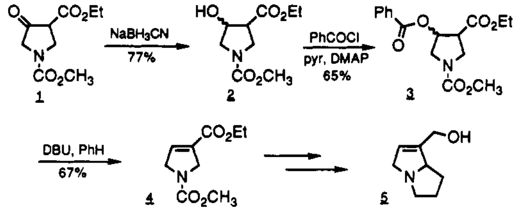
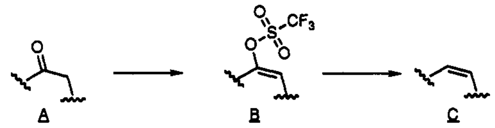
Scheme I



Scheme II



rakis(triphenylphosphine)palladium(0), which produced 4 in 63–70% overall yield from 1 after purification.

In summary, significant improvement on the published route to the title compound was made, and the method described herein should provide ready access to other related derivatives.

Experimental Section[12]

3-(Ethoxycarbonyl)-*N*-(methoxycarbonyl)-2,5-dihydro-1*H*-pyrrol-4-yl Trifluoromethanesulfonate (6). Pyrrolidone 1[5] (1.7 g, 7.9 mM) and 1,8-bis(*N*,*N*-dimethylamino)naphthalene (Proton sponge, 1.8 g, 8.7 mM) were dissolved in dry dichloromethane (20 mL). The solution was cooled to 0 °C in an ice bath and trifluoromethanesulfonic anhydride (1.42 mL, 8.5 mM) was added dropwise over 10 min. The mixture was stirred for 3–4 h at room temperature, filtered through coarse silica gel (Merck 60–200 mesh), and then concentrated under reduced pressure. The reside was used crude in the next experiment. An analytical sample of 6 was obtained by flash chromatography (SiO_2, hexane/ethyl acetate, 3:1): mp 52 °C (hexane); ^1H NMR ($CDCl_3$, 270 MHz) δ 4.45 (s, 4 H), 4.31 (q, J = 7.2 Hz, 2 H), 3.76 (s, 3 H), 1.34 (t, J = 7.2 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz) δ 159.9, 154.7, 125.6, 119.8, 61.9, 53.0, 51.4 (t, J = 13.4 Hz), 50.9, 30.5, 14.0; IR ($CHCl_3$) 1710, 1670, 1390, 1122 cm^{-1}. Anal. Calcd for $C_{10}H_{12}NO_7SF_3$: C, 34.58; H, 3.48; N, 4.03. Found: C, 34.53; H, 3.49; N, 3.99.

Ethyl *N*-(Methoxycarbonyl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate (4). In a dry 100-mL round-bottom flask flushed with dry argon, tetrakis(triphenylphosphine)palladium(0) (147 mg, 0.127 mM) and lithium chloride (808 mg, 19.1 mM, dried overnight at 0.1 mmHg) were mixed with dry THF (20 mL). The crude triflate 6 (2.0 g, 6.35 mM) in dry THF (20 mL) was added rapidly. Freshly distilled tributyltin hydride (2.07 mL, 7.68 mM) was added slowly over 2 min, and the mixture was heated at reflux for 3 h and cooled then diluted with hexane (40 mL). The reaction mixture was washed with 5% aqueous ammonium hydroxide (3 × 50 mL) and saturated sodium chloride (2 × 100 mL), filtered through silica gel, and then concentrated under reduced pressure. The residue was purified by flash chromatography (SiO_2, hexane/ethyl acetate, 3:1) to yield 0.89 g (70%) of a white solid with spectral characteristics identical with the literature values:[3] mp 75 °C (hexane/ethyl acetate); ^1H NMR ($CDCl_3$, 270 MHz) δ 6.74 (br d, J = 9.0 Hz, 1 H), 4.40–4.32 (complex m, 4 H), 4.24 (q, J = 7.0 Hz, 2 H), 3.74 (s, 3 H), 1.31 (t, J = 7.0 Hz, 3 H); ^{13}C NMR ($CDCl_3$, 67.5 MHz), δ 162.4, 155.1, 136.2, 132.3, 60.6, 54.1, 52.4, 51.8, 14.1; IR ($CHCl_3$) 1692, 1630 cm^{-1}.

Acknowledgment. Financial support was received from the National Science Foundation and is gratefully acknowledged.

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[12] All reagents were obtained from Aldrich Chemical Co., Inc., Milwaukee, WI, and were used without further purification except where noted. Melting points are uncorrected.

Additions and Corrections

Vol. 55, 1990

Charles A. Panetta,* Stephanie M. Garlick, H. Dupont Durst, Frederick R. Longo, and J. Richard Ward. Synthesis of 4-Alkyl-2-iodosobenzoic Acids: Potent Catalysts for the Hydrolysis of Phosphorus Esters.

Page 5202, replace Phosphorous in title with Phosphorus.

Page 5203, column 1, line 1, replace phosphorous with phosphorus.

Yoshinori Yamamoto* and Naoki Asao. Copper Azide as a New Reagent for Syn-S_N2 Displacement of γ-Sulfonyloxy α,β-Unsaturated Esters.

Page 5303. CAUTION: Copper azide should be handled carefully behind a safety screen in a hood. Avoid heating this compound or shocking the reagent (see: Fedoroff, B. T.; et al. *Encyclopedia of Explosives and Related Items*, Vol. 1; Picatinny Arsenal: Dover, NJ, 1960; pp A534–A535). We appreciate Dr. Horst G. Adolph at Naval Surface Warfare Center for bringing this to our attention.

Vol. 56, 1991

John S. Lomas,* Sylvette Briand, and Dominique Fain. Reactions of Thermally Generated *tert*-Butyl and Di(*tert*-alkyl)ketyl Radicals in Toluene: Cage Effects and Hydrogen Transfer.

Page 166. Professor H. Fischer (Zurich) has informed us of unpublished data from his Laboratory (Dütsch, H. Doctoral Thesis, 1982; Münger, K. Doctoral Thesis, 1985) on hydrogen transfer from toluene to the *tert*-butyl radical. The Arrhenius plot of these data ($\log A = 5.9$; $E_a = 6.9$ kcal mol^{-1}) intersects ours ($\log A = 9.8$; $E_a = 14.4$ kcal mol^{-1}) at about 145 °C, all our data lying above Fischer's plot and the greatest difference between the two plots corresponding to a factor of 4.

Michael Lehd and Frank Jensen*. Improved Radical Stabilization Energies.

Page 884. The AUHF method was originally developed by T. Kovar and T. Clark and consequently ref 4 should include the following: (b) Kovar, T.; Clark, T. Manuscript in preparation.