitro functionalities[6] have each been reported previously, the simultaneous occurrence of both types of reaction appears to be without precedent.

The reactions leading to the formation of the *N*-oxides were straightforward (Table I). Briefly, addition of diethyl (diethoxyphosphinyl)succinate to a solution of sodium and **1** in cold ethanol or ethanol–dimethylformamide resulted in a dark reaction mixture. Progress of the reaction was followed by thin-layer chromatography, and, after 1–3 h of stirring, the ethanol was evaporated and the residue purified by recrystallization or by chromatography. While yields were only modest, the effects of reaction variables such as temperature, solvent, and base were not investigated. In the case of **2a**, authentic material also was prepared by the reaction of **3a** with H_2O_2 in acetic acid. The **3a** used in the latter process was obtained by esterification of the corresponding dicarboxylic acid, a low-yield oxidation product of acridine.[7]

The NMR spectra of the quinoline/quinoline *N*-oxide pairs generally were similar except for the signals exhibited by the proton at position 8 of the quinoline nucleus. For example, in the spectrum of **2c** this proton produced a doublet centered at δ 8.83 (J = 8 Hz) while in that of **3c** the same doublet appeared at δ 8.17, an upfield shift engendered by reduction of the *N*-oxide moiety.

The proclivity of the modified Wittig reaction to afford *trans*-olefins is well-known.[4] Consequently, the likely initial product arising from the reaction of **1** with diethyl (diethoxyphosphinyl)succinate is the *trans*-cinnamate derivative **4** in which the juxtaposition of the acidic



4

methylene group and the nitro substituent appears to favor the condensation, probably through a radical anion mechanism,[6] culminating in **2**.

Experimental Section

Data on each quinoline *N*-oxide **2** and quinoline **3** are shown in Table I. Melting points are uncorrected. Starting aldehydes were obtained commercially except for **1d** which was prepared according to ref 8.

Diethyl Quinoline-2,3-dicarboxylate *N*-Oxide (2a). A cold solution of 3.8 g (0.025 mol) of 2-nitrobenzaldehyde and 0.62 g (0.027 mol) of sodium in 45 mL of ethanol was treated, during 15 min, with a solution of diethyl (diethoxyphosphinyl)succinate[9]

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